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Greetings from the General Chair

Welcome to the 2016 Solid State Sensors, Actuators, and Microsystems Workshop; the seventeenth in the biennial series of regional meetings that alternate with the International Transducers Conference. The goal of the Workshop - since its inception in 1984 - has been to provide a forum for presentation of new ideas and leading-edge research achievements, in a setting that facilitates exchange of ideas and community building.

Tina Lamers has assembled and led an expert program committee in support of the Workshop's goal. Mind-expanding plenary presentations by leading academic and industry experts will kick-off each day, followed by oral and poster sessions of contributed papers. The Workshop schedule remains organized around single session oral and poster presentations. We have continued the tradition of significant unstructured time for informal discussions to support exchange of ideas and community building. Be sure to use these intervals for technical brainstorming and collaboration building, as well as career and business development.

All papers were selected on the basis of high-quality content and leading-edge contribution. Those deemed of broader interest to the attendees were designated as oral and those of special interest as poster. The "shotgun" format is used to inform the attendees of the content of the upcoming poster session of the day. Late News Oral and Poster papers selection process occurred in mid-March and bring the latest research to the attendees. In addition, we again have something new and exciting planned for the period formally known as the "Rump Session." A tremendous "thank you" goes to Tina Lamers and the Technical Program Committee for putting together the technical content of the Workshop, which required studying, debating, ranking, and selecting from 190 submitted quality abstracts.

James Walker (Chair), Michelle Bourke, Gary O'Brien, and Leland "Chip" Spangler worked tirelessly to create the commercial support and exhibition contributions of this meeting. A special thanks also goes to the Transducers Research Foundation for their travel grant support for the presenting student authors. The involvement of these individuals, and the financial support of the exhibitors and corporate sponsors are valuable and essential features of the Hilton Head Workshop, and I am most thankful to have had these outstanding people and companies participate.

Sara Stearns and her team at Preferred Meeting Management, Inc., above all, deserve special recognition and thanks for all of their hard work and superb organization in pulling this meeting together and for making it run so smoothly. The work they do "behind the scenes" over the two years of preparation to make this meeting special is exceptional; our community and our meeting has been the beneficiary of their excellence and commitment.

This meeting belongs to all of you; thank you for your enthusiastic participation. My best wishes for a memorable gathering in Hilton Head Island this June 2016.

Marsall

Mark G. Allen General Chair

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Not Pictured: Janhavi Agashe, Moses Noh, Mina Rais-Zadeh, and Mark Sheplak

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Monday, June 6

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7:45 am Welcome Mark G. Allen, University of Pennsylvania, USA Tina Lamers, Akustica, Bosch Group, USA

Plenary Presentation I

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8:15 am	INNOVATIVE SWITCHING TECHNOLOGIES FOR FUTURE TUNABLE
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	Pierre Blondy, A. Mennai, K. Nadeau, and A. Crunteanu

Universite de Limoges, XLIM, FRANCE

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Session Chair: T. Lamers, Aksutica, Bosch Group, USA

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- 4:15 pm See page 9 for listing of poster presentations

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MEMS & Sensors Industry Group Discussion Panel

Moderator: K. Lightman, MEMS & Sensors Industry Group, USA

7:00 pm - INDUSTRY'S GUIDE TO TECHNOLOGY TRANSFER AND 7:45 pm HOW TO COMMERCIALIZE YOUR MEMS/SENSORS PRODUCT

Panelists: Mary Ann Maher, *SoftMEMS, USA* Eric Pabo, *EV Group, USA* Jason Tauscher, *Microvision, USA*

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Diversity Event

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INNOVATIVE RF SWITCH TECHNOLOGIES

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ABSTRACT

The presentation will highlight new technologies developed for wideband, low loss and linear RF switch technologies. New switch structures, like phase transition and phase change materials will be described, along with emerging MEMS zero-level packaged capacitive switch devices. Through several examples, we will show the advantages and drawbacks of these innovative technologies.

INTRODUCTION

The ever-growing demand for wideband, wireless internet access is pushing the demand for reconfigurable, multi standard RF-front-ends, as shown in Fig. 1. Most RF architectures will make heavy use of RF switches, to configure antennas, filters and multiband amplifiers and antennas.



Figure 1: Typical 4G cellular phone architecture. The tunable components and switches are shown in the red squares.

RF-switches will be key element for the fabrication of multi standard reconfigurable and tunable front ends.

RF-switch performances stem from their on and off state characteristics like on-state resistance, and off state capacitance. In 50 Ω radiofrequency systems, the on-state resistance, R_{on} needs to be in the 1-10 Ω range in order to obtain reasonable loss (~0.1-2 dB). The isolation at microwave frequencies is dominated by the off state capacitance of the switch, C_{off5} and practical values are between 10-100 fF. The off-state resistance is usually high enough so that it can be neglected at high frequencies, but it may limit the isolation is lower end of the band.

For comparison purpose, the Ron. Coff product is a usual figure-

of-merit for RF switches, the lower values being the best.

By putting several devices in parallel, it is possible to reduce the overall insertion loss, by dividing the on state resistance, at the expense of degraded isolation. Similarly, putting several devices in series decreases the series capacitance, and improves isolation, but degrades insertion loss.

RF switches are dominated by semi-conductor technology, and most devices are currently using CMOS-derived technologies, using insulating substrates and stacked-gate low voltage transistors in order to obtain low loss, high power switches. Current state-ofthe-art CMOS RF switches have FoM around 100 fs, can handle 2 Watts of RF power, and have very low DC power consumption. Another important aspect is that CMOS technology is widely available and allows having secure, low cost technology sources for RF system providers.

However, the specific transistor gate-stacking technique developed to improve power handling makes increasing the operating frequency difficult, and other technologies have to be sought for future 5G broadband systems. For instance, there is a very limited number of options for high power switch technologies above 5 GHz, while 5G communication systems will permit commercial use of frequency band as high as 24 GHz.

Innovative material-based switches are among the possible candidates for building low cost, low loss broadband microwave switches. These materials can be relatively easily integrated into planar RF transmission lines and the resulting in-line planar switches have excellent performances [2].

A planar in-line RF switches cross section and layout is shown below. In the example shown below, the material based on phase change materials, but the structure is essentially the same for phase transition materials like VO_2 devices that will be discussed later in the paper.



Figure 2: Layout and cross section of a planar inline RF switch based on PCM and PTM materials.

As shown in Fig. 2, a heater is integrated in the device and allows the material reaching its transition temperature in the case of VO_2 switches. The same structure allows applying long or short temperature pulses to switch a PCM material to its crystalline, conductive state, or to set it back to the off state. The typical device length, analog to a transistor gate length, is between 1 and

10 µm, while typical widths are between 20-100 µm.

The planar nature of the switches allows obtaining very high isolation, with an off state capacitance less than a few tens of femtoFarads.

PHASE TRANSITION RF SWITCHES

Phase change materials, like Vanadium Dioxyde (VO₂), are excellent candidates for the fabrication of small size, low loss RF switches. VO₂ is a phase change material, with a Metal Insulating Transition (MIT) temperature at 68° C [1-6]. In practice, the resistivity of the material decreases by several orders of magnitude when the material temperature is higher that its transition temperature, compared to room temperature.

Vanadium dioxide layers can be obtained on sapphire substrates using E-beam evaporation of vanadium at high temperature in oxygen ambient. After several steps of thermal annealing, the layers present a sharp transition in resistivity when the temperature is increased.

Measured VO_2 resistivity versus temperature is shown in Fig. 3.



Figure 3: Measured electrical resistivity of a 200nm thick vanadium dioxide layer versus temperature.

From this resistivity diagram, it is possible to design microwave switches with a specific on-state resistance and very much like transistor gate, the device width and length can be tailored for a given application. A practical example of a VO_2 switch is shown below in Fig. 4, with a 3x20 μ m material length and width.



Figure 4: Picture of a fabricated Vo₂ microwave switch.



Figure 5: Measured performances of VO_2 switches at 25°C and 90°C.

The measured transmission parameters are shown in Fig. 5, for temperatures below (25°C) and above (90°C) VO₂ transition temperature.

It can be seen that the insertion loss are extremely small, with an equivalent series resistance of 2 Ω and an off state capacitance of 7.7 fF. The figure of merit of this switch is 15.4 fsec.

PHASE CHANGE MATERIAL (PCM) RF SWITCHES

Very recently, phase change materials (PCM), like GeTe binary chalcogenide alloys have been evaluated to develop RF switches and similarly to VO₂, they presents a high resistance in the amorphous off-state and low resistance in the crystalline on-state [5]. These alloys are very well known for the fabrication of DVD-RW discs, where their reflectivity is changed by the application of short/long optical pulses. For RF applications, these alloys have enough contrast to be used in low impedance systems, and can be used in similar circuit configurations like VO₂

Ternary alloys like $Ge_2Sb_2Te_5$ (GST) phase change material can also be used for the fabrication of RF/microwave switches. In a similar fashion as GeTe, GST presents reversible structural amorphous (a-GST) to crystalline (c-GST) thermally induced phase change [7]. This phase change is accompanied by intrinsic properties change of material, in particular a drastic resistivity drop when it passes from amorphous to crystalline state (up to 5 order of magnitude). For several years, GST and GeTe have been developed for non-volatile phase change memories (PCRAM) devices with a number of cycle between the two states greater than 10^5 cycles and a transition time of about 50 ns. For these properties, GST and GeTe are excellent candidates for RF switching applications because of large R_{off} / R_{on} ratio with a good compromise in terms of stability and transition time [6].

Compared to currently used RF technologies, the main advantage of all PCM-based switches is that they do not require permanent bias to hold a given state. This is a key feature compared to any other electronic switching technology. Indeed, bistable elements are common in high impedance logic circuits, but mechanical relays still dominate 50 Ω RF systems. For instance, space-borne switching matrices have to be configured on ground, before a satellite payload is launched, and such application requires the system to be working without running a remote configuration procedure.

Therefore, these RF switch matrices are still using bulky arrays of mechanical relays, and this part of satellite payloads would greatly benefit from PCM switch technology.

The bi-stable nature can be understood by looking at the resistance-temperature diagram below. Upon relatively slow heating, the material goes from amorphous to crystalline state, with a sharp conductivity increase. When a short, high-temperature pulse is applied, the crystal order is broken, and the material resistivity goes up again, and permits to switch the device back to its off state.



Figure 5: Resistance-Temperature diagram of a PCM switch, and typical melting (T_m) and crystallization (T_c) pulses.

The crystallization temperature is between 200°C and 300°C for GST and GeTe alloys, with a tradeoff between heating duration and temperature.

The melting temperature, taking the material back to its amorphous state, is above 700°C, for a very short amount of time, typically less than 1 μ sec. The time constant of the system has to be carefully optimized to avoid any over heating and precise applications of these short temperature profiles.

The endurance of these switches is still an open question and recent results have reported 10^5 cycles without any noticeable degradation in the contrast of the switch [6], and further optimization should lead to lifetimes better than 10^6 cycles



Figure 6: Optical picture of a PCM switch.



Figure 7: Typical measured S parameters of a GST alloy PCM switch.

Typical measured insertion loss of the devices is shown in Fig. 7 [ref], with reasonable insertion loss, and very high isolation.

The off state capacitance is only 7 fF while typical on-state resistance is 15 Ω , leading to a figure of merit of 105 fs.

DISCUSSION AND OTHER TECHNOLOGIES

Other technologies like RF-MEMS, have made significant progress over the past few years, and finally made their way into consumer electronics applications like cellular phones. The application of this technology is tunable capacitors for antennas, where very low loss and low power consumption are of prime interest.

Packaging of these devices is now part of the fabrication process allowing improved manufacturability and full CMOS processing compatibility.

While reliability has long been Achille's heel of this technology, packaging integration has allowed making great progress and commercial MEMS capacitors have lifetimes better than 10^{10} cycles [9]. A typical sketch of a zero-level packaged RF-MEMS switched capacitor [10] is shown below.



Figure 8: Cross section and microphotograph of a zero-level packaged RF-MEMS switched capacitor array.

These devices only include metal electrodes and the capacitive contrast between the actuated state and the un-actuated state is used as a switching element. Typical on state resistance are less than 0.5 Ω , while off-state capacitance can be as low as 12 fF, leading to figure of merit lower than 10 fs. However, this is obtained on devices with moderate capacitive contrast, well suited for antenna tuning, but not enough to obtain RF switches with significant isolation. Therefore, more work is needed to improve this contrast using similar MEMS structures, before this technology becomes a viable alternative to low-cost semiconductor switches mentioned in the introduction.

PCM switches are in the early stage of research, but they readily offer high performance devices, as shown by several universities and research institutions [6-8]. Cycling and reliability is an open question for these devices as expected for an emerging technology.

However, bi-stability is a clear and unique advantage for several applications in space borne systems, or programmable arrays of switched elements. Moreover, this technology is relatively simple, and easy to integrate into standard microelectronics processing.

Technology	Phase Transition (VO ₂)	Phase Change (GeTe/GST)	MEMS
R _{on} .C _{off} typ. (fs)	10-20	100-200	5-20
Actuation Bias	Temperature	Temp. Pulses	Electrostatic
Switching speed (µsec)	14	1	
Power /consumption	>10 mW	<10 nJ per transition	<100 µW
Power handling	Moderate	High (>1W)	High (>1W)
Pros	RF performances Small size	Small size Bistable Linearity	Low loss Low power Linearity
Cons	Power consumption Temperature sensitive	Limited cycling	Complex technology Low contrast

Table 1: Comparison of new RF switching technologies.

Phase Transition switches have a clear advantage in performances, but they require large bias currents, and temperature regulation. Still, their planar nature, and the sharp transition in resistivity that can be obtained allow them achieving wideband, high performance switching, and they may be the only option for very high frequency, millimeter wave and terahertz switches[11].

CONCLUSIONS

High data rate wireless networks will require the development of high performances, low cost microelectronics switches. Innovative materials like PTM and PCM can be used to build high performance RF switches, outperforming semi conductor devices figure-of-merit by at least one order of magnitude. They both have specific advantages and should find applications in the future.

MEMS have also the potential for fulfilling the requirements for high performance microwave switches. New structures, including hermetic packaging in processing, have excellent figure of merit and very small size at the same time.

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EXTREMELY WIDE TUNING RANGE OF MECHANICAL OSCILLATION OF A SILICON WAVEGUIDE DRIVEN BY OPTICAL GRADIENT FORCE

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ABSTRACT

MEMS oscillators are well-suited for filter and timing applications owing to high resonator quality factors. Active compensation of frequency variation in conventional commercial MEMS oscillators is limited by their tuning range, which is typically <1,000ppm. This paper demonstrates an extremely wide tuning range for oscillations of a silicon clamped-clamped beam via the optical spring effect. Frequency tuning from 3.1MHz to 19.4MHz is achieved by varying the intra-cavity power, corresponding to an optical-tuning coefficient of 1312.5ppm/ μ W. To the best of our knowledge, this is the largest reported tuning range in literature for MEMS oscillators.

INTRODUCTION

MEMS resonators are attractive for oscillator design on account of their extraordinary small size, high level of integration, low cost and high volume manufacturing capability. MEMS oscillators can be integrated either on the CMOS die or as two-chip solution using a separate CMOS die for interface electronics in a single package [1]. Typically high quality factors of MEMS resonators also enable superior noise performance and frequency stability compared to electrical oscillators. MEMS oscillators thus fill the gap between high-performance, non-CMOS compatible technologies on the one hand, and low-performance CMOS compatible technologies on the other.

Frequency stability is one of the most important parameters of an oscillator. Since the output of an oscillator is the frequency of oscillation, it is possible to use the frequency measurement in an active feedback loop to counter the mechanism that causes the shift in frequency. The frequency variation of MEMS oscillators is typically dominated by temperature fluctuations in the device. Compared to quartz, silicon resonators have much larger temperature coefficient of frequency (TCF) [2]. To overcome the linear TCF of MEMS resonators, recent advances [3, 4] have relied upon using oxide plugs to reduce the TCF magnitude to sub-25ppm and a quadratic temperature dependence of frequency. Achieving even lower TCF requires ovenization [5]. For commercial oscillators, tuning ranges on orders of few hundred ppm have been demonstrated [2, 4]. For voltage controlled crystal oscillators (VCXO), typical tuning coefficients are on the order of ~100ppm/V. To counter frequency variation due to manufacturing imperfections and package stresses, and long term frequency drift due to aging, having a method to achieve larger tuning range without significant added power consumption and die-space is desirable.

Contrary to the small tuning range achieved by electrostatic spring softening, the optical spring effect has been leveraged to achieve significantly larger tuning ranges in opto-mechanical resonators [6, 7]. In this paper, we demonstrate an all-optically transduced silicon opto-mechanical oscillator, and leverage the optical spring effect to show large frequency tuning range of 525% for an n=1 in-plane bending mode of a clamped-clamped silicon beam from 3.1MHz to 19.4MHz. The following section describes the theory of operation for the oscillator and the optical spring effect. The subsequent sections detail the device design, fabrication and experimental results.

THEORY OF OPERATION

Optical gradient force driven mechanical oscillations

Our group has previously demonstrated and explained the phenomenon of optical radiation pressure driven oscillations in an opto-mechanical resonator [8]. Alternatively, optical intensity gradients found in the near-field of guided wave nanostructures can be harnessed to create large gradient-like optical forces [9]. In contrast to the radiation pressure force, the gradient force depends upon the transverse evanescent-field coupling between adjacent cavity elements. One example of such a system that will be employed in this paper is a suspended optical waveguide coupled to an opto-mechanical cavity resonator. When the light wavelength is chosen such that it is detuned to an optical resonance of the resonator, the light intensity inside the cavity is enhanced due to its large optical quality factor. Thus if Pin represents the input laser power, and Q_{opt} is the optical quality factor, the intra-cavity power is denoted by $P_{cavity} = Q_{opt}P_{in}$. This results in a large field gradient between the light field in the cavity and light field in the waveguide (Pin) over a small gap.

The optical gradient force acting on the resonator results in an optical anti-damping force acting on the waveguide and the resonator. The anti-damping rate Γ_{opt} is proportional to the circulating optical power and the detuning of the laser light from the cavity resonance [10]. By adjusting both the power and the detuning, the anti-damping rate can be increased to overcome the intrinsic mechanical damping rate (b= $\Omega_m/2Q_{mech}$, where Ω_m and Q_{mech} denote the mechanical resonance frequency and quality factor respectively) in the resonator or the waveguide. This results in self-sustained mechanical oscillations of the device.

Optical spring effect

According to the derivation in [9], the mechanical spring constant of the cavity (k') depends on the input laser power (P_{in}) as follows:

$$k' = k_0 (1 + \eta_{os} P_{in}) \tag{1}$$

where k_0 is the intrinsic mechanical spring constant and η_{os} is the optical spring coefficient. The optical spring coefficient varies with the detuning as follows:

$$\eta_{os} \propto d(Q_{opt})^2 / [\Omega_0 (d^2 + 0.25)]^2, d = (\lambda_0 - \lambda_{in}) / \Delta \lambda_{LW}$$
(2)

In the equation above, d denotes a normalized representation of the detuning that is in turn dependent on the input laser wavelength (λ_{in}), the optical cavity resonant wavelength (λ_0) and the line-width of the cavity resonance ($\Delta\lambda_{LW}$). The spring coefficient is directly proportional to the optical quality factor (Q_{opt}) and inversely proportional to the intrinsic mechanical resonance frequency, suggesting that the largest tunability is noticed for low frequency mechanical modes excited with a high quality factor resonance. Oscillations occur only when the laser is blue detuned with respect to the cavity (d>0). The optical spring coefficient is thus always positive (spring stiffening), and has the highest magnitude at d \approx 0.3. Operating at this detuning results in the largest deviation from the natural mechanical resonance

978-1-940470-02-3/HH2016/\$25©2016TRF DOI 10.31438/trf.hh2016.2 Solid-State Sensors, Actuators and Microsystems Workshop Hilton Head Island, South Carolina, June 5-9, 2016 frequency for the oscillator. As the laser wavelength is increased and swept into resonance, the normalized detuning transitions from 1 to 0, and the resulting mechanical resonance shifts from a rising trend to a falling trend, crossing a local maximum at $d \approx 0.3$.

DESIGN AND FABRICATION

As noted in equation (2), the largest tuning range is observed for low frequency mechanical modes. This demonstration uses a silicon opto-mechanical resonator coupled to a suspended silicon waveguide. Since the micro-ring resonator is designed for higher frequency modes (radial breathing mode at 175MHz and compound radial mode at 1.1GHz) [11], for this experiment we focus on the waveguide modes, which are at much lower frequencies (fundamental in-plane bending mode at 3.4MHz). The device comprises of a silicon waveguide of length 30μ m and width 400nm. The waveguide is coupled to a silicon opto-mechanical resonator. Each individual ring in the coupled resonator has a width of 3.8μ m. The device thickness is 220nm. The waveguideresonator gap is <100nm. The fabrication process flow was described in detail in earlier work on multi-GHz opto-mechanical oscillations of the coupled ring resonator [11].

Fabricating the device involves a five mask process flow on a custom silicon-on-insulator (SOI) wafer (undoped 250nm device layer for low optical loss and 3µm thick buried oxide for optical isolation from the silicon substrate). The top silicon is thermally oxidized to obtain a thin oxide hard mask layer of thickness 60nm atop a 220nm thick silicon device layer. The devices are defined in the oxide mask using electron beam lithography and a CHF₃/O₂ based reactive ion etcher. The pattern is then transferred into the silicon device layer using a chlorine based reactive ion etch. The mechanical resonator, the electrical routing beams and the bondpads are doped via boron ion implantation using a second photolithography mask to protect the optical section of the device. A third mask is then used to deposit metal over the bond pads for electrical contact using lift-off lithography. The metal stack used for the bond pads comprises of 25nm Nickel, 25nm Titanium and 50nm Platinum, respectively in the order of deposition. For the experiment described in this paper, the second and third masks are not necessary. A fourth mask is used to define release window for the waveguide and opto-mechanical resonator via photolithography, followed by a timed release etch in buffered oxide etchant to undercut the devices. The samples are then dried using a critical point dryer to prevent stiction. Fig. 1 shows a scanning electron micrograph (SEM) of the fabricated device.



Figure 1: Scanning electron micrograph (SEM) of the device used for the experiments described in this manuscript.

EXPERIMENTAL RESULTS

Optical characterization

To characterize the optical transmission spectrum of the

resonator, light from a continuous wave (CW) Santec TSL-510 tunable diode laser is coupled into the on-chip waveguide through grating couplers and the transmitted power is measured using an optical power meter. The wavelength of the laser is swept to identify optical resonances of the device. Fig. 2 shows an optical transmission spectrum of such a device. At low laser power (0Bm), the optical resonances are clearly seen as dips in the optical transmission spectrum. At higher laser power (15dBm), the thermal nonlinearity of the optical resonator combined with the mechanical oscillations of the waveguide distort the optical transmission spectrum.



Figure 2: Comparison of optical output transmission spectra at 0dBm and 15dBm input laser power. Oscillations set in as the laser is swept beyond 1563nm when operating with 15dBm power, manifesting as a distorted optical transmission spectrum.

Mechanical oscillation

To probe the mechanical mode of the waveguide, the transmitted light is passed to a Newfocus 1647 Avalanche Photodiode with gain setting of 6,000V/W. The motion of the waveguide and the opto-mechanical ring resonator leads to phase modulation of the intra-cavity light, which is converted into amplitude modulation via shaping by the optical resonator transfer function [11]. The ring resonator is designed such that it does not have mechanical modes in the vicinity of the resonance frequency of the fundamental in-plane and out-of-plane bending modes of the waveguide. The RF signal is analyzed on an Agilent 5052B signal analyzer. Fig. 3 shows a sketch of the experimental setup.



Figure 3. Experimental setup used to study opto-mechanical oscillation of the silicon waveguide.

As the laser wavelength is swept into an optical resonance of the cavity, the intra-cavity optical power in the resonator builds up. When the built up power is sufficiently high such that the antidamping introduced by the optical gradient force overcomes the intrinsic mechanical damping in the silicon waveguide, mechanical oscillation of the waveguide ensues. The optical power threshold required to launch oscillations is minimized when the detuning of the laser frequency (ω_{in}) from the cavity resonance (ω_0) is equal to the mechanical resonance frequency (Ω_m) i.e. when ω_{in} - $\omega_0 = \Omega_m$. Fig. 4 below shows a comparison of the spectrum of the photodetector output for sub-threshold and above threshold operation. The Brownian noise peak of the n=3 in-plane bending mode of the waveguide is seen in the spectrum for the laser wavelength corresponding to sub-threshold operation. The tethers supporting the waveguide act as the anchors for the clamped-clamped beam. These are designed as a multi-mode interference (MMI) waveguide arm to minimize optical losses at the tether, and are not optimized for anchor losses of the mechanical mode. As such, the n=3 mode has very low mechanical quality factor of 250. The spurs seen in the spectra are on account of the piezo controllers on the mirrors of the tunable laser.



Figure 4. Comparison of Brownian noise spectrum of the n=3 inplane mode of vibration of the silicon waveguide (inset: FEM simulation) to onset of oscillations ushered in by detuning the wavelength to above-threshold operation.

Frequency tuning of mechanical oscillation

As described in the 'Theory of Operation' section above, the optical spring constant is dependent on the detuning of the laser to the optical resonance of the cavity. In our experiment, we manually sweep the laser wavelength from 1573nm to 1577.5nm in steps of 0.02nm, thereby scanning across multiple optical resonances of the cavity shown in Fig. 2. The input laser power is maintained at 14dBm. When the laser wavelength is swept, the coupling changes from one optical resonance to another. Thereby the optical spring coefficient varies and passes through the local maxima for each resonance, as seen in the rising trend in oscillation frequency followed by a falling trend as seen in Fig. 5. The n=1 in-plane bending mode is thus tuned from 3.1MHz (natural resonance frequency at d=0) to 19.4MHz, corresponding to a 525% tuning range.



Figure 5. Variation of RF oscillation power and oscillation frequency of the fundamental harmonic with laser wavelength. The laser wavelength is manually tuned from 1573nm to 1577.5nm and oscillation spectrum is recorded at every wavelength. At 1576.9nm, the oscillation frequency abruptly drops as a result of the oscillation switching to another mechanical mode. The FEM simulated mode-shapes for the fundamental n=1, n=2 and n=3 inplane bending modes of the waveguide are shown on the right.

Fig. 6 shows a comparison of three traces recorded at different wavelengths highlighting the vast tuning range. This tuning range is achieved with an input laser power of 14dBm. Accounting for the optical losses for the unoptimized grating couplers (8dB each at input and output grating couplers), this corresponds to 6dBm \approx 4mW optical power incident on the device following the input grating coupler. The observed tuning coefficient is thus 525*1e4ppm/4mW \approx 1312.5ppm/ μ W.



Figure 6. Comparison of RF oscillation spectra at three different wavelengths highlighting the wide tuning range enabled by the optical gradient force. Presence of oscillation harmonics as noticed in this plot, is inherent to non-linear modulation in optomechanical oscillators [8, 13].

As the laser wavelength is varied to 1576.9nm, the oscillation frequency shows a sharp transition to the n=2 in-plane bending mode as shown in Fig. 5. This phenomenon is similar to the transition from 'buckled up' to 'buckled down' mode of operation in nano-mechanical resonators due to opto-mechanical interactions described in [12]. As the laser wavelength is increased further beyond 1577nm, the oscillations show another sharp transition to the higher frequency n=3 mode.

Non-linear modulation of the laser light on account of the mechanical motion of the resonator results in generation of oscillation harmonics in opto-mechanical oscillators, as previously noted by us and other groups [8, 13]. The wide tuning range is also noticed for these harmonics. The intensity map shown in Fig. 7 illustrates this observation by plotting out the measured RF power at all frequencies from 100kHz to 50MHz as the input laser wavelength is manually varied. The bright regions in this plot correspond to the oscillation signals. The frequency range of tuning is naturally twice as large for the second harmonic as compared to the fundamental oscillation signal for the oscillating mode, as the percentage variation in frequency for all harmonics is constant.



Figure 7. Contour plot showing tuning range for fundamental and higher order harmonics with variation of laser wavelength (Yaxis). The color map represents the RF signal power at every frequency point in the spectrum. The spectrum is recorded from 100kHz to 50MHz (X-axis).

CONCLUSION AND FUTURE WORK

The optical spring effect demonstrated in this paper can be used as a powerful tool to tune the frequency of mechanical oscillators. The tuning coefficient of 1312.5ppm/ μ W for the optical spring in this work is significantly larger than ~100ppm/V typically achieved by VCXOs. The silicon beam oscillator chosen here is not an ideal choice for reasons well known to the MEMS oscillator community on account of its low effective mass. For low phase noise and frequency stability, choosing a resonator with a high mechanical quality factor and large effective mass is important as it directly translates to low phase noise and low jitter and hence improved frequency stability. Future work could explore adopting this scheme to an opto-mechanical resonator with high mechanical and optical quality factor with an active laser wavelength control loop to demonstrate autonomous sub-ppm operation.

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ELECTRICAL AND OPTICAL TRANSDUCTION OF SINGLE-CRYSTAL 3C-SiC COMB-DRIVE RESONATORS IN A SIC-ON-INSULATOR (SICOI) TECHNOLOGY

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ABSTRACT

We report on a study of electrical and optical transduction of single-crystal 3C-SiC comb-drive lateral resonators. These SiC resonators were surface micromachined from high-quality single-crystal 3C-SiC films on SiC-on-insulator (SiCOI) substrates made using a bonding-free transfer technique previously reported by our team. We have measured multiple modes in such resonators using all-electrical transduction with capacitive actuation and detection, as well as optical readout. We have observed the onset of nonlinearity for optical displacement transduction in the 'knife-edge' measurement scheme, which helps to precisely calibrate the vibrational amplitude of each mode. These devices, along with such signal transduction schemes, shall enable new multimode and integrated resonant sensors from single crystalline 3C-SiC.

INTRODUCTION

Silicon carbide (SiC) is a technologically important material owing to its exceptional electrical, optical, thermal, and mechanical properties. SiC is a wide bandgap polymorphic semiconductor ($E_g \approx 2.36 - 3.23 \text{ eV}$, depending on the crystalline polytype) especially useful for power electronics and hightemperature devices. It is also a natural near-zero-epsilon material at ~10.3µm [1], which offers promises for useful optical and photonic devices. Besides being chemically inert, radiation hard, and high temperature capable, it lends itself to bulk and surface micro/nanomachining [2,3], allowing for the fabrication of robust and versatile SiC micro/nanoelectromechanical systems (MEMS/ NEMS). Furthermore, its excellent mechanical properties (e.g., Young's modulus, E_{Y} ~450GPa) make it attractive for enabling resonant MEMS/NENS operating at radio frequencies (RF). While over two hundred different polytypes of SiC have been identified, only 4H-, 6H-, and 3C-SiC are available in single crystalline form and have established manufacturing protocols for large wafers.

It is much desirable to develop single-crystal SiC-on-insulator (SiCOI) technologies for emerging applications in electronics, transducers, and photonics. However, such developments have been particularly challenging because the most widely used SiC single crystals, *i.e.*, 4H-SiC and 6H-SiC, come in bulk substrates instead of thin films; and their growth or deposition on insulators (such as SiO₂) has been elusive. To date, only polycrystalline 3C-SiCOI wafers are available with high-yield growth [2,3]. Mainstream techniques for realizing single-crystal SiC-on-insulator, such as wafer bonding or 'Smart-Cut', suffer from low-yield bonding and transfer of the single-crystal SiC thin film to the insulating layer, and high-cost ion implantation processes [4,5].

We have regularly reported progresses in surface micro/ nanomachined polycrystalline SiC MEMS/NEMS devices since the early 2000's. In comparison, our studies on surface micro/ nanomachined single-crystal SiC devices have been far fewer. In 2003, we described a method to produce single crystalline 3C-SiC on insulator substrates using a transfer technique that involves growing a polycrystalline silicon (poly-Si) layer to a wafer-scale thickness by CVD on top of an oxide capped 3C-SiC film followed by etch back of the single crystal handle wafer used for 3C-SiC epitaxy [6]. This bonding-free method was used to produce 100 mm-diameter 3C-SiCOI substrates with 100% transfer yield. To demonstrate their utility in surface micromachining, comb-drive lateral resonators were fabricated from these substrates and characterized for resonant behavior. In this work we revisit the comb-drive lateral resonators fabricated from single crystalline 3C-SiCOI. Specifically, we study their resonance characteristics using both electrical and optical transduction techniques.

RESULTS AND DISCUSSIONS

The devices in this work are folded-beam lateral comb-drive MEMS resonators. The thicknesses of the 3C-SiC and thermallygrown SiO₂ layers are 1.5 μ m and 1.7 μ m, respectively. Figure 1 briefly illustrates the fabrication process. An Al etch mask is sputtered onto the surface of the 3C-SiCOI wafer and patterned using standard lithographic techniques. Subsequently, the 3C-SiC layer is etched using an RIE process at 3 Torr and 150 W. The chemistry recipe used includes O₂, CHF₃, and He with flow rates of 22.5, 5.6, and 55 sccm, respectively. The etch process is anisotropic, but not selective to the Si-containing layers, thus etching not only the SiC layer, but also the SiO₂ layer and ~5 μ m of the polysilicon substrate. After etching, the Al etch mask is removed with an Al wet etchant, and the devices are released with a 5 min HF etch of the buried oxide layer. Figure 2 shows the images and SEM images of representative device designs.



Figure 1: The 3C-SiCOI comb-drive resonator fabrication process. (a) The single-crystal 3C-SiCOI sample. (b) The Al etch mask is patterned by standard lithographic techniques. (c) SiC is etched using RIE. (d) Removal of Al etch mask. (e) The comb-drive resonator is released via a timed wet etch of the buried oxide layer.

Resonance behaviors of the 3C-SiC comb-drive resonators are characterized using a motion detection apparatus which enables the measurement of device resonances with both optical and electrical transduction schemes. That is, it allows for measurements that involve (i) electrical actuation with electrical detection, and (ii) electrical actuation with optical detection of multimode in-plane and out-of-plane responses, simultaneously, via interferometric readout and 'knife-edge' techniques [7]. All measurements are performed in moderate vacuum (~20mTorr) and at ~300K.

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Figure 2: Fabricated comb-drive resonators based on 3C-SiCOI. (a)-(b) Optical images. (c)-(e) SEM images. (f) Side view SEM image of fabricated 3C-SiCOI electrical bonding pads, clearly showing the SiC, SiO₂, and Si layers. Scale bars: 300 μ m in panels (a)-(c), 40 μ m in panels (d)-(e), and 5 μ m in panel (f).



Figure 3: Simplified illustration of the combined optical and electrical resonance detection system. A network analyzer is used to capacitively drive the resonator. Resonances are detected using both optical and electrical transductions. The shuttle is polarized by a DC power supply. A low-noise voltage preamplifier with 40dB gain is employed in the electrical detection scheme.

In the optical motion transduction scheme, we employ a customized home-built detection system (see Fig. 3). It can detect both out-of-plane and in-plane resonant motions through optical interferometry and the 'knife-edge' effects, respectively.

To attain the optical detection, a 633nm laser is focused on devices using a 50× microscope objective. The typical on-device spot size in the system is \sim 1µm. To efficiently transduce in-plane motion using the 'knife-edge' technique, the laser spot is placed on the boundary of the comb-drive device structural features such that

~50% of the laser spot area is on the device, to achieve the highest responsivity from device motion to optical modulation [7]. Resonances are electrostatically excited in the devices by applying an AC voltage to the comb finger pads. Given the direction of the capacitive forces, in-plane resonance modes are efficiently excited.



Figure 4: Measured multimode responses of the comb-drive resonator with electrical actuation and sensing. (a) The broadband response of the comb-drive resonator with 3 electrically actuated modes. (b)-(d) The 3 resonance modes, measured when V_{DC} is fixed at 20V and the P_{drv} is varied from -20dBm to 10dBm.

In the electrical transduction scheme, resonances of the devices are electrically detected by a simple two-port transmission measurement. The AC signal is applied to the comb fingers using a network analyzer, and the output signal from the comb-drive resonator is amplified using a low-noise voltage preamplifier with a gain of 40dB. In addition, a polarization DC voltage is applied to the comb-drive shuttle to investigate the electrical tunability of the resonance frequency of the comb-drive resonator.

Figure 4a shows the wide-range response of the simple twoport transmission measurement, where three resonances are readily observed. These three modes are at $f_1 \approx 23.95$ kHz, $f_2 \approx 37.8$ kHz, and $f_3 \approx 46.5$ kHz, with quality factors (*Q*s) of $Q_1 \approx 650$, $Q_2 \approx 260$, and $Q_3 \approx 220$, respectively. The detection of multiple modes provides new opportunities for possible applications, such as multimode oscillators and integrated multimode resonant physical sensors. For each mode, we fix V_{DC} at 20V and gradually increase the driving power from -20dBm to 10dBm on the output port of the network analyzer. As shown in Fig. 4b to 4d, the amplitude of the resonance is greatly increased, and the resonance frequencies of all three modes are shifted toward lower frequencies with an increase in driving power. In addition, the first resonance mode, f_1 , exhibits a clear mechanical nonlinearity at a driving power of $P_{drv}=10$ dBm.

Figure 5 shows the electrical frequency tuning effects by

varying the $V_{\rm DC}$ (polarization voltage applied on the comb-drive shuttle). The DC voltage $V_{\rm DC}$ is swept from 5V to 27.5V for each mode and the response of the resonance is measured (see Fig. 5). For the first mode, f_1 shifts from 24.06 kHz to 23.72 kHz as $V_{\rm DC}$ increases from 5V to 27.5V, exhibiting capacitive softening of the resonance frequency. In contrast, resonance frequencies of the 2nd and 3rd modes both upshift as $V_{\rm DC}$ increases, showing stiffening of their resonance frequencies.



Figure 5: Measured frequency tuning of 3 modes. (a)-(c) Frequency tenability as V_{DC} is varied from 0V to 27.5V.



Figure 6: Electrically actuated comb-drive resonator with shorter folded-beam arms. (a) SEM images of the comb-drive resonator. (b) Measured resonance, with electrical readout, as the driving signal, P_{drav} is varied from -20dBm to 10dBm, at 20V V_{DC} .

We also investigate resonance characteristics in comb-drive resonators with folded-beam supporting arms shorter relative to the device shown in Fig. 3. We measure resonances with different driving strength from -20dBm to 10dBm at V_{DC} =20V; and we find resonance frequency of $f_1\approx15.10$ MHz with $Q\approx500$.

In optical detection, we are able to measure resonances up to the 5^{th} mode (see Fig. 7a). Optical detection is sensitive to both inplane and out-of-plane displacements. As such, the new modes at 25.18kHz and 57.53kHz may likely be attributed to out-of-plane resonances. We test frequency tunability in the first three modes.

As with the electrical transduction scheme, we are able to optically transduce both frequency softening (Fig. 7b & 7c) and frequency stiffening (Fig. 7d) behaviors as the DC voltage increases. Further, resonance amplitudes change with V_{DC} . We find the highest resonance responses for the first three modes take place when V_{DC} is between 15V and 20V. The resonance responses decrease and then increase as V_{DC} is swept from 22.5V to 27.5V, for both the 2nd and 3rd modes. This can be explained by the 'knife-edge' effect. At low V_{DC} , static displacement of the device is small, allowing the device to operate within the constant responsivity regime [7]. While under high DC polarization, a large static displacement changes the laser spot position relative to the device, causing the device to move outside the constant responsivity regime, hence a largely modified resonance response.



Figure 7: Measured resonances with optical readout. (a) The wide range spectrum. (b)-(d) Frequency tuning with $V_{DC}=0V$ to 27.5V.



Figure 8: Electrically actuated resonances with optical readout, as P_{drv} is varied from -20 dBm to 10 dBm with a step of 5 dBm. $V_{DC}=20V$. (a)-(c) Measured data from the first 3 modes.

Next, we fix the DC polarization voltage at V_{DC} =20V and gradually increase the driving force. Figure 8 shows the three optically detected resonance modes from the comb-drive resonator. Similar to the electrical transduction results, intensities of the three resonances increase as the driving strength increases. The 1st and the 3rd modes in the optical measurement exhibit mechanical nonlinearity when the driving strength is higher than 5dBm, showing high power handling of these comb-drive resonators [8].

With capacitive actuation and optical readout, we observe the optical readout nonlinearity as the driving power increases: once the amplitude of resonance exceeds 600μ V, intensity of the signal

decreases as the driving strength continues to increase. This is because when the displacement of the comb-drive resonator is small and within the radius of the laser spot, displacement linearly transduces into optical modulation based on the 'knife-edge' effects integrated in the interferometry; but once the displacement amplitude goes beyond the linear responsivity regime, the mechanical motion to optical signal transduction gain changes. In this latter case, the responsivity (i.e., transduction gain) from mechanical to optical transduction becomes smaller, and eventually takes on the opposite sign, thus decreasing the efficiency and gain of signal transduction. In our measured results, amplitudes of resonances show saturation near 600µV (this corresponds to a peak resonance displacement amplitude equal to the laser spot radius) which occurs at $P_{drv} = 0$ dBm to 5dBm input driving power levels. Beyond this saturation point, higher mechanical displacement decreases resonance response due to negative responsivity, leading to optical readout nonlinearity.



Figure 9: Illustration and analysis of transduction responsivity in the optical measurement scheme. (a)-(b) Schematic illustrations of in-plane and out-of-plane shuttle motions. (c)-(d) Laser spot positions for in-plane and out-of-plane motion detection. (e)-(f) Calculated responsivity of in-plane and out-of-plane motions.

To further investigate the nonlinearity of signal transduction in optical detection, we compute responsivity from the mechanical motion to optical signal transduction [3] (see Fig. 9) based on the actual come-drive device structures. For in-plane motions, we use the ~1 μ m laser spot size and it is focused on the beams that comprise the inner structures of the shuttle (see Fig. 9c). For outof-plane displacement, we consider that laser spot is focused on a flat area of the shuttle (see Fig. 9d) as SiC is transparent. We find optical responsivity changes when the device oscillates with large displacements, and it takes on a different sign (*e.g.*, negative to positive value) when device motions are larger than ~1 μ m or ~60nm for in-plane or out-of-plane motions, respectively. The responsivity variation leads to saturation of measured resonances shown in Fig. 8. Our calculations can enable direct conversion from measured signal to mechanical displacement for both in-plane and out-of-plane resonant motions.

CONCLUSION

In summary, using the single crystalline 3C-SiC-on-insulator technology, we have demonstrated SiC comb-drive resonators that could not be realized by conventional monolithic approaches, and investigated their resonance characteristics using a simultaneous optical and electrical displacement detection system. We have observed multimode resonances, nonlinearity in optical signal transduction, and frequency tunability. In addition, using optical detection nonlinearities arising from 'knife edge' interferometric effects, we have computed responsivity in the measurement.

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AUTONOMOUS OSCILLATIONS WITHOUT EXTERNAL POWER SUPPLY IN A MEMS OSCILLATOR THROUGH INTERNAL RESONANT MODE COUPLING

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ABSTRACT

Oscillators are systems that produce stable periodic signals without external frequency reference. A typical oscillator consists of a resonator that determines the output frequency, and an external sustaining feedback scheme to replenish the energy loss due to the dissipation from the resonator. Here we demonstrate that, by driving the constituent resonator to the internal resonance (IR) condition, where two different vibrational modes of the same resonator are coupled commensurately, there is efficient coherent energy exchange between the two modes. This strong coupling allows the observed mode to draw stored energy from the coupled mode, hence maintaining both its oscillation amplitude and frequency without an external power supply.

INTRODUCTION

Self-sustained oscillators that autonomously create prescribed frequencies are critical in modern technology, with applications such as time keeping, communication and navigation [1]. A typical oscillator consists of a mechanical resonator that determines the output frequency of the oscillator and a sustaining feedback element. During the oscillations, the mechanical resonators inevitably dissipate energy to the environment, through mechanisms such as viscous damping, clamping losses, and thermoelastic dissipations. In order to compensate such losses and maintain the vibrational amplitude, the sustaining element draws energy from external sources and supplies the energy to the resonator. Examples of sustaining elements include wound spring and electronic amplifiers. At steady state, the energy influx equals the energy dissipation, hence satisfying the well-known Barkhausen criterion, and the oscillator produces a stable, continuous signal.

In this classic design of oscillators, the mechanical resonator is operated in the linear regime, in which the resonant frequency fis independent of the vibrational amplitude, A. However, as the critical dimensions of the resonator shrink toward micro- and nano-meter scales as in the cases of microand nano-electromechanical systems (MEMS and NEMS), the size of the linear regime also decreases accordingly. For example, the critical vibrational amplitude of a doubly clamped resonator scales as $L/Q^{0.5}$, where L is the resonator length and \hat{Q} is the quality factor [2]. When the oscillation amplitude is above this critical value, (also known as onset of nonlinearity), the resonant frequency becomes dependent on the vibrational amplitude, known as the A-f effect [3]. Since the onset of nonlinearity diminishes for MEMS and NEMS, small levels of excitation, or even mere thermomechanical noise can put the resonators into the nonlinear regime [4]. Recent investigations of nonlinear MEMS and NEMS oscillators have demonstrated improved frequency stability [5], enhanced synchronization range [6], and superior performance in energy harvesting applications [7].

In this study, we exploit the A-f effect in a high-Q silicon MEMS oscillator to invoke internal resonance (IR), where the resonant frequency of the in-plane mode is tuned to match one-third of the resonant frequency of its own torsional mode. Previously we have shown reduced phase noise [8] of the oscillator at IR; here we explore the dynamical response at IR, and find

anomalous ring-down behavior of the in-plane mode where it keeps oscillating without external energy supply for as long as 227 milliseconds. As a practical demonstration of this discovery, we show continuous operation of the oscillator when the external sustaining circuit is only engaged intermittently.

DESCRIPTION OF INTERNAL RESONANCE (IR)

For a nonlinear, *N*-degree-of-freedom system, in which each degree of freedom represents a vibrational mode with a unique linear resonant frequency, important cases emerge as two or more resonant frequencies are commensurable or near commensurable. Internal resonance describes the situation when such commensurable relationship agrees with the order of nonlinearity of the system [9]. The hallmark of IR is that, if energy is initially imparted to only one mode, there will be continuously energy exchange among all the modes involved in the IR, while all the modes will also dissipate energy to the environment due to their respective damping.

Let us consider a system that consists of 2 modes (x and y) as follows:

$$\ddot{x} + Q^{-1}\dot{x} + x + \beta x^3 = f_d \cos(\omega_d t) + J_y y$$

$$\ddot{y} + q^{-1}y + \Omega_y^2 y = J_x x$$
(1)

Here Q and q are the quality factors (inverse of dissipations) of each mode, respectively. The linear resonant frequencies of mode x and y are 1 and Ω_y . We only consider cubic nonlinearity in mode x (Duffing-type), with corresponding nonlinear coefficient of β . We also only consider linear couplings between the two modes, with strength of J_y and J_x . Additionally, only mode x is being driven externally, with forcing amplitude of f_d and frequency of ω_d ; therefore, external energy is only injected to mode x. If there is no inter-mode coupling ($J_y = J_x = 0$), then mode x is a simple driven nonlinear resonator, and mode y is a damped, undriven linear resonator.

Since the order of nonlinearity in this system is 3, IR happens if $\Omega_y \approx 1/3$ or 3. However, due to the nonlinear nature of mode x, namely, the *A*-*f* effect, the resonant frequency of mode x can be greater than 1 for positive β and less than 1 for negative β . Therefore, it is possible to encounter the IR condition for values of Ω_y different from 1/3 or 3. For exmple, if β is positive, the *A*-*f* effect will result in the resonant frequency of mode x being larger than 1, hence IR can occur for $\Omega_y > 3$ as long as Ω_y equals three times the nonlinear resonant frequency of mode x. To demonstrate the possibility of IR under such a condition, we perform long-time numerical integration of equation (1), with Q = q = 50, $\beta = 1$, $\Omega_y =$ 3.3, and $J_y = 2$, $J_x = 1$. The simulated results are shown in Fig. 1, for different values of f_d and ω_d . For each simulation, the value of f_d is fixed, and the value of ω_d is swept upward.

The steady state amplitude of mode x, shown in Fig. 1a, displays the normal frequency tuning effect for small f_d , where the resonant frequency ω_x – at which point the amplitude reaches maximum value – increases with f_d . However, for intermediate values of f_d , we find that ω_x becomes less dependent on f_d and stays around 1.1, which is 1/3 of Ω_y . Therefore, we conclude that IR is reached, and the surpressed frequency tuning is one of its characteristics. The surpressed frequency tuning can be understood
as follows: when more energy is imparted to mode x (as one increases the excitation amplitude f_d), this energy is transferred to mode y through IR, hence the amplitude (and frequency) of mode xremains nearly constant. The relation between ω_x and f_d is better presented in Fig. 2, where we clearly observe a plateau in ω_x . At IR, we also find a sudden increase in the amplitude (hence kinetic energy) of mode y, shown in Fig. 1b. Since mode y can only obtain energy through coupling to mode x, such an abrupt increase in mode y's energy illustrates the enhanced energy transfer between the two modes at IR. For large f_d , which pushes ω_x above 1.1, the normal frequency tuning resumes, and the IR condition is no longer active.



Figure 1: Steady state amplitudes of mode x and mode y, obtained from long-time numerical integrations of equation (1). The forcing amplitude f_d is increased from 0.001 to 0.051, with 0.002 step size. The linear resonant frequency for mode x is downshifted from 1 to 0.9, due to the coupling to mode y.



Figure 2: Resonant frequency of mode x, ω_x , as a function of forcing amplitude f_d . Data is extracted from the simulation results shown in Fig. 1a. The resonant frequency ω_x is defined where the amplitude of mode x reaches its maximum value.

EXPERIMENTAL EVIDENCE OF IR

We use single crystalline, doubly clamped silicon MEMS resonators to experimentally investigate the characteristics of IR. The devices are fabricated from the SOIMUMPS process from MEMSCAP. A typical device is shown in the inset of Fig. 3. The MEMS resonator is excited electrostatically with interdigitated comb electrodes, and the mechanical motion is transduced electrostatically with another set of comb electrodes. We use COVENTOR to determine the mode shapes and frequencies of the first three vibrational modes, as summarized in Table 1. The discrepancy between the simulated and measured resonant frequency most likely results from variations in the fabrication process.

Table 1: Simulated and measured linear resonant frequency for the first three vibration modes. The linear resonances for the out-of-plane and the torsional mode are measured with an optical interferometer.

Mode shape	Simulated frequency (kHz)	Measured frequency (kHz)	
In-plane	55.734	61.570	
Out-of-plane	179.071	159.832	
Torsional	201.082	194.636	



Figure 3: Vibrational amplitude of the in-plane mode of the MEMS resonator with different ac excitation. The applied DC bias voltage is 7 V. The amplitude is measured with a lock-in amplifier (Zurich Instrument HF2LI). Inset: Scanning Electron microscopic image of the doubly clamped silicon beam.

The mode of interest (mode x) here is the in-plane vibrational mode, with linear frequency of 61.570 kHz and quality factor of 120,000, measured in vacuum. The high quality factor results in an onset of nonlinearity of about 10 nm (Fig. 3), occurring with small alternative current (ac) excitation just below 100 μ V. This small onset of nonlinearity allows for large resonant frequency tuning through the *A*-*f* effect when the drive voltage is increased.

In order to identify the presence of IR in our MEMS resonator, we turn to closed-loop, oscillator-like schematics, where the MEMS resonator serves as the frequency determining element. This is equivalent to the situation of replacing the external excitation term $f_d \cos(\omega_d t)$ in equation (1) with a feedback excitation term. Specifically, we employ two types of oscillator architectures. In design A (see Fig. 4), the feedback loop includes

electronic amplifiers and a phase-locked loop (PLL), and the amplitude of the feedback excitation is controlled by the output of the PLL. In this configuration, as we increase the forcing amplitude, we find that the resonant frequency shifts up steadily from 61.6 kHz, before "accumulating" around 64.9 kHz, which coincides with 1/3 of the resonant frequency (194.6 kHz) of the torsional mode (mode y). Therefore, the IR between the in-plane mode and torsional mode is clearly observed. The linear resonant frequency of the torsional mode is measured separately with an optical interferometer. In design B (see Fig. 5), we replace the PLL with a phase shifter, and the stable oscillations are achieved by the A-f effect [11]. In either case, we have observed IR manifested itself within a wide range of control parameters (driving force for design A and phase-delay for design B). For design circuit B, there is a regime where multiple oscillation frequencies exist for the same phase-delay Δ (for Δ around 40°), this is due to the multi-value nature of the nonlinear resonator [9]. We will use design B for the rest of the characterizations because it has simpler closed loop components.



Figure 4: Oscillator design A, in which the feedback force is controlled by the output amplitude of the PLL. The IR persists for a wide range of driving amplitudes. Note that the x-axis is in logarithm scale.



Figure 5: Oscillator design B, in which the feedback loop consists of electronic amplifiers and phase-shifter. The oscillation frequency is controlled by the phase-delay Δ introduced by the phase shifter. The IR is characterized as the regime where the oscillation frequency is insensitive to Δ .

DYNAMICAL RESPONSE AT IR

In order to study the dynamical behavior of IR, especially the energy exchange between the two modes, we turn to explore the temporal response at the IR condition. We perform ring-down measurement on the in-plane mode to examine its dynamical behavior. After the MEMS oscillator reaches steady state, we remove the feedback excitation by disengaging the switch S, and record the transient revolution of its amplitude with a digital (Tektronix, DPO5204, oscilloscope sampling rate of 2 MSample/second). If the oscillator is positioned outside IR (point 1 in Fig. 5), the vibrational amplitude decays immediately after the external excitation is removed (Fig. 6), with a decay rate dictated by the quality factor. In the case of nonlinear oscillators, the instantaneous frequency will also decay toward the linear resonance due to the A-f effect [10].



Figure 6. Ring-down responses of the nonlinear oscillator outside IR. (a) Measured temporal evolution of the oscillation amplitude after the excitation is removed. (b) Temporal frequency response of the oscillation. The power spectrum at each nominal time instance t_i is obtained by performing non-overlapping fast Fourier transform (FFT) of the time domain data (a) in a narrow window around t_i .



Figure 7. Ring-down responses of the nonlinear oscillator at IR. (a) Measured temporal evolution of the oscillation amplitude after the excitation is removed, showing a period of 227 milliseconds where the amplitude does not decay. (b) Temporal frequency response of the oscillation, where the oscillation frequency stays at 64.9 kHz during the first 227 milliseconds, before expected exponential decay towards linear resonance.

However, when the oscillator is operating at the IR condition (point 2 in Fig. 5), the vibrational amplitude does not decay for as long as 227 milliseconds (Fig. 7a), after which the amplitude starts to decrease as in previous ring-down experiments. Moreover, during this period of time, the oscillation frequency stays constant and stabilized at 64.9 kHz (Fig. 7b). The origin of this unexpected behavior of nearly constant amplitude and stabilized frequency demonstrate the presence of the coherent energy transfer at IR: at steady state (before ring-down), extra mechanical energy is transferred to and stored in the torsional mode through IR. Once the external power supply is removed as the ring-down occurs, the in-plane mode starts to receive energy from the torsional mode, thus compensating the energy loss due to its own energy dissipation. Once the energy of the torsional mode is depleted, the in-plane mode will begin normal ring-down as a nonlinear oscillator.



Figure 8. Continuous oscillations with a 50% duty cycle. (a) The duty cycle profile that controls the switch S. The cycle period is 120 milliseconds. (b) Measured oscillation amplitude, where the slight jumps during the switching events are caused by the impedance mismatch of the RF switch used to control the feedback loop. The inset shows zoomed-in oscillation around t = 0 s, where no sign of discontinuity is observed. (c) Temporal frequency spectrum during the cycles.

During this 227 millisecond period, the MEMS oscillator runs solely on the energy stored in the coupled mechanical mode, and continues to operate as when it was in the closed loop. Therefore it allows the external sustaining circuitry to be switched off intermittently. As a demonstration, we apply an on/off signal with a 50% duty cycle and a period time of 120 milliseconds to the switch that controls the feedback loop (S in Fig. 5), and record the temporal response of the oscillator, as shown in Fig. 8. Over the period of more than 2 seconds that is being recorded, the vibrational amplitude doesn't show any sign of decay during the "off" period. Furthermore, there is no discontinuity in the waveform between the "on/off" events, shown as the inset of Fig. 8b. The slight jumps of the overall envelop of the vibrational amplitude during the switching events are caused by the impedance mismatch of the switch S. As a direct result of the constant amplitude, the oscillation frequency during the whole period also stavs constant (Fig. 8c). This observation highlights the advantage of the IR for practical applications: not only can it improve the frequency stability of the oscillator [5], the usage of pulse-like patterns will also enable new power-efficient protocol for MEMS oscillators.

CONCLUSION

We provide the first demonstration of an autonomous oscillator that can be operated without an external power supply while keeping a finite amplitude and stabilized frequency. We use a nonlinear silicon MEMS oscillator that is driven to the condition of IR where there is strong mutual energy transfer between the coupled modes (in-plane mode and torsional mode). By characterizing the ring-down responses of the in-plane mode outside and inside IR, we show that at the IR condition, the coherent energy transfer from the torsional mode to the in-plane mode is able to keep the latter oscillating with constant amplitude and stabilized frequency for as long as 227 milliseconds. These autonomous oscillators show potential to be used as timing references for low-power applications or in extreme environments with limited power supply. Our findings therefore enable a new paradigm for harnessing nonlinearities in MEMS and NEMS.

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ARRAY OF MICROMECHANICAL MASS SENSORS ENABLES HIGH-THROUGHPUT SINGLE-CELL GROWTH-RATE MEASUREMENTS

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ABSTRACT

Technologies that rapidly assess cell growth would be transformative for many applications, including drug susceptibility testing for pathogens and cancer cells. Resonant micromechanical mass sensors can precisely measure growth, but are limited to measuring one cell at a time. Here, we demonstrate an approach to precisely and rapidly measure growth rates of many individual cells simultaneously. We flow cells in suspension through a microfluidic channel with multiple resonant mass sensors distributed along its length, weighing each cell repeatedly over the small fraction of a cell cycle it spends traversing this channel. Because multiple cells traverse this channel at the same time, we obtain growth rates up to 60 cells/h, while retaining a resolution of 0.2 pg/h for mammalian cells. Our system reveals subpopulations of cells with divergent growth kinetics and enables assessing cellular responses to antibiotics or antimicrobial peptides within minutes.

INTRODUCTION

Single cells are capable of remarkable variability across a range of behaviors, of which variation in growth is perhaps one of the most fundamental and important forms. From bacteria to unicellular eukaryotes to metazoan cells, even genetically identical cells may grow at drastically different rates, due to a combination of intrinsic molecular noise and various deterministic behavioral programs. This variation is typically not observable via populationbased growth assays, yet it has important consequences for human health.

Despite its importance, precisely and rapidly quantifying single-cell growth remains a technical hurdle. One approach is to measure a cell's outline in a microscopic image and then calculate its volume based on assumptions about its three-dimensional shape [1]. Another approach known as quantitative phase microscopy can be used to estimate a cell's dry mass over time [2]. Recently, inertial methods for measuring single-cell growth have been developed based on resonating micromechanical structures [3] that provide much higher precision than methods based on microscope imaging but are hindered by low throughput. Inertial methods exploit the fact that a micromechanical resonator's natural frequency depends on its mass. Adding cells to a resonator alters the resonator's mass and causes a measurable change in resonant frequency. One such class of resonator mass sensors is suspended microchannel resonators (SMRs), which consist of a sealed microfluidic channel that runs through the interior of a cantilever resonator. SMRs work by measuring the resonant frequency of a micro-cantilever suspended in vacuum, which is extremely sensitive to changes in mass. A feedback loop keeps the cantilever oscillating at its resonant frequency, while cells flow through a Ushaped microfluidic channel running the length of the cantilever. As a cell passes through the cantilever, the cantilever mass is transiently changed by the cell's buoyant mass (cell mass minus mass of the fluid it displaces), inducing a brief detectable change in the oscillation frequency. In water, a cell's buoyant mass is roughly proportional to (and is typically about one-quarter of) its dry mass [4]. SMRs are extremely precise, weighing single



Figure 1: Independently and simultaneously oscillating a large array of mechanical resonators is challenging. A phase-locked loop (PLL) array implemented on a FPGA platform (bottom) can control individual resonators, if their resonances are separated in the frequency domain (top). This scalable approach requires carefully engineered resonator-PLL dynamics for the highest precision and minimum crosstalk.

mammalian cells with a resolution of 0.05 pg (0.1% of a cell's buoyant mass) or better [3]. By flowing a cell back and forth through the cantilever repeatedly, SMRs can be used to measure a cell's growth rate, but are limited to measuring one cell at a time [5]. This limitation in throughput has prevented the wider application of SMRs across a range of biological and clinical efforts.

Here we introduce an SMR-based technique for highthroughput growth rate measurements that retains the SMR's mass and growth rate precision. We use an array of SMRs fluidically connected in series and separated by "delay" channels between each cantilever. These delay channels give the cell time to grow as it flows between cantilevers. After a cell exits a cantilever, other cells are free to enter it and be weighed. As a result, we are not limited to flowing only one cell through the array at a time, but can have many cells flowing in a queue. This enables high-throughput precision growth measurements across a wide array of suspended cell types.

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Figure 2: Linear phase-domain model of resonator-PLL system [6]. The PLL and the resonator are modelled at the top and the bottom boxes, respectively. We introduced additional poles at τ_k in the PLL model for achieving an overall n^{th} order Butterworth type response (Table 1). No additional pole is required for a first-order response. τ in the resonator model is equal to $2Q/\omega_0$, where ω_0 and Q are the resonant frequency and quality factor of the corresponding resonance.

ARRAY OF MECHANICAL RESONATORS

In order to retain the extreme precision of a single resonator in an array, each element must be oscillated independently. However it is challenging to isolate individual detection and actuation signal paths of the resonators due to fabrication and control complexities. To solve this problem, we design resonators with unique resonant frequencies (Figure 1, top, solid) and use a single physical actuation and a single physical detection channel. Therefore we can still independently actuate and detect each resonator individually (frequency division multiplexing) without the need of dedicated signal paths. We use an array of phaselocked loops (PLL) on an FPGA platform (Figure 1, bottom), where each PLL is assigned to control a single resonance [6]. We engineered the parameters of each PLL such that *i*. There is minimal crosstalk between the resonator channels and *ii*. Each PLL tracks the frequency of the corresponding resonator as closely as possible.

For the highest precision, we required resonator-PLL closedloop system for each mode has a transfer function identical to a Butterworth-type low-pass filter with controllable bandwidth. Therefore, we first derived the phase-domain transfer function of a harmonic oscillator around its resonant frequency [6] and used it to calculate the transfer function of a generic resonator-PLL system (Figure 2). Then, we showed by setting the PLL parameters (proportional gain, k_p , integral gain, k_i and additional poles, τ_k) using Table 1 that the resonator-PLL system simplifies to a Butterworth filter with desired bandwidth, β and order, *n*. We used these findings on an array of PLLs implemented on a FPGA platform.

<u>*Table 1:*</u> PLL parameters for n^{th} order Butterworth-type response with bandwidth, β .

\boldsymbol{n}	k_p	k_i	$ au_1$	$ au_2$	Additional Poles
1	β	k_p/ au			0
2	$\beta/\sqrt{2}$	k_p/ au	$\sqrt{2}/2\beta$		1
3	$\beta/2$	k_p/ au	$(1\pm j)/2eta$	$(1\mp j)/2\beta$	2



Figure 3: Schematic of the SMR array chip (A) along with an dark-field optical micrograph of the array of 12 cantilevers (inset) each with a unique resonant frequency (B). Each element in the PLL array is engineered such that the pass-bands of the resonator-PLL loops are flat, with a user-defined bandwidth (C).

SERIAL MICROCHANNEL RESONATOR ARRAYS

We utilized the PLL array approach described in Figure 1 for oscillating twelve SMRs (Figure 3A) with uniformly-spaced resonant frequencies (Figure 3B). The frequencies and quality factors of each resonator in the array were used to set the corresponding PLL coefficients using Table 1 in order to achieve a first order response from the resonator-PLL control loops. Each PLL is configured such that it will track its cantilever's oscillation frequency with a bandwidth of 100 Hz (Figure 3C).

To simultaneously measure the vibrations of all cantilevers in the array, we employed an optical lever setup in which all cantilevers are simultaneously illuminated and a single photodetector measures the superposition of their deflection signals. The resulting deflection signal, which consists of the sum of 12 signals from the cantilever array, goes to the array of PLLs where each PLL locks to the unique resonant frequency of a single cantilever. Each PLL tracks its assigned cantilever's resonant frequency by demodulating its deflection signal and then generates a sinusoidal drive signal at that frequency. The drive signals from each PLL are then summed and used to drive a single piezo actuator positioned directly underneath the chip, completing the feedback loop.



Figure 4: Simulated data showing frequency peaks originating from single cells flowing through a series of SMRs separated by delay channels. Cells grow as they traverse the array. After grouping frequency peaks originating from the same cell, that cell's growth rate can be obtained by regressing its buoyant mass versus time. Because many cells can traverse the array simultaneously, this device can achieve much higher throughput than a single sensor.

After acquiring the frequency signals for each cantilever, we convert them to mass units via each cantilever's sensitivity (Hz/pg), which must be known precisely. Therefore, we measured the cantilever sensitivities during each experiment by spiking in inert monodisperse polystyrene particle size standards into all samples. Since the particles are highly uniform (~1% coefficient of variation in diameter), we can easily distinguish them even when they are of similar size to the cell sample of interest.

We utilized long serpentine "delay" channels between each SMR for giving the cells enough time to grow between mass measurements (Figure 3A). After a cell exits an SMR, other cells are free to enter it and get weighed. As a result, we are not limited



Figure 5: A) Growth data of lymphoid cell lines extracted from frequency shifts of cantilevers, color-coded by cantilever (blue is first cantilever, green is 6th cantilever, red is 12th cantilever). 7 and 9 μ m inert polystyrene particles are added as calibration and negative control, respectively. B) Lag-phase yeast culture growth dynamics in rich media measured at single-cell resolution.

to flowing only one cell through the array at a time, but can have many cells flowing in a queue. This enables high-throughput precision growth measurements across a wide array of cell types. The time between measurements can be controlled by adjusting the flow rate. Therefore, we can easily tune the device's behavior to trade throughput for resolution by changing the flow rate, i.e. faster flow yields higher throughput but poorer resolution, and conversely for slower flow.

Here we demonstrate proof-of-concept SMR arrays to monitor the growth of steady-state L1210 cells (Fig. 5A), a mouse lymphoblast cell line previously studied with SMRs [7]. In a 200minute experiment, we measured the size and mass accumulation rate of 150 cells. In contrast, a single SMR measuring each cell for 20 minutes could measure ≤10 cells as it takes some time to switch between cells. We next investigated whether serial SMR array could be used to study yeast (S. cerevisiae), a model organism of interest in biology. We measured the growth of single yeast cells in lag phase – a brief period of slow or no growth following transfer from spent media to fresh media. In a 3.5 hour experiment, we measured the mass accumulation rates of 208 cells (or cell clumps, as daughters often remain adhered to mothers following division), as shown in Fig. 5B. Here we've plotted these cells' mass on a logarithmic scale because single yeast cells grow exponentially. On a semilog plot, the slope of a cell's mass trajectory may be interpreted as an exponential growth rate, which is equivalent to its mass accumulation rate per unit mass.

CONCLUSIONS

We envision that this device will be applicable in many areas of research and potentially in clinical settings. The ability to detect rare growing cells in complex mixtures from patients could provide the possibility of correlating these measurements to disease states, and ultimately assessing drug susceptibility in rare cells. Yet the approach described here could be optimized further. Improving the actuation and detection schemes might enable an order of magnitude improvement in growth rate precision, and this extra precision can be exchanged directly for higher throughput by increasing the flow rates of cells transiting the array. Furthermore replacing the optical lever setup with embedded piezoresistive sensors could enable controlling hundreds of SMRs on a single chip.

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DESIGN AND OPTIMIZATION OF A SINGLE-CELL PROTEIN ASSAY FOR CLINICAL UTILITY

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ABSTRACT

We report a novel microdevice for both handling and single-cell protein analysis of rare patient-derived cells. By micropatterning a polyacrylamide gel layer on a glass slide for active cell settling and protein separation, we have created a platform that integrates handling samples of rare cells and analysis of proteins at a single-cell resolution. Centrifugation is used to rapidly settle single cells into microwells, followed by an electrophoretic separation of proteins and immunoprobing. Glioblastoma (brain cancer) and breast cancer cell lines are used as model systems to design and optimize our device. To ensure clinical utility, we design for efficient operation in near-patient settings.

INTRODUCTION

Cell-to-cell variation is a hallmark of life processes (development) and diseases (cancer). In cancer, the development of drug resistance is emerging as a major challenge. Drug resistance emerges from altered protein signaling in subsets of tumor cells (and not changes in DNA) [1]. Therefore, single-cell resolution protein analysis tools are needed. The de facto standard for protein analysis is the immunoassay. Regrettably, subtle chemical changes on proteins implicated in drug resistance are not detectable with available antibody probes [1, 2]. In a paradigm shift, we surmount this gap by introducing a 'single-cell western blot' that prepends an electrophoretic separation of proteins to the backend immunoassay [3]. By resolving proteins by both size and antibody binding, the assay can detect key targets not detectable by assay alone (similar size proteins). Precision control of fluid - conferred by microfluidic systems - makes these first-in-kind single-cell protein measurements possible [3-6]. Here we overcome the "macro-to-micro" interface challenge of handling sparingly available cells derived from fine needle aspirates (biopsy) and circulating tumors cells (CTCs) purified from blood [7, 8], by reporting an integrated microdevice for both handling and single-cell protein analysis of sparingly available cells from fine needle aspirates or circulating tumor cells.

To seat single cells in microwells, multiple groups have employed passive gravity-based sedimentation [3, 9, 10]. Using this accepted approach, we have observed successful capture of just 0.05% of cells from millions of starting cells [3]. Unfortunately, with patient-derived biospecimens, starting samples comprise just 50 to 500 of CTCs per an enriched sample [7, 8]. Random passive-gravity settling and sparsely located microwells (only 0.1% slide area is patterned with microwells) result in poor cell settling [3].

Many techniques are developed to actively sediment cells. With magnets and magnetic microbeads coated with antibody, cells of interest can be bound to microbeads and manipulated by magnetic forces [7, 11]. However, this method requires antibody-receptor binding between magnetic beads and target cells. Using an alternative current, non-uniform electric field is created to induce dielectrophoresis (DEP) force to actively settle cells. However, DEP requires a complex circuit fabrication with electrodes, and needs to exchange buffers after settling and electrophoresis [12-14]. Centrifugation is used as a means to settle samples with a rare number of cells. With a conventional spinning motor, a centrifugal system is robust and suitable for cell handling; centrifugation is independent of buffer properties (i.e. conductivity, detergent, salt concentration, pH), which affect electrophoresis [15, 16]. A centrifugal method has previously reported with a very high capture efficiency over 90% and can be incorporated with electric field and magnetic field [17, 18].



Figure 1. Patterned polyacrylamide gel with microwells integrates centrifugal cell settling and single-cell western blotting technique for sparingly available cells. (A) Two chambers with flanking microwells, fabricated with polyacrylamide gel on a glass slide and a polyester film as a lid, is shown. A bottom diagram shows three apparent forces acting on a cell in a fluid chamber in a rotating reference frame: centrifugal ($F_{centrif}$), Coriolis ($F_{Coriolis}$), drag (F_{drag}). In the right image, three false-colored cells in are settled in microwells due to the centrifugal force in a rotating reference frame. (B) A workflow of centrifugal single-cell western blotting is illustrated. After centrifugation, cells that are settled in microwells are chemically lysed, and an electric field is applied to separate proteins. UV is applied to activate benzophenone methacrylate (incorporated in the gel) for protein capture. Fluorescent antibodies are introduced to probe for target proteins.

Consequently, we designed, developed, and demonstrated an efficient and low-loss single-cell western that rapidly guides cells to microwells with centrifugal force in a rotating reference frame (Fig.

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Solid-State Sensors, Actuators and Microsystems Workshop Hilton Head Island, South Carolina, June 5-9, 2016 1A). Once in a microwell, we performed single cell western blotting on each cell. Initially, fluorescent microbeads were tested to optimize centrifugal velocity. Subsequently, we tested both centrifugal settling and western blotting with glioblastoma (brain cancer, U373) and breast cancer (BT474 and SKBR3) cell lines.

METHODS

Fabrication of centrifugal single-cell western blotting platform

Our polyacrylamide gel platform was molded against a silicon wafer with SU-8 features. The SU-8 3050 (Y311075, MicroChem) mold is composed of two layers by standard soft lithograph methods with the OAI UV light system with a mask aligner (Model 200IR, OAI). Two Mylar masks (CAD/Art Services) were used to make the two-layer molds: the bottom layer consists of fluid chamber sides (80-µm thickness), and the top layer is composed of microwell posts (40-µm thickenss). Standard guidelines from MicroChem were followed for UV exposure, baking, and developing. The wafer with SU-8 mold was coated via vapor-deposition of 100 µL of the hydrophobic silane dichlorodimethylsilane (DCMC, 440272, Sigma-Aldrich) for 40 minutes in vacuum. Two fluid chambers and flanking microwells were cast on a standard microscope glass slide with polyacrylamide gel against this hydrophobic wafer with SU-8 features (Fig. 1A, 2A). An 8%T, 2.7%C polyacrylamide gel was diluted from a stock 30%T, 2.7%C acrylamide/bis-acrylamide solution (A3699, Sigma). A 3mM benzophenone methacrylamide co-monomer (in-house) and 75mM Tris HCl pH8.8 (T1588, Teknova) were incorporated into the polyacrylamide gel precursor, and the gel was polymerized chemically with ammonium persulfate (A3678, Sigma) and TEMED (T9281, Sigma). To seal the fluid chambers, a top lid is composed of a hydrophilic polyester film (GelBond[®], Lonza) with inlet and outlet openings. The lid was topped on the wetted polyacrylamide gel slide, and a liquid superglue (Loctite) was applied on edges between the glass slide and the lid. A 1% BSA solution was added into device chambers via an inlet and incubated for 5 min at room temperature.

Cell/microbead injection and centrifugation

FITC-labeled and 15-µm diameter polystyrene microbeads (F21010, Invitrogen) was diluted in 1x phosphate-buffered saline (PBS, 21040CV, Corning). For glioblastoma (U373-GFP) and breast cancer (BT474, SKBR3) cell lines, cells were cultured in a tissue-culture flask with ATCC recommended cell culture media. dissociated U373-GFP cells were with trypsin:EDTA (0.055:0.53mM, 400-150, Gemini), while BT373 and SKBR3 cells were dissociated with 0.5 mM EDTA (AM9260G, Ambion). Dissociated cells were counted with a hemocytometer and diluted in an ice-cold 1x PBS. Microbead or cell solution with a desired volume $(1 \sim 20 \,\mu\text{L})$ was pipetted into an inlet of the device chamber. Then, a Kimwipe wiper (Kimtech) was applied on an outlet for 1 sec to inject the microbead/cell solution inside the device chamber. Then, a conventional upright spinner was used to spin the device at a desired rotational duration and velocity.

SDS-PAGE and photocapture of proteins

After centrifugation of the device, the top lid was cut and slowly slid off from the polyacrylamide gel slide. To lyse cells, a 10 mL of a RIPA-like lysis/electrophoresis buffer consisting of 1% SDS (L3771, Sigma), 0.1% v/v Triton X-100 (X100, Sigma), 0.25% sodium deoxycholate (D6750, SigmaAldrich) in 12.5 mM Tris, 96 mM glycine, pH 8.3, (161-0734, Bio-Rad) was poured over the slide for 15 seconds. A 200V (40V/cm) was applied across the slide to electrophorese proteins for 20 seconds. Immediately after the protein electrophoresis, we shone the slide with UV for 45 seconds to activate benzophenone moieties, which immobilize proteins in the polyacrylamide gel. The slide was washed with 1x TBS with tween (TBST, 77500, Affymetrix) for 10 min before probing with antibodies.



Figure 2. Fluid chamber design and rotational speed is optimized by numerical simulations. (A) A top view of the centrifugal single-cell western blot device (upper image) and a schematic diagram of a left fluid chamber (bottom image) are shown. During centrifugation, drift velocity (U_{drif}) determines time and settling efficiency for moving cells to microwells in chamber. On the other hand, deflection velocity ($U_{deflection}$) affects distribution of cells across the flanking microwells. (B) In a rotating reference frame, force body diagram and equations to calculate drift and deflection velocities of a cell inside a fluid chamber. Drag force is created against centrifugal ($F_{centrif}$) and Coriolis ($F_{Coriolis}$) forces. By balancing these forces, numerical estimations of drift and deflection velocities are calculated. (C) Drift and deflection velocities with various rotational speeds. Drift velocity is over 100 μ m/s at 2000 rpm, which generates a low deflection velocity (0.02% of drift velocity) and ensures every cell in a fluid chamber to travel to microwells within 2 minutes. (D) The settling efficiency is over 80% for both beads and cells after spinning at 2000 rpm for 2 minutes.

Immunoprobing

Primary and secondary antibodies were diluted with 1xTBST with 2% BSA. The slide area, where microwells and immobilized proteins are present, was soaked in a 30 µl of 0.1 g/L primary antibodies for 2 hours at a room temperature. Afterwards, we washed the slide with 1x TBST for 30 min. Subsequently, the slide area was incubated with a 30 µL of 0.05 g/L secondary antibodies with fluorescent tags for 1 hour at a room temperature, followed by a 1xTBST wash for 1 hour. Finally, the gel in the slide was dried off completely by a nitrogen stream and imaged with a fluorescent microarray scanner (GenePix 4300A). GAPDH primary antibody was purchased from Sigma (SAB2500450), β -Tubulin from Abcam (ab6046), HER2/ErbB2 from Thermo Fisher (MA5-13105), and PR from Cell Signaling (3513).

RESULTS AND DISCUSSION

Optimization of centrifugal cell settling by numerical simulation and empirical iterations with microbeads and cells

We first investigated how fast cells are moving towards microwells during centrifugation inside a fluid chamber of the device (Fig. 2). In order to settle into microwells, cells were injected via an inlet and had to be moved about less than or equal to a 10 mm distance to reach microwells (Fig. 2A). During centrifugation, a fluid (i.e. 1x PBS) inside the fluid chamber was stagnant. However, in a rotating reference frame, three apparent forces are present on particles inside the chamber: centrifugal, Coriolis, and drag forces (Fig. 2B). Because Reynold's number was far less than 0.01 at below 5000 rpm inside a fluid chamber, Stokes' law could be used to calculate a drag force acting on the bead. A force balance between centrifugal and drag forces resulted in a drift velocity (U_{drift}), which represents how fast cells are moving to microwells. Similarly, Coriolis and drag forces determine a deflection velocity. The deflection velocity, perpendicular to the drift velocity and the row of flanking microwells, affected distribution of cells and had to be minimized to prevent multiple cells from piling on microwells in a corner of the fluid chamber (Fig. 2C).



Figure 3. Centrifugal force is effective and efficient for rapid seating of glioblastoma cells into microwells in preparation for subsequent single-cell protein analysis in a rotating reference frame. (A) False-color fluorescence micrograph of before (left) and after (right) centrifugation for 2 min at 2,000 RPM. Green signal indicates fluorescing cells in microwells. No cells are detectable at microwells prior to centrifugation, as cells are deposited outside the field of view. (B) Cell occupancy distribution from the centrifugal seating approach shows most microwells contain 1 cell, as desired. (C) Comparative performance metrics for passive gravity-based cell settling (previous method) and active, centrifugation based cell settling (new method introduced in this work). Note that across all metrics, active centrifugal settling outperforms passive settling. These high-performance metrics indicate the new technique is appropriately designed for analysis of single cells in limited samples, including fine needle aspirates as is underway.

To investigate an efficiency of centrifugal settling in our system, homogeneous and fluorescent polystyrene microbeads ($14.8 \pm 0.1332 \ \mu m$, $1.06 \ g/cm^3$) were first used to test settling in microwells by centrifugation. Assuming that the bead acceleration was instantaneous, drift velocities of microbeads towards traps at 3000 rpm were calculated as 50 μ m/s, respectively. Over 80% of

polystyrene beads were settled into microwells in 2 minutes at 2000 rpm (Fig. 2D). Subsequently, we tested with glioblastoma cancer cell line with GFP fluorescence (U373-GFP) to test the centrifugal cell settling. At 2000 rpm for 2 min, we successfully settled 80% of cells in microwells (Fig. 2D). Sample seating times were 10% of the time required for gravity-based, passive seating (Fig. 3C), as corroborated with numerical simulation estimates. Deflection velocities and trajectories (due to Coriolis force) inform chamber geometry and size so as to minimize cell accumulation along just one wall of each sample chamber (i.e., ~0.05 μ m/s at 2000 rpm, Fig. 2C). Fluorescence imaging of glioblastoma cells revealed >70% of microwells contain single cells (Fig. 3A), even when starting with just 100 cells. Importantly, centrifugation results in settling efficiencies increased by > 5-fold while shortening settling time by > 10-fold (Fig. 3C).



Figure 4. The integrated centrifugation settling and single-cell western blot microdevice successfully analyzes multiple proteins in single cells from two types of cancer. (A) False-color fluorescence images of glioblastoma cells (U373-GFP) seated in microwells (left image) and subsequent western blot of 3 protein targets in each cell (b-tubulin, GAPDH, and GFP). Fluorescence signal along the separation axis is also shown. Multiple cells are handled and assayed simultaneously. (B) False-color fluorescence images of two different types of breast cancer cells, with Case #1 having cells express the protein HER2 but not the protein PR; whereas Case #2 sees all cells express HER2 (as expected) but cell-to-cell variation in expression of the protein PR (unexpected, see peak marked with a *). Measuring cell-to-cell variation is critical to understanding cancer progression and response to treatment.

Single-cell western blotting after centrifugal cell settling

After validating centrifugal cell handling, we integrated with single-cell western analysis. We successfully completed the full western by detaching the polymer film lid, then subjecting each cell to 10-sec in-microwell chemical lysis, 15-sec protein electrophoresis separation, UV-activated immobilization of proteins to the polyacrylamide gel layer (incorporating light-sensitive benzophenone), and an in-gel immunoassay for each of our three target proteins. In the integrated device, we observed the protein targets at the appropriate molecular masses (β -tubulin, 51 kDa; GAPDH, 35 kDa; and GFP, 27 kDa; Fig. 4A). In a striking result, the integrated assay fully resolved proteins with just an 8 kDa mass difference (Fig. 4A).

After validating protein separations and probing in U373-GFP cells in our single-cell western blotting device, we have started to investigate cell-to-cell heterogeneity of receptor proteins in breast cancer cell lines, BT474 and SKBR3 [19]. Currently, a breast cancer diagnosis consists of three protein markers: estrogen receptor (ER), progesterone receptor (PR), and human epidermal growth factor receptor 2 (HER2) [20]. Based on qualitative analysis of these proteins from biopsies, breast cancer patients would receive various treatments for their cancer. Quantitative measurement of these receptor proteins at a single-cell resolutions would provide valuable proteomic-information from sparingly available cells derived from fine needle aspirate biopsy to understand correlations among these receptors and elucidate drug resistance from monoclonal antibodies for effective breast cancer treatments.

We tested our centrifugal single-cell western blot to measure HER2 and PR protein expressions from BT474 and SKBR3 single cells. Our preliminary data also showed presence of HER2 in both BT474 and SKBR3 cells, while SKBR3 cells did not express PR (Fig. 4B). Interestingly, we found cell-to-cell variation of PR in BT474 cells. We are currently performing more cells on this centrifugal single-cell western blot and probing western blot data with other breast cancer markers.

Our integrated rare cell handling and protein analysis platform enables direct measurement of aberrant protein signaling during development of drug resistance in brain and breast cancer cells. We are measuring cell-to-cell variation in fine-needle aspirate specimens and will report on our findings during development of cancer drug resistance.

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FABRICATION OF NEURAL MICROELECTRODE ARRAYS FROM EXTRACELLULAR MATRIX

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ABSTRACT

This paper reports a suite of microfabrication strategies that allow electronic structures to be fabricated on and within protein extracellular matrix (ECM)-based substrates. ECM-based implantable neural electrodes (NEs) are presented here as a case study. ECM proteins are processed to form flexible substrates to promote neural growth and function. A stiff delivery needle or needle array is integrated with the ECM-NEs to facilitate successful insertion and is subsequently removed. Preliminary animal validation experiments using larger ECM-NEs showed *in vivo* intracortical recording capabilities of the ECM-NEs from rat motor cortex.

INTRODUCTION

Advances in neural microelectrodes (NEs) have enabled interfacing with and extracting information from neural circuits [1] and have led to many significant advances such as neural recordings, deep brain stimulation, and brain-controlled prosthetic devices [2, 3]. Current state-of-the-art NEs in use, particularly in clinical settings, are typically fabricated from silicon or noble metals [4]. These devices face two major challenges which hinder their sustainable communication with neurons over long periods of time. First, materials composing these devices are typically foreign to their implanted environment, resulting in a sustained glial response that eventually leads to neurodegeneration and decreases the fidelity of the recorded neural signals [5]. Second, the large mechanical mismatch between the soft neural tissue and the rigid devices results in large stress and strains and causes local inflammation at the device-tissue interfaces, which impede the sustained functionality of these devices [6].

To resolve these challenges, biomolecule-based coatings including ECM molecules [7] and anti-inflammatory drugs [8] have been implemented on NE surfaces, and showed initial improvement in promoting neural cell growth and preventing glial scar formation at the tissue-device interfaces, ultimately enhancing neuronal attachment and achieving improved initial recordings in vivo. However, these results were unsustainable at longer time scales and, thus, could not support chronic neural interfacing [9, 10]. Research suggested that reduction in the mechanical modulus of the materials comprising the device is expected to provide a more tissue-complaint interface, thereby lowering the strains induced on the tissue. However, soft/flexible NEs face a materials challenge because they must also be sufficiently stiff to penetrate into the neural tissue while minimizing stresses and damages. The mechanical compatibility [11] and biocompatibility [12] of ECMbased materials suggest that, in contrast to a coating, fabricating almost the entire NE from ECM material may be fruitful. We have recently developed ECM-NEs that are primarily composed of collagen [13]. Due to the neuronal compatibility and mechanical flexibility of collagen, the collagen-based NEs produced increased neural compatibility and reduced inflammatory responses compared with inorganic approaches. However, the dimensions of those collagen-based NEs were 310 µm in width, which is an order of magnitude larger than the cortical neuron diameters. The

relative large sizes of those ECM-NEs could result in significant injury to the neural environment during device implantation. To further reduce this mechanical injury, it is desirable to reduce the sizes of ECM-NEs to approach neuronal feature sizes. However, as sizes reduce, these flexible NEs may reach a mechanical limit that they cannot be sufficiently stiff for implantation. In these cases, an implantation tool is needed to deliver these ECM-NEs into the neural tissue. In addition, to further improve biocompatibility, it is desirable to fabricate NEs using laminin, a brain ECM protein that can provide better compatibility with the neuronal environment [14].

Here we reported a set of fabrication processes that allow the fabrication of ECM-NEs with width of 100 μ m using both the collagen substrate and a collagen-laminin composite substrate, where collagen can provide mechanical flexibility and toughness and laminin can allow improved neuronal compatibility. Moreover, these processes are integrated with a stainless steel delivery vehicle on the ECM-NEs to facilitate implantation of these smaller devices. Further, fabrication of ECM-NE arrays in both 2D and 3D conformations, desirable for high-spatial resolution neuronal interrogation over a larger volume of tissue, is enabled.

DESIGN AND FABRICATION OF ECM-NE



Figure 1: Schematic of (a) ECM-NEs, (b) ECM-NEs integrated with stainless steel delivery needle, and (c) rolling the 2D array to 3D arrays. The delivery needle is separated from the ECM-NEs and removed from the brain after implantation, leaving only ECM-NEs implanted in the brain and interfacing with neurons.

The shape of each ECM-NE shank followed those of commercial Michigan electrodes that use a planar needle design. Each ECM-NE comprises an ultrathin (6 μ m in thickness) parylene microelectrode core embedded within a 40 μ m thick flexible ECM

protein substrate as shown in Figure 1a. The ECM substrate provides a large amount of neuronal ECM matrix protein to promote neural growth and function and potentially minimize the inflammatory responses and biocompatibility concerns associated with the implanted devices. Previously fabricated collagen-NEs were 300 µm in width, three times wider than the commercial Michigan arrays [4]. The relatively large size of those previous arrays could increase the mechanical injury to the tissue. In this work, we designed and developed ECM-NEs that are 100 µm in width, comparable to the sizes of commercial Michigan arrays. To facilitate the implantation of these ECM-NEs, a thin (25 µm) stainless steel needle layer was integrated with the ECM-NEs as shown in the schematic in Figure 1b. These stainless steel needles could be released from ECM-NEs and removed from the tissue after implantation of the ECM-NEs. To enable high spatial resolution neural interrogation over a larger volume of neural tissue, 3D needle arrays were achieved by rolling the linear 2D needle arrays around a hypodermic needle that defines the inner diameter of the 3D array (Figure 1c). In addition, these stainless steel needles also serve as shadow masks during the laser micromachining of ECM-NEs, eliminating errors generated during the alignment and positioning of the devices under the laser beam.

The fabrication processes of the ECM-NEs are illustrated in Figure 2. A thin (3 μ m in thickness) Parylene C (poly-monochloropara-xylylene) film was deposited on a silicon carrier wafer (Figure 2a) using a chemical vapor deposition process (SCS Labcoater, Special Coating Systems, Inc., Indianapolis, IN, USA). A 200 nm thick gold layer was deposited using electron beam evaporation and patterned by lift-off, yielding interconnecting lines and pads (Figure 2b). Another thin (3 μ m in thickness) layer of Parylene C was deposited and then patterned by reactive ion etching, leaving only the pads at the ends of the lines exposed (Figure 2c).

The preparation of collagen films that are ultimately used as substrates for further processing were described in [13]. The collagen-laminin composite film in this work was prepared as follows. Type I rat tail collagen in a 3 mg mL⁻¹ solution (Corning®, Corning, NY) was gently mixed with 10X phosphatebuffered saline (PBS), 0.1 M NaOH and 6 mg mL⁻¹ laminin (Trevigen, Gaithersburg, MD) at a ratio of 13:2:1:1 by volume until small fragments started to form. The mixed solution was then cast into an acrylic mold, followed by polymerization at 37 °C and 96% humidity for 24 hours, and then dried on glass slides in air at 37 °C for 24 h. This collagen-laminin composite film was rinsed with DI water multiple times until a fully transparent film was observed. After rinsing, the film was air dried, forming a uniform dried collagen-laminin composite film (thickness of 40 µm). This collagen-laminin composite film was subsequently removed from the acrylic mold and glass slides for subsequent use as substrates.

The first layer ECM (collagen, or collagen-laminin composite) substrate in sheet form was then applied under hydrated condition on top of the parylene core. After the ECM dehydrated, the parylene cores were embedded within the ECM substrate (Figure 2d). This ECM substrate with embedded parylene core was then peeled off from the silicon carrier wafer, flipped, and embedded with another layer of ECM hydrogel substrate. After the second layer of ECM substrate dehydrated, no obvious interface was found between the two ECM layers under SEM. This ECM substrate encapsulating the parylene core was then aligned, rehydrated, and dehydrated on top of a stainless steel layer that was previously patterned into 2D needle arrays using 532nm green

laser micromachining so that the collagen substrate is attached to the stainless steel (Figure 2e). This ECM substrate attached to the stainless steel substrate was then flipped, and patterned to form 2D arrays of ECM-NEs using a 193 nm excimer laser etching with the stainless steel serving as a contact shadow mask (Figure 2f). The use of stainless steel contact mask eliminated the alignment of the ECM-NE patterns with the laser beam, thus avoided errors that would be generated during the otherwise needed alignment step.



Figure 2: Fabrication processes of the ECM-NEs: (a) Parylene was deposit on Si carrier wafer, (b) gold traces were deposited and patterned using lift-off, (c) top layer parylene was deposit and patterned using reactive ion etching (RIE), (d) parylene core was embedded within ECM substrate, (e) ECM substrate with embedded parylene core was peeled off from the structure, flipped and encapsulate with a 2^{nd} layer ECM substrate, and then aligned on the pre-patterned stainless steel layer, (f) the ECM substrate attached to the stainless steel was flipped, and patterned to form a 2D array of ECM-NEs using excimer laser etching with stainless steel serving as a contact shadow mask, and (g) 2D linear array was rolled around a hypodermic needle to form the 3D array.

Dense 3D NE arrays are desired for high spatial resolution neural interrogation over a larger volume of tissue. To fabricate the 3D ECM-NE arrays (3D ECM-NEAs), we first fabricated long 2D linear arrays on a stainless steel insertion vehicle. A slit (1 mm in width and 5 mm in length) was cut at the tip of the 26G hypodermic needle using a 532 nm green laser for use as a clip to hold the 2D linear array. This linear array was then rolled around the 26G hypodermic needle (Figure 2g) to form a 3D array with cylindrical shape.



Figure 3: Fabricated ECM-NEs and stainless steel delivery vehicles: (a) collagen-NE (shank width: 310 μ m, shank length: 2 mm) floating in DI water, (b) tip of a collagen-NE (shank width: 310 μ m, previous work), (c)-(d) collagen-laminin-NE (shank width: 100 μ m, shank length: 5 mm), (e) 3D array rolled around a 26G hypodermic needle, (f) close-up view of the 3D array, (g)-(h) AFM height images of (g) collagen substrate, and (h) collagen-laminin composite substrate.

Figure 3 shows a representative picture of the fabricated devices. After being hydrated in DI water, the ECM-NEs kept their as-fabricated shape as shown in Figure 3a. No rapid degradation or change in shape was observed when the devices were immersed in DI water for 24 hours. The tip of a 310 μ m wide collagen-NE fabricated using previous method is shown in Figure 3b. Compared with the previous method, the method described herein allows fabrication of smaller ECM-NEs partly due to the reduced alignment error during the excimer etching of ECM substrate. Figure 3(c) and 3(d) show pictures of a fabricated 100 μ m wide and 5 mm long collagen-laminin NE using the new approach.

Laminin has long been recognized and used as a standard coating material to promote cortical neural regeneration, growth, and function [14]. However, laminin thin films are too fragile to sustain mechanical handling during microfabrication. To overcome this challenge, we have fabricated collagen I-laminin composite films. These collagen-laminin composite films exhibit increased toughness compared to laminin alone, and we found a collagen I-laminin (13 wt%) composite film is able to sustain the fabrication process described here without micro-cracks or damage manifesting in the ECM-NEs. This is due to the long fibrils and 3D

structure of collagen I providing toughness to the composite films [11]. These collagen fibers normally have diameters of tens of nanometers [15]. Atomic force microscopy of the collagen substrate and collagen-laminin composite substrate show structure that is consistent with fibrous structures (Figure 3g-h).

A dense 3D needle array is demonstrated in Figure 3(e) and 3(f). The inner diameter of the 3D array is defined by the diameter of the hypodermic needle tip, which is approximately 450 μ m. The, spacing between two adjacent rolls is defined during the rolling process. Optionally, a sacrificial spacer material comprising a layer of PVA or dextran coated at the base of the linear array can assist in adjacent roll spacing.

INSERTION OF ECM-NE



Figure 4: Insertion of ECM-NEs into a brain phantom material (1% agarose gel). (a) insertion of a single shank ECM-NEs and (b) release and removal of the SS delivery needle after insertion, leaving only the single shank ECM-NE inserted; (c) insertion of a four-shank 2D ECM-NE array, and (d)-(e) after removing of the 2D SS delivery needle, leaving only the four shanks ECM-NE inserted; (f) inserted 3D ECM-NE arrays after removal of the 3D SS delivery array.

The ECM-NEs were integrated with thin (thickness of 25 μ m) microfabricated stainless steel (SS) needles to allow successful implantation into a brain mechanical model (Figure 4a and 4c). Within one minute after implantation, the delivery needle was released from the ECM-NEs by hydration and was subsequently removed, leaving the flexible ECM-NEs at the implantation sites. Figure 4 (b) shows an inserted single shank ECM-NE, Figures 4 (d) and (e) show a top view and side view of a 4-shank 2D ECM-NE array inserted in the brain phantom after release and removal

of the 2D SS delivery array, and Figure 4 (f) shows the side view of a 18-shank 3D ECM-NE array inserted in the brain phantom after release and removal of the 3D-SS delivery array. These results demonstrated that the insertion method by fabricating and inserting the ECM-NEAs on SS delivery array together and subsequently removing the SS array upon insertion and hydration is a convenient and reliable method for insertion of ECM-NEA.

ELECTRICAL FUNCTIONALITY OF ECM-NE

We have previously shown that ECM-NEs are electrically functional and can record electrophysiological signals from rat barrel cortex upon whisker stimulation using larger ECM-NEs without insertion devices over a 54-day period (Figure 5a-c) [13]. These larger ECM-NEs supported square electrode sites 80 μ m on a side. When the sizes of the ECM-NEs are reduced, the electrode sizes must also be reduced. Here, we show that the ECM-NEs with diameter of 20 μ m show an impedance of approximately 500 k Ω at 1 kHz. This impedance agrees with the predicted model, and is within the allowable impedance range for NEs to record intracortical neuronal signals. Electroplating the NEs with nanoporous Pt could be considered to lower the device impedance for better signal to noise ratio [13].



Figure 5: Electrical functionality of ECM-NEs. (a)-(c) In vivo electrophysiological neural signal recording data from previously fabricated ECM-NEs [4]. (a) Representative 90 s of high-speed recording showing neural spikes upon whisker stimulation, (b) mean waveform and ISI histogram, and (c) mean SNR of the largest signal over a 54-day time period. (d), EIS of ECM-NEs with smaller (20 μ m in diameter) recording sites.

Enabling microfabrication with ECM proteins is expected to lead to implantable microelectronics for optimum neuronal compatibility. Our current focus is on enabling microfabrication technologies to produce microelectronics with a broader range of ECM materials to enable optimal intracellular signaling and cell recruitment, tuning the mechanical properties of these devices to closely match those of the host tissue to minimize undesired stress and strains at the device-tissue interface, and advancing MEMS technologies to minimize device feature sizes to minimize the insertion trauma. The development of ECM-NEs is expected to resolve both materials and mechanical challenges associated with implantable NEs and support sustainable neural interfaces.

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FAST AUTONOMOUS FLIGHT WITH MICRO UNMANNED VEHICLES

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ABSTRACT

The last decade has seen a proliferation of Micro Aerial Vehicles (MAVs), particularly multirotor vehicles. Indeed quadrotor and hexrotor platforms can be bought off the shelf, and the basic technology has been commoditized. However, high-speed agile flight in GPS-denied environments remains a challenge. This is because the weight of the onboard sensors and processors that are required for autonomy increases the size and inertia of the vehicle, and decreases its maneuverability. In this paper, we present the FALCON, a quadrotor MAV) capable of autonomous high speed flight in complex environments using onboard sensors for state estimation. We discuss the design considerations required for high speed flight and the energetics of quadrotor platforms, and provide specifics on the FALCON design and its performance.

INTRODUCTION

The last decade has seen rapid progress in the use of micro aerial vehicles (MAVs) as a research platform. Rotorcrafts constitute an important class of MAVs. They are of particular interest due to their ability to hover in place, and quickly transitioning from hover to forward flight and back. They include conventional and co-axial helicopters[1], quadrotors^[2], hexarotors and ducted fans^[3]. Of these, quadrotors have proved to be the most commonly used aerial platform in robotics research labs. In this class, the Hummingbird and Pelican quadrotors manufactured by Ascending Technologies, GmbH[4], with a tip-to-tip footprint of under 75 cm and weighing less than 1kg have proven to be remarkably capable and robust platforms as shown in [5][6]. The basic design for a quadrotor is simple and the cost of the basic components is low. Indeed the number of drone companies using this design today shows that the barrier to designing and manufacturing quadrotor platforms is low.

Rotorcrafts are used today in a variety of applications including inspection, photography, and data collection in the entertainment, construction and insurance industry. Quadrotors have been proposed for surveillance and search and rescue missions in confined indoor and outdoor environments due to their small size, agility, and ability to hover. In such missions, it is essential that the MAV is capable of autonomous flight to minimize operator workload. Their capability to execute aggressive trajectories [6] and maneuver in 3D constrained environments, autonomously generate 3-D maps of unknown environments [7], autonomously build 3-D structures using grippers [8], and form swarms of robots flying in formation [9] opens the door to a wide variety of new applications for this technology.

This paper presents the design considerations for a quadrotor platform capable of high-speed agile flight in GPSdenied environments. We use scaling laws to illustrate the benefits of small size and empirical results to show the value of developing light weight platforms. Finally, we present the detailed design of the FALCON platform, which has an unprecedented thrust-to-weight ratio of 2.5 and a maximum speed of 20 m/s.



Figure 1: CAD model of the FALCON robot



Figure 2: The FALCON robot

MECHANICS

Most MAVs are equipped with sensors like MEMS IMUs, GPS, cameras, and laser scanners etc. Robust state estimation for autonomous flight in GPS-denied environments frequently requires multiple sensors such as laser scanners[10], monocular cameras[11], stereo cameras[12], and RGB-D sensors[13]. Sensors increase the payload that must be carried, increasing the size of the MAV and its battery.

Scaling up in size to accommodate heavier payloads has a dramatic effect on aspects such as inertia of the robot and flight time[14]. As shown in our previous work [15], the blade tip speed, v_b , for small quadrotors scales with the square root of the characteristic length L. This is often called the Froude scaling paradigm in which the Froude number, given by

$$Fr = \frac{v_b^2}{Lg} = \frac{\omega^2 r^2}{Lg} \tag{1}$$

is a constant, where r is the rotor radius and g is the acceleration due to gravity. Assuming that the rotor radius r scales linearly with the characteristic length of the robot, L, and observing that

$$\omega = \frac{v_b}{r},\tag{2}$$

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Solid-State Sensors, Actuators and Microsystems Workshop Hilton Head Island, South Carolina, June 5-9, 2016 and the inertia of the robot, I, scales inversely with the size of the robot,

$$I = \frac{1}{L^5},\tag{3}$$

we can show that the maximum angular acceleration α [15] scales as:

$$\alpha \sim \frac{1}{L} \tag{4}$$

This analysis suggests that by reducing the size of the vehicle by a factor of two, the vehicle's maximum angular acceleration doubles.

A simple rigid body dynamics model can be used to analyze the maneuverability of a quadrotor. Figure 3 illustrates the effect of improved angular acceleration on maneuverability. Increasing the angular acceleration decreases the turning radius, and this increase is particularly significant at high speeds. The same model shows that the distance taken for the vehicle to come to rest at a hover position decreases similarly with higher angular accelerations.

In [14], we also show that quadrotors for which L is roughly 1 meter consumes approximately 200W/kg. If we are designing a vehicle for a prescribed flight time, increasing the payload of the vehicle results in an increase in power consumption which also increases the weight of the battery. Thus the weight penalty increases substantially. Unfortunately sensors such as laser scanners are heavy and can weigh hundreds of grams. And yet these are necessary for reliable detection of obstacles since they provide estimates of distances to most obstacles in the environment under a wide range of operating conditions.

FALCON represents our effort to design a quadrotor platform that is capable of autonomous navigation and yet is highly maneuverable. While the presence of heavy sensors drives up the payload, the use of appropriate motors and propellors maximizes the thrust to weight ratio. In this paper, we present the 3 kg FALCON which is equipped with an IMU, a gimballed laser scanner, stereo cameras, laser altimeter, and magnetometer. All the computation for the FALCON is performed onboard on an Intel NUC computer with a 5th generation i7 processor.



Figure 3: Maneuverability at high speeds. The minimum turning radius is shown for two different limits on angular acceleration (governed by the inertia and the thrust) for a thrust to weight ratio of 2.0. The minimum turning radius is also equal to the stopping distance for maneuvers in which the robot must brake from an initial speed of v_{max} to come to a stop.

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AUTONOMY

There are several elements essential for fast autonomous flight. Perhaps the most important element is *state estimation*, the reliable estimation of position and orientation and their derivatives at high rates even when GPS is not available. Next, we require nonlinear *controllers* that are able to control the position and attitude of the vehicle even at high speeds, at states that are not close to the hover state. We also need the ability to create dense, 3-D maps of the environment allowing the vehicle to detect obstacles. *Dense mapping* allows *motion planning* in three dimensions allowing the vehicle to safely navigate to the goal. Each of these elements is discussed next.

State Estimation

High-speed flight presents a variety of challenges for state estimation. If we are interested in light weight and low inertia solutions, cameras and IMUs are the best sensors. Recent research in vision-based navigation by our group [16] has demonstrated robust algorithms in feature-rich environments. The algorithms use a monocular camera with image features being tracked across time fused with measurements from an IMU to get an estimate of the motion of the robot. In the tests reported in this paper we use the Semi-direct monocular Visual Odometry (SVO) algorithm [17]. A disadvantage of these systems is the inability to obtain an estimate of scale from measurements obtain from a stationary starting configuration. Using stereo systems allow us to get the scale immediately [18]. However, during high-speed flight, two major challenges manifest themselves. The first challenge involves the attitude estimator must incorporate models of drag and large accelerations when using measurements from the IMU. Second, it is difficult to achieve robustness with feature tracking during high speeds, especially with high pitch and roll rates because of motion blur, particularly in poor illumination.

Nonlinear Controllers

Sustained high speed flight involving large attitude changes poses challenges for non only for motion estimation but also for control. We use nonlinear controllers[19] with feed-forward control inputs enabling us to handle large deviations from hover for aggressive maneuvers.

Dense Mapping

Building maps for motion planning at high speeds with light weight platforms poses several challenges. First, to build dense maps over the time scale of 10 m (see Fig. 3) at 30 Hz. using cameras we need computational resources that significantly exceed what is available on standard, light-weight CPUs. The alternative is to use an active imaging system such as a 3-D lidar, which increases the weight substantially. As a compromise, we used a 2-D laser scanner mounted on a horizontal servo to enable mapping of 3-D obstacles (see Fig. 4). Second, the process of building a 3-D global map for motion planning is also computationally intensive. Instead, we create a body-centric representation of the occupancy around the vehicle and represent the free space as a union of convex polyhedra. We are able to generate smooth motion plans in free space at a minimum of 30 Hz.



Figure 4: The 2-D nodding laser scanner represents a compromise between the heavier 3-D lidar and the lighter cameras which do not lend themselves to dense mapping.

SYSTEM DESIGN

The FALCON is largely based on commercially available off the shelf (COTS) components. We chose the DJI[20] F450 frame and E600 motors with 12in propellers to obtain the maximum thrust to weight ratio with a compact footprint and the Pixhawk[21] autopilot for attitude estimation and control. Two Point Grey Chameleon3 cameras synchronized with a VectorNav VN-100 IMU enable state estimation, in addition to a Hokuyo UST-20LX LiDAR on a custom built nodding head gimbal. Additionally, the FALCON includes a downward facing sensor module consisting of a BlueFOX MLC200w camera and a LidarLite laser altimeter. The primary CPU is an Intel NUC with a 3.1GHz i7 processor with 16Gb of RAM and a 120Gb solid state hard drive. Communication with the robot for monitoring experiments takes place via an onboard high power, long range 802.11b/g/n WiFi transceiver. A custom designed Dragonfly power distribution board provides power conditioning, DC-DC converters, power connectors, and battery monitor. Finally, a 6S 4000mAh lithium polymer battery provides the base platform (without sensors) a thrust to weight ratio of 5, with a 15 minute flight time. Fully loaded, the weight with all components is 3.0kg with a thrust-weight ratio of 2.5. This thrust to weight ratio allows the robot to reach maximum speeds of 20 m/s with a maximum acceleration of 6 m/s^2 .



Figure 5: Dragonfly Power Distribution Board

In the "Dense Mapping" Section above, we briefly discussed the trade-offs between choosing 3-D lidars and cameras to minimize the total weight. A major consideration in choosing the present configuration with a nodding 2-D lidar instead of a camera-only configuration was the lack of light weight processors. It is worth noting that new tools are coming online that point to promising avenues for development. As seen in Table 1, new processing units are approaching ratios that exceed 160 MFlops/gram, which is nearly 10x processors available only 2 years ago.

As shown in Figure 6, the sensors and processors (along with cabling) contribute to 45% of the mass of the vehicle. This weight can be substantially reduced by integrating the sensors and processors into light weight packages. Using lighter weight composites will further reduce the total mass of the frame and the cage which contribute nearly 20% of the vehicle. It is unlikely that the Lithium polymer batteries or alternative powers will improve substantially in terms of either specific power or specific weight in the near future, even though advances are being made in terms of energy per unit volume. Finally, because quadrotors consume approximately 200 Watts/kg., as shown in Figure 7, nearly 90% of the power consumption arises from the weight of the vehicle. Thus light weight computing /sensing is more important than low power computing/sensing.



Figure 6: Mass distribution of the Falcon UAV



Figure 7: Power consumption at hover

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Computer	Intel NUC (i3-5010U)	Intel NUC (i5-5250U)	Intel NUC (i7-5557U)	Odroid XU3 (Exynos 5422)	Qualcomm Eagle (Snapdragon 801)
Cores	2	2	2	4	4
Clock Speed (GHz)	1.7	2.1	3.1	2	2.5
MFLOPS (Single-Core)	1900	2730	3440	1030	1200
MFLOPS (Multi-Core)	4250	5400	7480	4270	4350
Mass (g)	210	210	210	38	27
MFlops / g (Single-Core)	9.05	13	16.38	27.11	44.44
MFlops / g Multi-Core)	20.24	25.71	35.62	112.37	161.11

Table 1: Light weight versus low power computers for autonomous flight.

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ULTRATHIN LOUDSPEAKERS FROM ALD

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ABSTRACT

Arrays of suspended microbridges (Figures 1 and 2) with sizes as small as 17 μ m \times 1 μ m have been fabricated and demonstrated to produce audible sound using the thermoacoustic effect. The microbridges were fabricated by atomic layer deposition (ALD) of 6.5 nm Al₂O₃ and 19.3 nm tungsten on sacrificial polyimide [1], with ALD performed at 130 °C and patterned with standard photolithography. Loudspeaker sensitivity of these first-generation devices is ~29 dB SPL/1 mW. These devices demonstrate industrially feasible nanofabrication of thermoacoustic transducers and a sound production mechanism pertinent to submicron-scale device engineering.

INTRODUCTION

The development of nanomaterials and nanoscale engineering has enabled new capabilities in nanoscale thermal engineering. These have, in turn, led to a resurgence of interest in thermoacoustic sound production. In this transduction mechanism, Joule heating creates temperature fluctuations in a solid material, heat conduction generates coupled temperature and pressure fluctuations in the surrounding fluid, and the pressure fluctuations propagate away from the device as sound waves. This transduction mechanism was first explored in the early 20th century [2]. Work on porous Si in 1999 [3] and CNTs in 2008 [4] initiated the wave of recent interest in thermoacoustic transducers. In addition to porous Si and CNTs [4]-[9], thermoacoustic loudspeakers have been demonstrated from nanowire arrays [10]-[13], and graphene [14]–[18]. This prior work has suggested the following principles to maximize nanomaterial sound production:

- Minimize substrate losses by suspending devices [11], [17].
 Minimize the state of t
- (2) Minimize the areal heat capacity of the transducer [16], [17]. The devices here have 0.18 J m⁻² K⁻¹ (calculated from Refs. [19], [20]), lower than any sample in Ref. [21].
- (3) Maximize or concentrate heat flux q_0 [J m⁻²] from the transducer (see Eqs. 1 and 2, below) [5], [12].
- (4) Maximize the heat transfer to the surrounding fluid, such as by maximizing the transducer surface area, and distribute power evenly through a sample [21].

Devices using ultrathin (< 50 nm) films produced by ALD allow engineering according to these principles. In particular, ALD provides control of heat capacity and thermal conductivity. Furthermore, ALD is readily integrated in manufacturing, having already been integrated into wafer-scale CMOS processing. It may be expanded to continuous roll-to-roll production, adapted for flexible substrates, and used to produce many materials including insulators and metals. Resolution is limited only by lithography.

Thermoacoustic Physics

Thermoacoustic loudspeakers operate at low voltages, providing an advantage over piezoelectric, electrostatic, and other capacitive acoustic transducers. Very good (85–100 dB SPL in air, 200 dB SPL for encapsulated devices in water) sound production has been measured from thermoacoustic devices [9], [21]. For most audio frequencies, the efficiency η (sound power out/electrical power in) is typically ~10⁻⁶ to 10⁻⁵ at best [5], [10]–[13], [17], [21]. In auditory frequencies these devices typically have a frequency-dependent behavior of sound pressure and



Figure 1: SEM image of suspended microbridges used for thermoacoustic sound generation. Beams are 17 μ m long, 1 μ m wide, 25.8 nm thick and suspended 3.5 μ m above the substrate. Anchor regions provide mechanical connection to the substrate.



Figure 2: Cross-section diagram of the fabricated devices.

efficiency, increasing with frequency f (Eq. 1) until saturating at some maximum level (Eq. 2) [3], [5], [12], [22]. Here, ρ_g is the density of air (~1.2 kg/m³), v_g is the isothermal speed of sound in air (~340 m/s), c_p is the specific heat of air (~1007 J kg⁻¹ K⁻¹), T_0 is ambient temperature (~300 K), and A is the area of a transducer.

$$\eta = \frac{\pi f^2}{4\rho_{\rm g} v_{\rm g} c_{\rm p}^2 T_0^2} q_0 A \tag{1}$$

$$\eta = \frac{q_0 v_g}{2\rho_g c_p^2 T_0^2}$$
(2)

Under certain conditions, i.e. plane wave production and $\omega >> \alpha_s/2L_s^2$ (where $\omega = 2\pi f$, α_s is the thermal diffusivity of the transducer film, and L_s is the distance through which heat propagates to a substrate), the frequency dependence of Eq. 1 may be mitigated, resulting in a relatively flat frequency response [22]. In encapsulated systems, other behavior may emerge such as $f^{-3/2}$ dependence [2] and mechanical resonance [5]. Optimization of these transducers is a balance among efficiency, electrical power consumption, sound output, manufacturability, and packaging.

THERMOACOUSTIC MODEL

The development of practical models for thermoacoustics is an area of ongoing inquiry and optimization [21]. The fundamental physical problem is expressed by coupled temperature

978-1-940470-02-3/HH2016/\$25©2016TRF DOI 10.31438/trf.hh2016.9 Solid-State Sensors, Actuators and Microsystems Workshop Hilton Head Island, South Carolina, June 5-9, 2016 and pressure field equations [12], [23]. Most models take a distributed approach or ignore some of the relevant conduction effects in the transducer materials. With some simplification [5], thermoacoustic sound generation can be interpreted as a source driving a temperature wave, which drives a propagating pressure wave. The thermal wave decays rapidly, with diffusion length μ_i (100 Hz) = ($\alpha/\pi f$) = 268 µm and μ_i (100 kHz) = 8.46 µm) [23], where α is thermal diffusivity of air (2.25×10⁻⁵ m²/s).

Suspended thermoacoustic bridges of finite length have not been thoroughly considered in literature, with the closest models assuming infinite length beams [12]. While this assumption allows assessment of the upper bounds of sound production efficiency (Eqs. 1-3), it ignores effects of thermal conduction within finite beams on the output frequency spectrum. The thermal diffusivity calculated for ALD W beam material is $\alpha_s = 2.25 \times 10^{-6} \text{ m}^2 \text{ s}^{-1} [19]$, Combined with typical thermal conduction lengths of [20]. suspended microbridges, the criterion $\omega >> \alpha_s/2L_s^2$ may be satisfied for low frequencies, such as 20 Hz for 600 µm beams. This suggests that models of thermoacoustic emission from nanoscale beams should account for heat flux through the beams in addition to emitted power. This is accomplished below in a new model for heat flow from thermoacoustic transducers that is distinct from previous models in that (1) it accounts for heat conduction and capacitance in microbridges of finite length, and (2) it does so using a lumped element approach.

Lumped Element Model for Microbridge Sound Production

In each transducer beam, energy production from Joule heating is balanced (Eq. 3) by emission of a thermal wave from the top surface of the beam (Eq. 4), heat conduction and storage in the beam (Eq. 5), and, from the bottom surface, both emission of a thermal wave and conduction through the air to the underlying fabrication substrate (Eq. 6). Here each \dot{Q} term is a lumped heat power flow and harmonic analysis is used to find solutions to these equations, which is a common approach in thermoacoustic analysis. Heat flow from the top surface is given by the 1D thermal wave output, Eq. 4, wherein κ is thermal conductivity of air (0.0263 W m⁻¹ K⁻¹), T_{AC} is the oscillating component of the beam temperature, L_B is the beam length, w_B is the beam width, and k_1 is the thermal wavenumber, given by $k_1 = (j\omega / \alpha)^{1/2}$. Heat loss in the beam is described by Eq. 5, where the thermal resistance $R_{beam} = (L_B / 4) / (\kappa_f \delta w_B)$ and thermal capacitance $C_{beam} = \rho_f c_f w_B$ $L_B/2$. Film thickness is δ , film density is ρ_f , specific heat of the film is c_{f_2} and thermal conductivity of the film is κ_{f_2} . Input power supplied to the transducer beam is a modeled as $P_{AC} = P_0 e^{j\omega t}$. In reality AC Joule heating power can have several frequency components. The power flowing from the bottom of the beam, Eq. 6, is a combination of a thermal wave and heat conduction to the substrate, with R_{gap} approximated as $R_{gap} = g / (\kappa_f L_B w_B)$, where g is the gap distance between the beam and substrate.

$$\dot{Q}_{in} = \dot{Q}_{top} + \dot{Q}_{bottom} + \dot{Q}_{beam} \tag{3}$$

$$\dot{Q}_{top} = -j\kappa k_1 T_{AC} L_B w_B \tag{4}$$

$$\dot{Q}_{beam} = T_{AC} R_{beam}^{-1} - j\omega C_{beam} T_{AC} e^{-j\omega t}$$
⁽⁵⁾

$$\dot{Q}_{bottom} = -j\kappa k_1 T_{AC} L_B w_B + T_{AC} R_{gap}^{-1} \tag{6}$$

Rewriting Eq. 3 using Eqs. 4–6 yields the system model Eq. 7. If P_{AC} is given, this allows calculation of T_{AC} , "piston velocity" u, rms sound pressure p_{rms} , and sound production efficiency η , Eqs.

9–12. Calculation of u uses the continuity and ideal gas equations [5], and assumes sound generated from each beam is distributed across the width d_n that is allocated to each beam in an array (d_n = beam width + beam separation).

$$T_{AC} = P_{AC} \left[-2\,j\kappa k_1 L_B w_B + R_{beam}^{-1} - j\omega C_{beam} + R_{gap}^{-1} \right]^{-1}$$
(7)

$$u = 2\left(\frac{\omega}{k_1}\right) \left(\frac{T_{AC}}{T_0}\right) \left(\frac{w_B}{d_n}\right) \tag{8}$$

$$p_{rms} = \rho_g v_g \frac{|u|}{\sqrt{2}} \tag{9}$$

$$\eta = \frac{\left(p_{rms}^{2}/\rho_{g}v_{g}\right)}{\left(\dot{Q}_{0}/L_{B}d_{n}\right)} = \frac{p_{rms}^{2}L_{B}d_{n}}{P_{AC}\rho_{g}v_{g}}$$
(10)

Although the P_{AC} appears in the denominator in Eq. 10, when Eq. 7 is carried through Eqs. 8–10, the result is that P_{AC} is in the numerator and $\eta \propto P_{AC}$, similar to Eqs. 1 and 2. If the losses of Eqs. 5 and 6 are omitted, Eq. 2 can be recovered from Eqs. 7–10. The sound pressure level (SPL) from one device is related to η using p_{rms}^2 , given SPL = 10 log₁₀ (p_{rms}^2/p_0^2), with reference pressure $p_0 = 2 \times 10^{-5}$ Pa. Figure 3 examines η and SPL under several conditions. When there are no losses through the beam or conduction through air, the sound output is near the maximum of Eq. 2. Losses in the beam maintain a relatively flat output. Conduction through the air to the substrate significantly decreases the low frequency output. In Eqs. 7–10, the only mechanisms to improve η are to increase P_{AC} , increase R_{beam} and R_{gap} , decrease C_{beam} , and to select beam size and spacing to maximize u and T_{AC} .



Figure 3: Near-field model result for applied power of 1 W/cm^2 (device total 0.195 W) from one of the 17-µm microbridge transducers described below. This figure compares the effect on sound production efficiency η of the different loss terms on the right-hand side of Eq. 7.

EXPERIMENT

Device Design and Fabrication

Microbridge arrays were designed such that series and parallel electrical connections among the microbridges would allow a total array electrical resistance of $10 - 20 \Omega$. A 500 nm Al layer was deposited on the W film in order to distribute electrical power throughout the array. One design used 12,000 microbridges 600 μ m × 2 μ m in size. A second variation used 576,000 microbridges 17 μ m × 1 μ m in size. Devices were fabricated on 15 mm × 15 mm Si chips as described in Figure 4.



Figure 4: Fabrication process. (i) Provide a silicon wafer substrate. (ii) Spin-coat a 3.5 μ m polyimide layer. (iii) Perform ALD of alumina (6.5 nm) and tungsten (19.3 nm) at 130 °C. (iv) Pattern the ALD layers using O_2/CF_4 RIE. (v) Deposit and pattern 500 nm of aluminum. (vi) Release the microbridge structures using an isotropic O_2 plasma etch.

Acoustic Characterization

An anechoic chamber with $18" \times 18"$ base and 15" height was built using with $\geq 6"$ of open cell foam surrounding all sides, of which 3" were pyramid-shaped. Current delivered to each transducer array was determined by monitoring the voltage across a 1- Ω resistor in series with the device. Sound waves were recorded (Figure 5) by a calibrated microphone (Dayton Audio EMM-6). Data were collected by an NI USB-6259 with 100 kHz sampling rate, and processed with LabVIEW and MATLAB. Acoustic performance was measured from device arrays with 0 – 10 V supplied by an Agilent 33250A function generator, or unbiased voltage from an audio amplifier (Dayton APA150).



Figure 5: Near-field experimental setup in anechoic chamber.

RESULTS AND DISCUSSION

Under a linear chirp (frequency varies linearly with time), over 50 dB SPL was generated at frequencies above 10 kHz (Figure 6). Measured device arrays with 0.0838 W input power displayed 48.5 dB sound pressure level at 1 cm distance (Figure 7). This is much less than the ~92 dB SPL that the model predicts for this power input. Spherical wave propagation, losses in electrical connections, and other device impedances resulting from the experimental setup account for a portion of this discrepancy and may accommodated with further optimization. For instance, device resistance was higher than designed, on the order of 40 – 100 Ω , largely due to solder connections. Efficiency of these firstgeneration devices ($\eta = \sim 10^{-10}$, Figure 7) is comparable to other thermoacoustic nanomaterial devices but much less than commercial headphones (~ 100 dB per 1 mW, or $\sim 10^{-3}$ for a device with 1 cm² area). Even with no beam and substrate losses, maximum T_{AC} at 1 W/cm² input is 92 K. Clearly the transducers can be driven with much higher power. The microbridge arrays displayed good fidelity, with relatively flat spectral response across the audible range, Figure 8. This may be expected because electrical impedance is due primarily to resistivity, providing an advantage over conventional electromagnetic voice-coil loudspeakers, which have resonance peaks due to inductance and mechanical dynamics.



Figure 6: Spectrogram of time-varying sound output. Electrical input: 50 Hz - 30 kHz unbiased linear chirp. Chip output: 100 Hz - 60 kHz due to frequency doubling from Joule heating. Measured at 5 cm microphone distance (add 14 dB for 1 cm equivalent).



Figure 7: Single tone frequency response measured at 1 cm microphone distance, 0 - 10 V, 5 kHz input.



Figure 8: Spectral output for thermoacoustic devices, compared with a conventional electromagnetic loudspeaker 37 mm in diameter. Sound output spectra in response to white noise electrical input. The conventional speaker displays resonant peaks in the 5 – 25 kHz range. The white noise was supplied to the thermoacoustic device ($R=83.8 \Omega$) as an electrical current of 0.103 A_{rms} , with 0.88 W total. The thermoacoustic device shows relatively broad frequency response, peaking around 25 kHz.

CONCLUSIONS

Planar microbridge array loudspeakers formed using ALD and microfabrication were produced and demonstrated to operate in the audible range, with frequency response clearly distinct from an electromagnetic loudspeaker. Transducers consisted of suspended 25.8 nm thick microbridges subject to Joule heating, producing sound by means of the thermoacoustic effect. This transduction mechanism provides a compact means of generating pressure fluctuations in a fluid, and can integrate readily with other microdevices. The operation of arrays of devices provides resilience against failure of individual microbridges, and demonstrates a macroscale effect from large-scale parallelization of microdevices. Analysis of the thermoacoustic transducers indicates that geometry and materials selection play critical roles in optimization of the efficiency and frequency response.

Although the efficiency of these devices in the audible range is below that of electromagnetic transducers, the frequency spectrum can achieve relatively flat response. The devices are scalable to both large area and nanoscale operation. Modeling predicts near-field sound pressure levels of > 90 dB from thermoacoustic microbridges. Further nanoengineering may allow the devices to reach their ultimate limits, through reduction of heat capacity, and improvement of substrate reflectance. Far field sound levels will improve with use of larger arrays. Device encapsulation may provide an additional mechanism to improve efficiency of audible sound production.

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ROBUST CATALYTIC GAS SENSING USING A SILICON CARBIDE MICROHEATER

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ABSTRACT

This paper reports the first use of a silicon carbide (SiC) microheater for stable low-power catalytic gas sensing. Catalytic combustion of hydrocarbon gases often requires high operating temperatures, which leads to instability in a previously developed low-power polycrystalline silicon (polysilicon) microheater. A silicon carbide microheater has been developed with low power consumption (20 mW to reach 500 °C) and improved stability, exhibiting an order of magnitude lower resistance drift than the polysilicon microheater after 100 hrs of continuous heating at 500 °C and during temperature increases up to 650 °C. When loaded with a high performance catalytic nanomaterial, the SiC microheater-based catalytic gas sensor exhibits fast response and recovery time (<1 s) and improved long-term stability for propane detection. The results show that a simple change of material from polysilicon to polySiC leads to a significant performance improvement of the microheater and the resulting sensor element.

INTRODUCTION

Catalytic gas sensing is important for personal safety and environmental monitoring in industrial and consumer settings. Current technology relies on a heated catalyst material, which promotes the catalytic combustion of the target gas. The resulting increase in temperature is detected by the sensing element, which in most cases is the same as the heater element. Due to the need for heating, commercially available catalytic gas sensors have a high power requirement (>100 mW), which prohibits this kind of sensor element from being battery-powered [1]. Miniaturization of the heater element can reduce the power consumption, but requires development of stable sensor elements and high performance catalytic materials to retain practical applicability.

Advancements in catalytic material design, including the recent development of an ultrahigh surface area, thermally stable boron nitride aerogel support for catalytic nanoparticles, has led to demonstration of fast and sensitive sensor response to combustible gases such as hydrogen and hydrocarbons [2,3]. However, at the temperatures required for effective activation of hydrocarbon combustion reactions (500 °C), the previously developed polysilicon microheater suffers from poor reliability. The robust performance of SiC at high temperature combined with the tunable conductivity and temperature coefficient of resistivity (TCR) makes this material an attractive replacement for polysilicon [4]. Prior demonstration of SiC microheaters have confirmed their suitability for high temperature applications [5,6], including metal oxide-based gas sensing [7], but they have not been applied to catalytic gas sensing.

This paper details the fabrication of a low-power silicon carbide microheater, following nearly the same process flow as the polysilicon microheater. Comparison of the electrical behavior of the two microheaters shows significant improvements in performance when using SiC, including lower resistance drift and full baseline recovery with changing temperatures. When an advanced catalytic material, platinum nanoparticle-loaded boron nitride aerogel, is integrated onto the SiC microheater platform, it can be used for propane detection with fast response and recovery (<1 s) and improved response stability.

FABRICATION

Microheater Fabrication

The microheater platform consists of four microheater elements per chip encapsulated in a low-stress silicon nitride membrane that is released through an anisotropic KOH back-etch (Figure 1a). The entire chip is $3.5 \times 3.5 \text{ mm}^2$. The thin heater and membrane layers provide thermal isolation that keeps the power consumption low (20 mW to reach 500 °C). An inset of one microheater in Figure 1b shows the pink SiC trace. The heated area is found at the thinnest part of the trace to give an approximately $50 \times 50 \ \mu\text{m}^2$ hot zone. The two white Pt/Ti sensing electrodes are not used in this work and are masked with a thin layer of silicon dioxide before sensor testing to avoid interference from the platinum. Figure 1c shows a cross-sectional schematic of the microheater. The closed membrane facilitates deposition of sensing material on top of the microheater. The metal contacts to the heater are made off the membrane to keep them cool and stable.



Figure 1. (a) Optical image of the chip $(3.5 \times 3.5 \text{ mm}^2)$ consisting of four SiC microheaters. (b) Close-up of one microheater. (c) Schematic cross-section of microheater with polySiC encapsulated within back-etched low-stress silicon nitride (LSN) membrane with windows opened for Pt/Ti metal contacts (Pt/Ti).

Polysilicon microheater fabrication is described in detail elsewhere [2,3]. For the silicon carbide microheaters, the basic process flow is maintained and only the deposition and etch chemistries for the silicon carbide steps are changed. Briefly, lowstress silicon nitride (LSN) and silicon carbide films are deposited (Figure 2a), using methylsilane, hydrogen, and dichlorosilane as silicon carbide precursors in a low-pressure (170 mTorr) hot-wall reactor at T = 835 °C with ammonia gas as the dopant source and the wafers in a closed-boat geometry with slots to limit the gas diffusion [8]. The deposited film is polycrystalline cubic 3C-SiC with a thickness of 130 nm and a sheet resistivity of 5,700 Ω /square. To define the microheaters (Figure 2b), the wafer is patterned with photolithography employing photoresist as the mask and reactive ion etching is done with HBr and Cl₂ gases to selectively remove the SiC [9]. Using a transformer coupled plasma system, a high power of 300 W is needed to have an appreciable etch rate (~1.5 nm/s), so the etch is done in 10 s increments to prevent over-heating. A second silicon nitride layer is deposited (Figure 2c) and windows are etched to allow for metal contact to be made to the SiC (Figure 2d). The metal contacts are created through evaporation and lift-off (Figure 2e). Finally, a

978-1-940470-02-3/HH2016/\$25©2016TRF DOI 10.31438/trf.hh2016.10 Solid-State Sensors, Actuators and Microsystems Workshop Hilton Head Island, South Carolina, June 5-9, 2016 back-side window is etched to expose the silicon substrate (Figure 2f), which is etched in a hot KOH bath to release the membrane (Figure 2g). The membrane ends up being approximately 200 nm thick through the LSN layers only.



Figure 2. Silicon carbide microheater fabrication process. (a) Low pressure chemical vapor deposition of a low-stress silicon nitride (LSN) layer and doped SiC layer. (b) SiC is patterned and etched to form the microheaters. (c) A second LSN layer is deposited to encapsulate the microheaters. (d) A window is etched in the top LSN layer. (e) Metal contacts are created through photolithography, evaporation, and lift-off. (f) A window is etched is etched in the back-side of the wafer. (g) The silicon substrate is etched with hot KOH to release the membrane. (h) Catalytic material is deposited on the microheater for catalytic gas sensing.

Microheater Testing

The microheater testing is done with a Keithley 2602A source-meter. LabTracer 2.9 software is used to run the currentvoltage traces. An open-source Java-based program called Zephyr is used for the continuous power and variable temperature tests. The microheater temperature is linearly proportional to applied power in the operating range. Measuring the heater resistance during external heating does not provide an accurate temperature calibration because the colder parts of the heater contribute a nonnegligible portion of the total resistance. Therefore, external heating where the entire chip is at a given temperature does not provide the same resistance as when only the hot zone of the microheater is at that temperature. Instead, temperature calibration of the microheaters is done by powering the heater to glowing and then fitting the emission spectrum with a Planck distribution [10]. The onset of visible glow is found to be ~700 °C which is 21 mW for the polysilicon microheaters and 28 mW for the SiC heaters.

Nanoparticle-decorated Aerogel Synthesis

The platinum nanoparticle-loaded boron nitride aerogel (Pt-BN) synthesis can be found in full detail in Ref. 3. Briefly, graphene oxide sheets are cross-linked into a gel, dried with supercritical CO₂ to retain the high surface area, and pyrolyzed at high temperature for graphitization. The resulting high surface area graphene aerogel undergoes carbothermic reduction in the presence of B₂O₃ vapor and N₂ gas at high temperature (1600-1800 °C) to form the boron nitride aerogel with a specific surface area of 450 m²/g. The platinum nanoparticle loading is done by infiltrating the aerogel with a 0.5 M solution of chloroplatinic acid (H₂PtCl₆) in ethanol and annealing under Ar at 600 °C for 2 hr to thermally reduce the Pt complex to pure Pt metal nanoparticles.

Sensor Fabrication and Testing

For sensor testing, an individual chip is wire-bonded into a ceramic dual in-line package. The Pt-BN is sonicated into suspension in isopropyl alcohol and a few microliters are dropped onto the chip while the microheater is heated to 80 °C to facilitate local deposition at the heated spot. The packaged sensor is exposed to gas in a 1 cm³ chamber. Propane (5% in N₂, Praxair), oxygen, and nitrogen gas flow rates are controlled with Bronkhurst mass flow controllers and Labview. The oxygen content is controlled at 20% and the total gas flow rate is held to 300 sccm. A Keithley 2602A source-meter and Zephyr software are used to control the sensor and record the sensor response. Sensing response is reported as (R-R₀)/R₀ x 100% where R is the sensor resistance during exposure to a given concentration of propane and R₀ is the sensor resistance in dry air only.

RESULTS AND DISCUSSION Microheater Characterization

The current-voltage sweeps of the SiC and polysilicon microheaters are shown in Figure 3. The SiC microheaters have a higher resistance, about 35 k Ω compared to 2 k Ω for the polysilicon microheaters. To reach the same applied power, the SiC microheaters require higher applied voltages, which is why the potential sweep goes to 30 V compared to 5 V for polysilicon. The microheaters also have different power requirements to reach 500 °C (15 mW for polysilicon versus 20 mW for SiC), which is attributable to the higher thermal conductivity of SiC compared to polysilicon [4]. The slight increase in power consumption can be offset through heater duty cycling, where the heater is turned on for only short time periods. The two microheaters have temperature coefficients of resistance that are opposite in sign, with the polysilicon resistance increasing with temperature (+350 ppm/K) and the SiC resistance decreasing with temperature (-700 ppm/K), which is comparable to previous work [11]. The sign of the microheater TCR is controlled by the competing effects of increased phonon scattering in the crystal grain (increasing resistance with temperature) and thermal activation of carriers through the grain interfaces (decreasing resistance with temperature). The magnitude of the SiC TCR is twice as high as the polysilicon, which becomes important for sensitivity during combustible gas sensing because the same change in temperature due to gas combustion results in a larger resistance change from SiC than from polysilicon.



Figure 3. Current-voltage sweeps for (a) SiC and (b) polysilicon microheaters.

When both microheaters are powered to 500 °C for 100 hours (Figure 4), the SiC microheater has an order of magnitude lower drift (-0.6%) than the polysilicon (7%). The higher noise in the SiC microheater may be due to the lower current level used due to the higher resistance. Previous reports have shown a similar long-term positive resistance drift of polysilicon at temperatures around 500 °C [12, 13]. The cause may be dopant diffusion along the

temperature gradient in the polysilicon layer or dopant segregation at grain boundaries. Over the first 100 hours, there is some decrease in the rate of drift and it is possible that with a long "burn-in" or conditioning time, the drift can be mitigated. However, during catalytic gas sensing, the heat of combustion leads to an increase in the heater temperature above the 500 °C baseline, so even with stability in the baseline, the microheater behavior with changing high temperature is the most critical to practical use.



Figure 4. Normalized resistance of SiC and polysilicon microheaters continuously powered to 500 °C *for 100 hours.*

To demonstrate this issue, applying a set of temperature changes between 10 to 150 °C above the 500 °C baseline (Figure 5a) results in major instability in the polysilicon microheater, including history dependence (Figure 5b). The same type of drift behavior is reported previously from a polysilicon heater element when the heater temperature is increased and decreased in a stepwise manner [12]. The authors also found that the resistance at low temperature depends on the high temperature previously reached by the polysilicon heater and the cooling rate back to low temperature [12]. Given how the resistance of the polysilicon microheater changes when it is returned to the 500 °C baseline from various high temperatures, this pattern appears to hold.



Figure 5. (a) Temperature profile applied to the microheaters and resulting (b) polysilicon and (c) SiC microheater resistance.

In contrast to this unpredictable behavior, the SiC microheater displays no such instabilities, showing stable baseline recovery even after being heated to 650 °C (Figure 5c). The wide bandgap and high chemical stability of silicon carbide make it highly resistant to thermally induced resistance drift and the superior choice for a sensing platform to be operated at high temperature.

Sensing Response

With the addition of Pt-BN (Figure 6a), the SiC platform can be used for catalytic gas sensing. Propane, a commonly used fuel, refrigerant, and process chemical, is used to demonstrate the sensor performance. Although the auto-ignition temperature for propane is 450 °C, there is no response without adding a catalytic material like Pt-BN to the microheater. Other researchers have shown that small microheater-based sensors are explosion-proof without the need for expensive packaging [14]. Propane has a lower explosive limit (defined as the lowest concentration capable of producing an explosion) of 2.1% or 21,000 ppm; thus, the sensor is exposed to concentrations below that value.

Figure 6a shows the Pt-BN drop-casted on top of the silicon carbide microheater. TEM images of the Pt-BN show that the average Pt nanoparticle size is 17 ± 6 nm and the lattice spacing is consistent with pure Pt metal [3]. High resolution TEM images also show that the boron nitride aerogel scaffold is 7-10 layers thick [3]. The high specific surface area allows for high nanoparticle loading with minimal support mass, improving the transfer efficiency of the heat generated during hydrocarbon combustion.



Figure 6. (a) Optical image of the Pt-BN catalyst drop-casted on top of the microheater. (b) Pt-BN SiC sensor response to 2% propane over 18 hours of operation compared to Pt-BN on the polysilicon microheater. (c) Response to varied propane concentrations for Pt-BN SiC sensor versus time showing good baseline stability and response reproducibility. (d) Pt-BN SiC sensor response and recovery time for 2% propane ($t_{90} < 1$ s).

The Pt-BN SiC sensor shows good response to propane with a heater temperature of 500 °C (Figure 6c) with a larger sensitivity than Pt-BN on polysilicon [3], thanks to the larger TCR of SiC. The baseline resistance of the Pt-BN SiC sensor shows good stability and the sensor recovers well to the baseline after gas exposure. The gas exposures also show good reproducibility. The sensor detection limit is just below 5,000 ppm propane at this operating temperature. The SiC platform can be used at higher

temperatures, which may allow for an improved limit of detection. Figure 6d shows a close up view of the starred response in Figure 6c when exposed to 2% propane and the subsequent recovery when the gas is switched back to clean air. The times to reach 90% of the full signal levels (t_{90}) for response and recovery of the Pt-BN SiC sensor are very fast (<1 s), comparable to prior reports [2, 3].

The response is highly stable over an 18-hour period of extended continuous operation where the sensor is exposed to clean air and various concentrations of propane (Figure 6b). The response to 2% propane is shown in Figure 6b for clarity, even though the sensor is exposed to varied concentrations during this test. Figure 6b shows a drop-off in response of about 15% in the first 18 hours for Pt-BN on a polysilicon microheater. With the SiC microheater platform, there is no drop in response over the same period of time. Just as prior work demonstrates an improvement in response stability through selection of a more thermally stable aerogel material (boron nitride instead of graphene), this work demonstrates the significant benefit of a thermally stable material in the microheater platform (silicon carbide instead of polysilicon).

CONCLUSIONS

Miniaturized microheater-based catalytic gas sensors can provide low-power consumption, but stable and reliable performance is critical for practical use. Switching from a polysilicon microheater element to one fabricated with polycrystalline silicon carbide allows for much improved performance during high temperature operation. The SiC microheater shows much more stable resistance during continuous heating at 500 °C and during temperature changes from 500 °C up to 650 °C. When a thermally stable catalytic material (platinum nanoparticle-loaded boron nitride aerogel) is added, propane detection with good reproducibility and response stability over 18 hours and fast response and recovery time (<1 s) is demonstrated.

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SCANNING DIFFRACTIVE OPTIC ELEMENTS FOR UNTETHERED EYE-TRACKING MICROSYSTEMS

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ABSTRACT

A microsystem for precise tracking of human eve movements has been developed to meet the stringent requirements of wearable augmented/virtual (A/VR) systems. Eyeglasses retrofitted with the reported microsystem achieve robust tracking with a 120Hz refresh rate and 0.4^o RMS noise within a 20mW power budget. The key component in the present system is a compact (650µm x 500µm) scanning diffractive optic element (DOE) that projects and scans a crosshair pattern over a 45° x 45° range.

INTRODUCTION

Eve Tracking Background

The movement of the human eve reveals a wealth of information about an individual's neurological health, ocular health, state-of-mind, and interests [1]. The study of microsaccadic eye movements has provided notable insight into the mechanisms of the oculomotor pathway [2], and may shed light on the pathogenesis of neural disease. Eye-tracking has enabled augmentative alternative communication (AAC) devices for those lacking speech capability and/or motor skills, and is also emerging as a human-computer-interaction (HCI) peripheral that promises to enhance the immersive experience of VR and AR systems.

The most widely used eye trackers today are video-based image processing systems. Such systems are bulky (>1cm³), expensive (>\$100), power-hungry (>100mW), and slow (~60fps) and have therefore not been broadly adopted in mobile or wearable technology. Although medical-grade systems may obtain 500Hz tracking with 0.5° resolution, these systems are massive, may require head stabilization, and often carry price tags that exceed \$50,000 USD. Eye-tracking sensors must be improved significantly to provide seamless human-computer interaction (HCI) with connected devices in a mobile, wearable context.

Of the >50 commercial V/AR headsets, there are none that offer un-tethered eye-tracking at the time of this writing, because of the high power consumption and computational expense of video-based methods. The present work may alleviate these constraints to enable wireless eye-tracking in mobile devices.

Eve Tracking Microsystem Design

The reported eve-tracking system achieves improvements in size, cost, power consumption, bandwidth and precision through the use of a simple design principle (Fig. 1). A low-irradiance (1-10 μ W/cm²), infrared (850nm), diverging (at ~50mrad) beam of light is steered by a scanner and reflected from the surface of the cornea at a glancing angle (60° to the normal) onto a photodiode. As the eye rotates, the beam tracks the unique point on the cornea that maximizes the received signal at the photodiode. А noteworthy aspect of the system is that the surface area of the photodiode acts as a spatial filter to remove high frequencies from the far-field pattern projected by the scanner. Large aperture micromirrors with flat surfaces that are ideal for imaging systems [3] are therefore not required. Instead, a small (300µm) Fresnel lens that projects a pattern of high spatial frequencies may be used.

Several improvements to the system reported in [4] have augmented the robustness, speed, resolution, and ease of integration of the present system.



Figure 1:Depiction of eye-tracking system geometry integrated within 3D-printed spectacle frame (inset). In this prototype, the VCSEL, scanner, and photodiode are housed in separate modules.

DEVICE GEOMETRY

The devices incorporate Fresnel zone plates that are mounted on 2DOF scanners (Fig.2). The scanner design reported previously is isothermal in one axis only; the scanner reported here consists of opposed serpentine actuator pairs. Upon release, the device remains flat due to the symmetrically opposed torques applied by each pair of actuators. To rotate the reflector, the temperature of one actuator is increased, while the opposed actuator's temperature is decreased proportionally. In [4], a square-root operation was performed on the DC voltages applied to a single isothermal pair; the orthogonal axis was non-isothermal.



Figure 2: Fresnel zone plate scanner with 2 degrees of freedom. Symmetrically opposed serpentine actuators are used to achieve angular deflections while suppressing thermal crosstalk.

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To control a 2D isothermal device, one may require 4 digitalto-analog converters and a controller that performs the square-root operation. Unfortunately, the cost and power consumption of such an arrangement are prohibitive. The present design may be operated using pulse-width-modulation (PWM), enabling an alldigital drive scheme that linearizes electrothermal actuator trajectories while consuming less power. In addition, 2DOF isothermal actuation suppresses the cross-talk between the orthogonal scan axes that was observed in prior work. The configuration in Figure 3 permits the use of 2 PWM channels to control both axes of the device.



Figure 3: Wiring diagram of PWM drive scheme that requires 1 PWM channel per axis of rotation.

The -3dB bandwidth of the scanners has been increased from 170Hz to 300Hz by reducing the thermal impedance between polysilicon heaters and the bimorph materials. The resonant amplitude has increased by 300% due to improved actuation efficiency at higher frequencies, and also due to a significant reduction in viscous damping caused by the DOE.

TRACKING METHODS

A beam with a \sim 1mm waist at the surface of the cornea was scanned by the Fresnel reflector. Tracking in the horizontal direction was performed with a fast linear trajectory (120Hz), while a hill-climbing algorithm was used for position sensing in the vertical direction. The signal captured by the photodiode over the course of a single line scan is shown in Figure 4. This data was obtained using a model eye in an enclosure. The top curve was captured with the eye pointing to the left, while the bottom curve corresponds to eye pointing to the right.



Figure 4:Oscilloscope captures of photodiode signal during a line scan. A peak is captured in each direction of the line scan. The delay between the peaks may be used to estimate gaze direction.

With human subjects, the tracking signal was often lost due to eye blinking or rapid vertical movements, so a complete raster scan of the corneal surface was required for recovery.

Improved tracking

The DOE in Figure 4 employs the superposition of an orthogonal pair of cylindrical Fresnel patterns to project a crosshair (Fig.5). A circular trajectory of the crosshair guarantees that pulses may be captured by the photodiode within every loop; the timing of these pulses correlates to eye position. Edge-triggered measurements offer significant improvements in SNR when compared to voltage measurements that are susceptible to ambient noise and variations in the optical path lengths caused by eye movement. The position noise in prior work was 5⁰ when tracking at 50Hz. The RMS phase noise of measured pulses indicates that flicker noise from ambient fluctuations dominates in unpackaged MEMS devices. Measurements obtained in an enclosure indicate that a packaged device may achieve 0.4^{0} of RMS phase noise while scanning at 120Hz.



Figure 5:2 degree-of-freedom scanner with a payload that consists of 2 cylindrical lens patterns that are arranged orthogonally to project a crosshair.

A compact scanner module was assembled in order to test the performance of the system integrated into a pair of spectacles (Figure 6). A lensed VCSEL projects a beam onto the DOE surface, which is oriented to scan the surface of the cornea at a glancing angle. The alignment tolerances between the VCSEL and micromirror are significantly more relaxed than the configuration reported in [4], and may therefore be readily assembled by hand. The lens may be removed from the VCSEL at the expense of tighter assembly tolerances. Such an optical arrangement may be achieved using wafer-level bonding techniques.



Figure 6, left: depiction of a crosshair sweeping a circular path over the surface of the cornea – peaks are detected at various points in the path. Right: compact scanner module.

The far-field pattern from the DOE contains high spatial frequencies, as measured with a beam profiler (Fig. 7). These features are filtered by the large aperture of the photodetector.

The static and resonant deflections of the crosshair pattern were measured with an IR camera. In [4], the resonant deflection was approximately the same amplitude as the DC deflection, in part because of viscous damping of the large mirror plate with relatively small etch holes. The DOE scanner experiences less drag and therefore achieves a resonant displacement that is 3x greater than the mirror's. In addition, the polysilicon heater placement in the DOE device provides greater efficiency at higher frequencies, due to a lowered thermal time constant.



Figure 6 (a):beam profile of crosshair pattern projected by DOE. (b) static deflection of crosshair pattern captured with an IR camera. (c) resonant deflection of crosshair pattern captured with an IR camera.

A calibration algorithm was developed to map measured eyeposition values onto a 2D coordinate system (i.e. a computer screen). Users were instructed to direct their gaze at several points on the screen, and a multivariate regression was used to extract polynomials to correct all subsequent measurements (Fig.4).

SUMMARY

Conventional methods of eye-tracking have enjoyed a wide range of applications including augmentative alternative communication, video game interaction, and clinical research; however, several barriers may prevent the adoption of video-based eye-trackers by the emerging wearable electronics enterprise. A microsystem based on scanning DOEs has been developed to achieve high-speed, low-power, low-cost, accurate tracking of eye movements.



Figure 7 (a):data from a calibration procedure in which the user is instructed to look at several points on the screen. (b) A multivariate regression is performed on the inner 9 points, resulting in the corrected values of the outer 9 points in red.

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TEXTILE-BASED ELECTROCHEMICAL SENSORS USING EMBROIDERED ELECTRODES

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ABSTRACT

Textile is an inexpensive, widely available material and offers unique advantages for disposable point-of-care diagnostics and wearable biosensors. Here, we report the first demonstration of a textile-based embroidered electrochemical sensor for analytical measurements. We have developed a simple method to generate conductive polyester threads which are used to fabricate flexible, mechanically robust electrodes on textiles. Proof-of-concept measurements of glucose show that this biosensor exhibits good specificity and linearity from 0 mg/dL to 720 mg/dL, which encompasses the clinically relevant concentration range in humans. Additionally, we demonstrate that this sensor can be easily fabricated in arrays using high-volume manufacturing processes for multiplexed measurements. Lastly, we show that this biosensor exhibits excellent resiliency against mechanical stress and good repeatability, which are important features for textile-based biosensors.

INTRODUCTION

Electrochemical sensors are a promising technology for analytical measurements due to their speed, small size, and high sensitivity [1]. Traditionally, electrochemical sensors are fabricated as multilayer laminate assemblies on rigid substrates, such as plastic or glass [2, 3]. However, these materials are too costly for point-of-care testing in the developing world and are difficult to dispose. Alternatively, the emerging era of wearable sensing requires sensor and sensor components that are flexible, robust and can be easily integrated with wearable materials. Textiles represent an excellent class of materials for point-of-care diagnostics and wearable sensing since they are inexpensive and widely used in the manufacturing of garments and clothing. In addition, the continuous contact of clothing with the wearer's skin facilitates the measurement of physiologically relevant data (e.g. pulse, skin temperature) and collection of bodily fluids (e.g. sweat). Furthermore, the large surface area of garments (e.g. undergarments, shirts, pants) provides substantial space for sensor placement at multiple locations and integration of corresponding electronic hardware. Previous efforts in textile electronics, such as woven transistors and printed sensors, have resulted in the development of wearable sensors for measuring skin temperature [4], heart rate [5, 6], and respiration [7]. With respect to chemical sensing, several groups have demonstrated screen-printed electrochemical sensors fabricated on textiles [8-10] and neoprene [11]. Recently, an electrochemical sensor was reported which employs conductive silk yarn woven into the fabric [12]. While this technique offers improved robustness compared with screenprinted sensors, it is limited to simple electrode geometries and substrates that are woven.

Here, we demonstrate a new type of embroidered electrochemical sensor that is mechanically robust, compatible with different types of fabrics, and offers custom electrode geometries. We also show that this embroidered sensor can be used for quantitative measurement of physiologically relevant analytes. Proof-of-concept measurements of glucose reveal that this embroidered sensor exhibits excellent specificity, linearity and repeatability. Furthermore, durability testing shows that this sensor can maintain measurement accuracy after repeated cycles of mechanical deformation, making it a promising candidate for wearable applications.

MATERIAL AND METHODS

Materials and Reagents

Glucose, glucose oxidase, uric acid and L-lactate were purchased from Sigma-Aldrich (St. Louis, MO). Silver/silver chloride (Ag/AgCl) and carbon inks were purchased from Conductive Compounds Inc. (Hudson, NH). Blocker Casein in PBS was purchased from Thermo Scientific (Tustin, CA). Deionized (DI) water was generated using a Barnstead Smart2Pure water purification system. For electrochemical measurements, analytes were resolved in PBS at room temperature. Samples were freshly prepared prior to experiments and remaining biochemicals were used without further purification.

Thread Preparation

Each electrochemical sensor consists of three electrodes, a reference electrode (RE), working electrode (WE) and counter electrode (CE), which are fabricated using conductive threads. Briefly, polyester thread (Brothers International Corp., Bridgewater, NJ) was coated with carbon or Ag/AgCl ink and cured at 120 °C for 40 min. Thread coated with carbon ink was used for the WE and CE, and thread coated with Ag/AgCl ink was use for the RE. Glucose oxidase was immobilized onto the WE by immersing carbon-coated thread in a glucose oxidase solution (645 U/mL) and air drying overnight at room temperature.

Thread Characterization

Optical microscopy (Nikon Eclipse TS100-F trinocular microscope and DS Fi1 camera) and scanning electron microscopy (SEM) were used to characterize the thread coating process. SEM images were captured using a JEOL 6620LV scanning electron microscope at 12 kV and 170× magnification. The electrical properties of the threads were characterized by measuring their electrical resistance using a Fluke 87-V digital multimeter. Threads were cut into 1 m-long pieces and attached to the multimeter using alligator clips.

Sensor Design and Fabrication

Electrochemical sensors were designed using AutoCAD software (Autodesk, Vernon Hills, IL) and converted into an embroidery file using SewArt software (S & S Computing). The digitized image was uploaded to a Brothers SE400 embroidery machine and fabricated on polyester fabric stacked with a stabilizer film (World Weidner, Ponca City, OK). After the sensors were embroidered, the stabilizer was removed and individual sensors were cut and stored at ambient conditions prior to experiments.

Electrochemical Measurements

Amperometric measurements were performed using a Genefluidics electrochemical workstation (Irwindale, CA). For each measurement, 35 μ L of sample was dispensed onto the sensing region using a pipette and a bias potential of -200 mV (across the WE and RE) was used. Data was obtained 1 min after dispensing the sample. All measurements were performed at room temperature under ambient conditions.

RESULTS AND DISCUSSION Thread Characterization

Conductive threads were characterized using optical microscopy and SEM to evaluate the effectiveness of the thread coating process. Optical images of uncoated thread (a) and thread coated with Ag/AgCl (b) are shown in Fig. 1. For the Ag/AgCl-coated sample, we can observe that the entire length of thread is uniformly coated with ink with negligible blotching or defects. Carbon-coated thread also exhibited similar surface coverage. SEM was used to further visualize the thread surface morphology after the coating process. A cross-sectional image of the thread shows that the ink permeates into the fibers at depths of ~ 50 μ m (Fig. 1 (c)). These results also suggest that the enzyme coating process has negligible impact on the surface morphology and the coating thickness.



Figure 1. Optical micrographs of uncoated thread (a) and thread coated with Ag/AgCl (b). Scale bar, 500 µm. (c) Cross section SEM image of thread coated with Ag/AgCl. Scale bar, 50 µm.

The electrical properties of the thread influences the performance of the embroidered electrochemical sensor. Therefore, we measured the electrical resistance of the carbon and Ag/AgCl-coated threads. Ag/AgCl and carbon-coated threads exhibited resistances of ~0.8 Ω /cm and ~140 Ω /cm respectively, which is similar to values reported in literature [12]. To prevent the oxidation of Ag/AgCl ink, we applied flux to the Ag/AgCl-coated threads, which reduced its resistance to 80 Ω /cm.

Sensor Embroidery

The embroidery process is an important part of sensor fabrication since there are several parameters that can affect the sensor performance. A few of these parameters, including the stitch separation distance and stitch length, were optimized to enhance the quality of the electrodes. For instance, decreasing the stitch separation distance resulted in a higher stitch density which improved electrode uniformity. However, using a higher stitch density required a larger amount of thread which increased the electrical resistance of the electrodes. We determined that a stitch separation distance of 0.2 mm and stitch length of 0.5 mm produced consistent uniformity while minimizing the electrode resistance.

Using the optimized embroidery parameters, we successfully fabricated embroidered electrochemical sensors with tightlystitched, discrete electrodes (Fig. 2) allowing for excellent signal consistency and signal-to-noise ratio (SNR). The dashed circle indicates the sensing region, where the sample is dispensed, which encompasses all three electrodes. Fig. 2 also shows that we can easily fabricate sensor arrays for multiplexed measurements using this technique.



Figure 2. A 3×2 embroidered electrochemical sensor array fabricated on polyester. The dashed circle indicates the sensing region. Scale bar, 1 cm.

Sensor Characterization

We briefly investigated the electroactivity of the embroidered electrodes using cyclic voltammetry (CV). CV measurements of 0.1 mM potassium ferrocyanide ($K_3Fe(CN)_6$) in 1 mM of potassium chloride (KCl) were performed at various scan rates at room temperature. The resulting voltammograms are plotted in Fig. 3, which shows a positive correlation between the anodic peak current and scan rate, and good reversibility.



Figure 3. Cyclic voltammograms of 0.1 mM $K_3Fe(CN)_6$ in 1 mM of KCl at various scan rates using embroidered electrochemical sensors. Inset shows the relationship between anodic currents and the square root of the scan rate.

Glucose Detection

To demonstrate the utility of this platform for the detection of physiologically relevant analytes, we use it for amperometric measurements of glucose. Samples were prepared by dissolving glucose in PBS at increasing concentrations from 0-700 mg/dL. 35 μ L of sample was used for each measurement. Due to the high wettability of the sensor, the sample could be fully absorbed within \sim 1 min which also provided sufficient time for a stable electrochemical reaction to be generated. The electrochemical signal from the final 10 sec was averaged and used to generate the dose-response profile shown in Fig. 4. The glucose assay exhibits a

highly linear response over the entire concentration range with a R^2 correlation coefficient of 0.996 and a lower limit of detection of 100 mg/dL. Additionally, the averaged data points exhibit very small standard deviations (SDs < 3%), which demonstrates the high accuracy and repeatability of this assay.



Figure 4. Amperometric measurements of glucose in PBS. Values are averaged over the final 10 sec of the detection signal. Each data point represents the mean \pm standard deviation (SD) of three separate measurements using new sensors.

Assay Specificity

The specificity of the electrochemical sensor was briefly studied by performing independent measurements of glucose (100 mg/dL), lactate (100 mg/dL), uric acid (100 mg/dL) and PBS using glucose oxidase-immobilized sensors. The data is plotted as the SNR (Fig. 5). From this plot, we observe that a significant detection signal is generated only for the glucose sample (SNR = 3.2), while the irrelevant analytes generate SNRs similar to that of the PBS blank control. These results indicate that the sensor exhibits good specificity with minimal interference due to analyte cross-reactivity. They also suggest that this platform can be used for multiplexed detection protocol.



Figure 5. Specificity of the glucose assay. Measurements were performed using glucose, lactate and uric acid diluted in PBS at a concentration of 100 mg/dL. Each bar represents the mean \pm SD of three separate measurements.

Sensor Durability Testing

For textile-based sensors, an important factor that needs to be considered is the influence of mechanical deformation on the detection performance. Specifically, wearable sensors need to maintain accurate and consistent detection response under repeated cycles of deformation caused by the wearer's movement. To mimic this effect, we manually folded and flattened the sensor for up to 100 times and performed measurements at intervals of 5, 10, 25, 50 and 100 bending cycles. By comparing the detection signals from mechanically folded sensors with those from control sensors that didn't undergo bending, we conclude that there is a negligible change in the signal after 25 cycles and a marginal decrease of \sim 9% after 100 cycles (Fig. 6). These results suggest that our embroidered sensor technology will be able to maintain its accuracy and exhibit high reproducibly under instances of repeated deformation, making it suitable for wearable applications.



Figure 6. Amperometric measurements of glucose (720 mg/dL) in PBS following cycles of bending. Each bar represents the mean \pm SD of three separate measurements. Inset shows the mechanical bend testing of the sensors.

CONCLUSIONS

We demonstrate a unique textile-based embroidered electrochemical sensor for quantitative analyte detection. A simple method for generating conductive threads was developed and used to fabricate electrochemical sensors on textile. Cyclic voltammetry measurements showed that this embroidered sensor exhibits good reversibility. For proof of concept, this platform was used for amperometric measurements of glucose in PBS, which exhibited high linearity, repeatability and assay specificity. Studies to assess sensor durability revealed that this embroidered sensor generated consistent, stable and accurate measurements of glucose after repeated cycles of mechanical deformation. Furthermore, this sensor can be fabricated using automated, high-volume manufacturing processes on various types of textiles. Future work will focus on developing sensor arrays for simultaneous measurements of multiple analytes and detection using raw clinical specimens (e.g. serum, blood).

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AN INTEGRATED LOW-COST RADIATION DOSIMETER UTILIZING MICROORGANISM AS RADIATION-SENSITIVE MATERIAL

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ABSTRACT

We present a simple, low-cost, and robust radiation dosimeter that uses a microorganism (yeast) as the radiation sensing material. The radiation damage to the yeast is used to modulate the impedance of a flexible sensor in which a yeast-glucose film constitutes the dielectric between two metallic films. Adding DI water to the sensor after radiation exposure results in the initiation of the fermentation process, the output of which is monitored by measuring the impedance between the electrodes. Radiation damaged yeast cells result in a reduced CO₂ formation, a decreased total amount of carbonic ions in the fermenting solution, and a drop in the electrical conductivity of the medium between the two electrodes. This is the first reported radiation sensor which utilizes microorganism as the radiation-sensitive material, offering a direct biological correlate to the radiation-induced DNA damage. A prototype sensor with dimensions of $18 \times 18 \text{ mm}^2$ offers a maximum sensitivity of 0.154 Ω/Ω_0 decade-rad and an average resolution of 5 rad during the first 10 rad of exposure.

INTRODUCTION

Ionizing radiations are commonly used/encountered in medical imaging, radiation therapy, nuclear power plants, food sterilization, and scientific research. Although the effects of high dose radiation exposure (Hiroshima, Chernobyl, and Fukushima) is well documented [1], long term exposure to lower levels used in medical imaging and other civilian application can also pose significant health risks [2]. Personnel monitoring dosimeters in various forms and shapes have been utilized to track workers in high-risk sites (e.g., hospitals, laboratories, power plants) [3]. Some of these such as film badges [4] and can be considered as the early versions of the wearable devices. More recently, solid-state diodes and RADFETS have made significant penetration into the personnel monitoring market [5, 6]. These devices, however, are expensive and many of them have to be shipped to the manufacturer for readout. Moreover, they only measure the number of ions generated via radiation on their (non-biological) sensing material. As such, they are not a direct indication of the biological damage. A more clinically-relevant dosimeter should be able to measure biological changes in response to radiation in order to better quantify the effective radiation dosage to human tissues and organs.

Living matter undergoes physical (sometimes lethal) damage to their DNA when exposed to the ionizing radiation [7]. If such physiochemical alterations to the DNA can be utilized to measure the absorbed dosage, it can provide an invaluable information regarding the biological damage to the organism. One particular organism that can serve such purpose (radiation surrogate) is the yeast, a eukaryotic microorganism with wide and ancient commercial applications in food and beverage industry. Yeast is a well-studied microorganism having homologous genetic sequence to humans [8, 9]. Moreover, it is a robust material (50 °C, > 250 MPa, 5-8 % humidity) with a long shelf life [10, 11]. Among different yeasts, Saccharomyces cerevisiae yeast has been among the most studied, due to its genomic similarity to mammalian cells. Researchers have also investigated its response to the radiation, according to which, yeast is found to be semi-resistant to ionizing radiation, i.e., not all yeast cells are completely inactivated by a given dose. Rather, radiation exposure to a population of yeast cells impairs their average metabolic activity [12, 13].

To facilitate personal radiation exposure measurements which are more indicative of biological damage, we have developed a lowcost, film-type, disposable radiation dosimeter that utilizes yeast as the sensing material. During the exposure, high energy ionizing radiation damages the DNA chain, inducing lethal or sub-lethal damage to the yeast cells, hence, interfering with their metabolic activity. This can be quantified by monitoring the fermentation rate after exposure to the radiation through the measurement of the produced CO_2 and formation of carbonic acid byproducts.

DESIGN

The schematic and operation principle of the dosimeter is illustrated in Figure 1. The simple but robust film type design is based on aluminum film and double-sided tape. Two aluminum pieces are used to create two parallel-plate electrodes sandwiching a bilayer of two tapes, one coated with yeast and the other with glucose. The tape is patterned with holes to reduce the electrical impedance between the two aluminum electrodes. For measurements, the film is activated by the introduction of moisture (DI water) initiating the fermentation process with ethanol and CO₂ as the byproducts. The CO_2 is then dissolved in the water up to its maximum solubility level (after which bubbles are generated); as it dissolves it creates carbonic acid, which decreases the electrical impedance between the electrodes due to the increase in charge carriers (i.e., H^+ and HCO_3^- ions), Figure 1b. If the film has not been exposed to radiation, then fermentation (and CO₂ dissolution) proceeds at its normal rate, with electrical impedance decreasing rapidly over time. In contrast, if the film has been radiated, the fermentation rate is reduced due to radiation-induced damage to yeast cells; as a result, the rate of CO₂ dissolution is lower, resulting in a lower rate of change of electrical impedance. Thus, the rate of change of impedance (up to the solubility level of CO₂) is representative of the radiation dose to which the yeast are exposed.

FABRICATION

The sensor is fabricated using a rapid prototyping technique and layer-by-layer assembly, Figure 2. First, two metal electrodes are created by laser-machining aluminum tape into "P" shape (electrode area = $10 \times 10 \text{ mm}^2$) using a 1.06 µm-wavelength fiber laser engraving system (PLS6MW, Universal Laser Systems, Inc.), Figure 2a. Next, a layer of double-sided tape (Scotch[®] double sided tape, 3M) is laser machined with a CO_2 laser (10.6 μ m) to create a square with a 5×5 array of millimeter-diameter holes. The holes are one millimeter apart from each other. The holes enable electrical conduits between aluminum electrodes, Figure 2b. Freezer paper is also laser machined into squares of $18 \times 18 \text{ mm}^2$ and used to enclose the materials into a film shape by the lamination process (the hydrophobic side of the freezer paper is used for lamination to seal and package the device at the end). To assemble the sensor, each aluminum tape is sandwiched between a layer of freezer paper (hydrophobic side) and the laser-machined tape layer as shown in Figure 2c. The tape layers are then plasma-treated to improve hydrophilicity for enhanced water penetration/spreading on the entire surface area of the double-sided tape, Figure 2d.

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Figure 1: (a) Illustration of the disassembled sensor structure and cross-section of the device. (b) Fraction of viable yeasts (yellow) is reduced after the radiation exposure (crossed out with red X). Carbonic acid produced by fermenting live yeasts contributes to the decreased resistivity between the electrodes.

After plasma treatment, 10 mg of yeast powder (Fleischmann's[®] Instant Dry[®]) is screen-printed onto the doublesided tape, Figure 2e. The process is repeated on the other tape using 10 mg glucose particles (D-(+) Glucose ≥99.5%, Sigma-Aldrich[®] Co.). Both yeast and glucose powders are ground to make them fine enough for uniform coating to the double sided tape. The outer boundaries of the symmetric freezer papers are then aligned with each other enclosing the double sided layers with yeast and glucose inside, and the sensor is sealed by thermal lamination as shown in Figure 2f. The lamination temperature was controlled to be 380 °C and only applied at the edges to avoid damaging yeast since the yeast can burn inside the sensor at such high temperatures. All edges of the freezer paper are laminated except for one side, where aluminum electrodes are wired out for electrical measurement. This opening is also where water is injected to initiate the fermentation. Fabricated samples are kept refrigerated (3 °C) when not in use to prolong their shelf life. Figure 3(a) is an image of an open sensor just prior to final packaging. Figure 3(b), shows a packaged one, ready to use.

EXPERIMENTAL SETUP

Prior to the radiation exposure, all samples and de-ionized water were kept in refrigerated conditions. To evaluate the effect of radiation on the yeast viability and hence the transducer output, various sensors (5 samples in each group) were exposed to different doses of radiation (0, 10, 100 and 1000 rad) using a Co-60 (1.13 MeV) source. After exposure, each sensor was injected with 0.1 mL of de-ionized water using a 30 G hypodermic needle (yeast concentration inside the sensor to 100 g/L) [14]. The sensors were then connected directly to an LCR meter (LCR-821, GW Instek), and the electrical impedance (Z = R + jX) was measured over time (30 minutes) at the frequency of 1 kHz.



Figure 2. Fabrication process of the film radiation sensor. (a-b) laser machine aluminum $(10 \times 10 \text{ mm}^2)$ and tape $(5 \times 5 \text{ array that}$ are a millimeter apart from each other) using laser engraver; (c) assemble tape and aluminum on hydrophobic side of freezer paper substrate; (d) plasma treat the double sided layer surface to increase the hydrophilicity; (e) deposit yeast powder on top of the double-sided tape side, then repeat the process through (a-d) to make a symmetric glucose layer; (f) sandwich tapes together and seal edges of the freezer paper to package with thermal lamination except the open-end at electrical connection side.



Figure 3. (a) A photograph showing the inside view of the sensor, (b) image of film radiation sensor after lamination.

Commercial baker's yeast (as used in this experiment) can contain other unknown elements (e.g., remnants from nutrients used during culturing) that could change the electrical response during the experiment. To evaluate the effect of such contaminants, we conducted a control experiment by measuring the impedance over time of a sensor maintained at 3 °C for at least 10 minutes; at this temperature yeast remained inactive and the change in resistance was only be the result of the dissolved impurities.

RESULTS AND DISCUSSION

Figure 4 shows the normalized impedance change as a function of time after the activation of the irradiated sensors by adding DI water. The results show two operation regimes. The first one (0-2)minutes) exhibits a drastic drop in impedance within a short time (large negative slope). The second phase, in contrast, shows a more constant impedance over time (zero or slightly negative slope). The two regimes can be attributed to the generation and dissolution of CO₂ during fermentation. Immediately after the onset of fermentation, any CO₂ that is generated is quickly dissolved in the water, creating carbonic acid (and an increase in charge carriers, H⁺ and HCO₃⁻ ions). This condition remains true until the concentration the maximum solubility of CO₂ reaches in water $(1.5 \text{ g CO}_2/\text{kg H}_2\text{O})$ [15]. The transition time can be predicted based on the generation rate of CO₂ as a function of the amount of liquid and yeast in the chamber (assuming glucose is sufficient not be a limiting factor). Previous experiments have revealed a CO₂ generation rate of approximately 5 mL CO₂/min using 3 g of yeast [14]; when scaled to the amount of yeast in the present device (0.01 mg), the rate becomes 0.016 mL CO₂/min. At this rate, the volume of water in the device (0.1 mL) can be saturated with CO₂ (0.076 mL CO_2) within 4.5 minutes. Up until this time, the impedance is expected to be a function of only ion due to the introduction of carbonic acid.

The second regime occurs when no additional CO_2 can be dissolved in the water. At this point, any generated CO2 remains in the gas form (i.e., bubbles), which produce a higher electrical impedance due to their higher resistance and lower permittivity, compared to water. Since the size and position of bubbles over time is a stochastic process, it is reasonable to expect larger variations in electrical impedance in this regime; this trend is visible in the larger error bars of Figure 4 in the latter end of the plots. The exact transition point between the two regimes may be shorter or longer due to specific kinetics variations of the particular yeast used. In our case, the transition point occurs between 3.5 and 6.5 minutes. Therefore, by measuring the rate of change of impedance within one minute of activation, we ensure that the device readings are not affected by the stochastic variations from bubbles, but are instead directly proportional to the generation (and dissolution) of CO₂. After one hour, all the DI water is evaporated or consumed, the impedance approaching its initial value in dry state.

The data from the control experiments (labeled 3°C in Figure 4) confirm that any possible impurities in the yeast do not significantly contribute to changes in impedance. Since the yeast cells are inactive at 3 °C, any change in impedance due to only the impurities would be visible in this data set. However, aside from a slight increase in impedance within the first 30 s (explainable by the positive permittivity change between electrodes with water injection), the impedance does not change significantly compared to the changes observed at room temperature.



Figure 4. Change in electrical impedance of irradiated film rad sensors response to 0, 10, 100, 1000 rad. The temperature controlled experiment was conducted at 3 $^{\circ}$ C while the rest were performed at room temperature.

Figure 5 shows the rate of change of impedance during the initial period, as a function of radiation dose. The data reveal a linear relationship, with the rate of change in normalized impedance after one minute of fermentation being -0.563 min^{-1} , -0.351 min^{-1} , -0.257 min^{-1} , and -0.102 min^{-1} for radiation doses of 0, 10, 100, and 1000 rad, respectively. The average sensitivity of the sensor is $0.154 \Omega/\Omega_0/\text{decade-rad}$ at 1 minute after testing, Figure 5, with an average resolution of 5 rad during the first 10 rad.



Figure 5. A linear characteristic of rate of change in impedance from irradiated sensor at 1 min.

CONCLUSIONS

We have developed a low-cost, wearable, film-type radiation sensor that utilizes yeast as the sensing material, hence, producing an output that can be directly correlated to the DNA damage and cellular inactivation/death. Our impedance-based sensor output is a function of fermentation byproducts (i.e., generated CO₂ and resulting dissolved carbonic acid) of the surviving yeast (S. cerevisiae) after exposure to the ionizing radiation. A prototype sensor with dimensions of $18 \times 18 \text{ mm}^2$ shows a maximum sensitivity of $0.154 \Omega/\Omega_0$ decade-rad. The sensor output clearly delineates two working regimes, with the first one occurring in the
initial few minutes, indicative of the generated ions in the water the result of the fermentation process, and the second one subsequent to the saturation of the water with the dissolved CO_2 , lasting much longer.

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MICROFLUIDIC MICROALGAL BIOREACTOR FOR HIGH-THROUGHPUT SCREENING OF CO2 CONCENTRATION CONDITIONS ON MICROALGAE GROWTH

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ABSTRACT

This paper presents a high-throughput microfluidic bioreactor capable of culturing and screening microalgal strains under different CO₂ envionmental conditions. The present bioreactor consists of a microfluidic microalgae culture unit, a CO₂ concentration gradient generation (CGG) unit, and an in-line optical detection unit for quantifying cell growth. A gas semipermeable membrane is designed between the microfluidic culture and CGG units to control mass transport of CO₂ into the cell culture channels. To validate the workability of this device, *Chlamydomonas reinhardtii* strain CC620 was cultured in the bioreactor and its growth rate under different CO₂ concentrations was quantified. In-line monitoring of cell growth is achieved using both light intensity measurement and colorimetric detection. The present bioreactor system will have a great potential to accelerate microalgae research.

INTRODUCTION

Microalgae are a promising renewable energy source [1]. It is important to understand algal gene regulation and optimize growth conditions (e.g., light intensity and cycle, CO₂, temperature, etc) for discovery of new strains [2]. Current microalgal studies are often conducted in shake flasks, open raceway ponds, and large size bioreactors [3-6]. However, considering the numerous microalgal strains and combinatorial nature of many growth factors, these conventional culture systems appear less suitable for rapid, large scale, high throughput screening and tracking microalgae growth responding to different environmental conditions. Also, due to large size of the conventional systems, it is relatively difficult to achieve spatially uniform environmental conditions inside these bioreactors without sufficient agitation by shaking, rotating, and/or bubbling. Furthermore, in-line monitoring and quantification of algal growth has rarely been achieved in the conventional systems.

Microfluidics has recently been widely researched and applied to enable miniaturization and automation of biochemical processes and analysis for many biological applications [7]. Cell culture on microfluidic devices has attracted much attention in the area of experimental cell biology. Microfluidic bioreactors have also been reported to screen microalgal oil production under different light intensity conditions, showing great promise of employing microfluidic technology to improve microalgae research [8-11]. In this paper, we report, for the first time, the development of a microfluidic algal bioreactor for high-throughput screening of CO2 conditions on microalgae growth. The microfluidic design of the system will bring about several benefits, including parallelizing multiple experiments of culturing microalgal cells under different CO₂ conditions, exposing cells to uniformly spatially distributed CO₂ inside each bioreactor, and automatically monitor cell growth with minimum labor.

DEVICE DESIGN

The microfluidic bioreactor is composed of a microfluidic cell culture unit, a gas concentration gradient generation (CGG) unit, and an optical detection unit for in-line monitoring microalgal growth (Fig. 1a). A gas semipermeable hydrophobic membrane is sandwiched by the culture unit and the CGG unit. This membrane holds the culture medium inside the upper growth channels and allows CO₂ to diffuse from the lower gas channels into the culture medium through the built-in nanopores of the membrane (Fig. 1b). The diffused CO2 is used as carbon source to support photosynthesis of microalgae. To compensate for possible medium loss due to evaporation, two miniature source reservoirs are formed at the inlet and outlet of each culture channel and loaded with fresh pure growth medium. This allows for automatic refilling of the growth channel when any possible medium losses occur at the semipermeable membrane. The device allows for a number of advantages. First, mass transport of CO2 into cell culture medium is faster because of a small diffusion length. Second, spatial distribution of CO2 within each culture channel is uniform because the hydrophobic semipermeable membrane allows CO₂ to diffuse into the channel evenly over the entire culture channel area. Third, different CO₂ concentrations can be flexibly generated, facilitating the study of their influence on cell growth.



Figure 1: (a) Schematic of the proposed microalgal bioreactor. (b, c) and (d, e) are the side view and the top view of a microfluidic cell culture device, respectively.

To realize in-line monitoring of cell growth, a transmitted light intensity (TLI) measurement setup is designed and interfaced with the cell culture device, where 532 nm wavelength laser is used to probe cell growth density. Generally, TLI decreases with increasing cell density. Time course measurement for all the culture channels is realized by placing the device on a programmable motorized stage. Besides, a simple colorimetric method is taken to estimate cell growth by taking pictures for all the growth channels using a digital camera and conducting gray-value analysis (GVA).

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ON-CHIP CELL CULTURE

To demonstrate the workability of the proposed bioreactor, *Chlamydomonas reinhardtii* strain CC620, a wild green microalgae strain, was used as our model microalgae. Its growth under different CO₂ conditions was characterized with the developed system. After loading *C. reinhardtii* strain into the growth channels, the miniature nutrient reservoirs were filled by none-carbon TAP medium and then sealed with polydimethylsiloxane or PDMS based plugs. The device was placed on the motorized stage for in-line TLI monitoring of cell growth. The cells were cultured at room temperature under a 9-watt fluorescent lamp. The distance between the lamp and the device was set to be 30 cm.

Different CO₂ concentration conditions were created using the CGG. To realize this, the two inlets of the CGG were respectively connected to a customized air cylinder with 50 ppm CO₂ and a normal air cylinder with 300 ppm CO₂. The flow rates of the input gas flows were controlled by two mass flow controllers. The eight outlets of the CGG unit were connected to the corresponding gas flow channels of the culture device.



Figure 2: (a) Schematic (cross-section view) of a single cell culture channel. (b) Simulated CO_2 distribution in a gas permeable hydrophobic membrane and the culture medium at t = 10, 100, 250, and 500 s. (c) Normalized CO_2 concentrations at different locations along the height of the growth channel as a function of time. See the locations of #1-#9 in (b).

RESULTS AND DISCUSSION

Fluid dynamic simulations ware taken to study CO₂

distribution in the gas permeable membrane and the culture medium using a finite element analysis (FEA) method based software (COMSOL). In this model, a single cell culture channel consists of three parts: a culture chamber with liquid medium, a semipermeable membrane, and a CO₂ chamber (Fig. 2a). Fig. 2b indicates the fast formation of a spatially uniform CO₂ environment in the culture channel within 300 sec, without using any agitation methods such as shaking, rotating, and bubbling.

Fig. 3a shows the fabricated CGG. Fig. 3b shows the simulated and experimental results of CO2 concentrations at the outputs of the CGG. Here, FEA simulation was performed using the models of laminar flows and transport of diluted species. The generated CO_2 concentrations were measured by a CO₂ meter. The mean values for the measured concentrations at eight outputs of the CGG were 272 ppm, 232 ppm, 211 ppm, 190 ppm, 159 ppm, 118 ppm, 60 ppm, and 50 ppm, matching well with the simulation result (Fig. 3b).



Figure 3: (a) Photo of a microfluidic CGG for generating eight different CO_2 concentrations. (b) Simulated and measured CO_2 concentrations at the outputs of the CGG.



Figure 4: Microscopic photos of C. reinhardtii strain after growing in the channels for 48 hours under different CO2 conditions.



Figure 5: Analysis of C. reinhardtii growth under 8 different CO₂ concentrations over 48 hours. (a) Growth images taken every 8 hours for all the channels at different CO₂ conditions: 272 ppm, 232 ppm, 211 ppm, 190 ppm, 159 ppm, 118 ppm, 60 ppm, and 50 ppm. (b) Gray values for the growth images in all the channels over 48 hours. (c) Gray value reduction rate over 48 hours under CO₂ conditions: 272 ppm and 50 ppm. (d) Strain cells area percentage under different CO₂ concentrations after 48 hours.



Figure 6: (a) Transmitted light intensity measurement setup. 532 nm wavelength laser is used. (b) Result of light intensity transmission under eight CO₂ concentrations in 48 hours at different CO₂ concentrations: 50, 60, 118, 159, 190, 211, 232, and 272 ppm. (c) Light intensity reflection rate under CO₂ concentrations: 272 ppm and 50 ppm.

Fig. 4 shows the growth of C. reinhardtii at 48 hours inside the culture channels of the device under eight CO₂ concentrations. It is obvious that the cell density increased differently, depending on the surrounding CO₂ conditions. To better quantify this environmental impact, we characterized cell growth based on both the GVA using image analysis software (Image J) and the TLI measurement for each channel. Fig. 5a shows the growth images of C. reinhardtii at 0, 8, 16, 24, 32, 40, 48 hours with different CO₂ concentrations. Due to the given difference in CO₂ supplies, the cell density increased at different rates. The higher the CO₂ concentration, the higher the cell growth rate. Specifically, at 48 hours, the gray value of the cell growth image at 272 ppm reduced to 52.5%, while only 42.7% reduction in gray value was found at 50 ppm (Fig. 5c). Another analysis was taken to obtain the percentage of the cell coverage area over the whole channel area after 48-hour culturing. The obtained highest coverage percentage was 50.1% at 272 ppm, compared to the lowest value of 27.19% at 50 ppm (Fig. 5d).

Fig. 6a shows the setup for the TLI measurement. The measured result (Fig. 6b-c) indicates a similar cell growth trend to the GVA result for different CO₂ conditions. Also, the on-chip cell growth results agree well with the plate-based cell growth results, indicating that the present device technology can be suitably used in screening CO₂ conditions for culturing different microalgal species and their strains.

Compared to conventional bioreactors, the volume of the microfluidic culture channel is only less than one milliliter. The small scale of the device will allow for a large number of growth conditions to be tested simultaneously, which is difficult to be done in large batch growth, as it is now utilized. More importantly, the miniaturized device with the gas permeable hydrophobic membrane allows for faster CO_2 mass transportation, and uniform spatial CO_2 concentration.

It should be also pointed out that current study is focused on studying the effect of CO₂ conditions on growth of one single microalgae strain. With minor structural modifications, the microfluidic device will be used to investigate growth of multiple strains under the same environment. Furthermore, in the future, we will build an integrated bioreactor to simulate more natural microalgal growth environments with multiple controllable factors, including pH, light condition, pH value, temperature, and nutrient.

CONCLUSIONS

A microfluidic microalgal bioreactor was developed to study the growth of microalgae under different CO₂ concentrations in parallel. *C. reinhardtii* strain CC620 was characterized using the platform. The system simulated natural growth environment for microalgae and provided a capability of real-time screening, with higher throughput and lower cost than traditional conventional bioreactors. Further, the system could be extended to study other culture factors. It is believed that this system can work as a powerful tool to investigate how algal growth are influenced by environmental conditions and other culture factors at significantly lower cost and shorter time, which would help further development of biorenewable fuel.

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COMBINING SOFT LITHGRAPHY AND PHOTO-POLYMERIZATION FOR ENGINEERED MICRO-ENVIRONMENT

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ABSTRACT

Chemical and physical interactions are commonplace in a natural microbial community. However, it is technically challenging to study natural microbial communities in situ. Engineering artificial environments can offer insights into how natural interactions work both qualitatively and quantitatively. In this paper, we present a method for engineering microenvironments by combining soft-lithography and photopolymerization of hydrogels. This method presents the opportunity to physical isolate micro-organisms while allowing chemical communication between two or more populations.

INTRODUCTION

Interactions of all sorts are ubiquitous in nearly all natural microbial communities including those in the oceans, soil, and human microbiome. Unfortunately there is a dearth of systems that can facilitate controlled experimentation on natural or synthetic microbial communities. Advances in microfluidics provide enabling technologies for this area including polydimethylsiloxane (PDMS) microfluidics, droplet microfluidics, photo-polymerized hydrogels, protein hydrogels, and combinations thereof[1]. Here, we present a method combining photo-polymerized hydrogels with PDMS microfluidics through replica molding. This method enables us to physically isolate bacteria in nanoliter scale chambers while facilitating chemical interactions.

METHOD

The fabrication method, as shown in Figure 1, involves replica molding of a hydrogel from soft-lithography fabricated PDMS molds. The process begins with PDMS soft lithography using SU-8 to create master molds. Then, the PDMS mold is used to pattern a UV-light cured hydrogel [poly(2-hydroxyethyl methacrylateco-ethylene dimethacrylate), HEMA-EDMA]. After the hydrogel is solidified, the PDMS mold can be peeled off.

HEMA-EDMA was chosen because modulating its precursor composition can alter average pore size[2], and thus permeability to various compounds. The average pore size achieved ranges from hundreds of nanometers to a few micrometers. Scanning electron microscope (SEM) is used to qualitatively characterize the average pore size, while dual chamber diffusion experiments are conducted to quantify the diffusivity of various molecules.

A micro-chamber array device was built with nanoporous HEMA-EDMA with the working principle as shown in Figure 2. The device can be used to culture multiple microorganisms, with different organisms occupying different chambers. In such devices, because the pore size of the nanoporous HEMA-EDMA is smaller than the size of the microorganism, the microorganism will be physically isolated in the chambers. However, the porous structure of the chamber walls facilitates chemical communication between microorganisms in different chambers. The overall porosity and the distance between chambers will limit the speed of communication between microorganisms.



Figure 1. Schematic illustration of the fabrication method. 1) Standard photolithography of SU-8. 2) Resulting SU-8 master mold. 3) Casting PDMS to the SU-8 master mold. 4) Resulting PDMS mold. 5) PDMS mold is used to pattern HEMA-EDMA hydrogel. 6) Fabricated NMMI as micro-well arrays of HEMA-EDMA on a glass slide.



Figure 2. Working principle of the HEMA-EDMA micro-well arrays. Individual micro-organisms can be statistically seeded into individual wells or homogeneous populations can be placed in individual wells. The walls of the wells are nanoporous, small enough to physically isolate cells, while still allowing interspecies chemical communication by diffusion.

RESULTS

The HEMA-EDMA in Fig. 3a shows SEM images of microporous HEMA-EDMA while Fig. 3b displays nanoporous

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HEMA-EDMA. The SEM images indicate that pore size of the microporous HEMA-EDMA is larger than 1 μ m, while that of the nanoporous HEMA-EDMA is less than 200 nm. Two representative molecules, glycine (molecular weight 75 Da) and bovine serum albumin (BSA, molecular weight ~66 kDa), are used in the dual chamber diffusion cell to characterize diffusivity across the HEMA-EDMA hydrogel. For nanoporous HEMA-EDMA, glycine diffusivity is 3.47 x 10⁻¹⁰ m²/s, while BSA ~ 0 m²/s. For microporous HEMA-EDMA, glycine diffusivity is 6.03 x 10⁻¹⁰ m²/s, and BSA 1.42 x 10⁻¹⁰ m²/s[3]. This indicates that diffusion of large molecules can be modulated depending upon the application of interest.



Figure 3. Scanning electron microscope (SEM) images of a microporous (a) and a nanoporous (b) HEMA-EDMA membrane. Scale bar in (a) is $5 \mu m$ and in (b) is 200 nm.

As a demonstration of the method, we've create HEMA-EDMA micro-well arrays to culture multiple bacterial strains in physical isolation while maintaining chemical communication, as shown in Fig. 4. The dimensions of each individual well can be as small as 100 μ m x 100 μ m with 25 μ m thick walls, enabling fabrication of roughly 10⁵ culture chambers on a standard 50 x 75 mm² glass slide.

In this particular study, a pair of quorum sensing (QS) strains constructed from *Escherichia coli* K12 are statistically seeded into the micro-well arrays such that initially there is on average one cell per well. The sender strain is transformed to constitutively express red fluorescent protein (RFP) and encode QS molecules (N-Acyl homoserine lactone, AHL) at high local cell concentration. The receiver strain is transformed to express green fluorescent proteins (GFP) upon receiving the AHL



400 µm

Figure 4. Optical microscope image of the fabricated nanoporous HEMA-EDMA micro-well arrays. The scale bar in the image is $400 \mu m$.



Figure 5. Co-culture of a quorum sensing pair of E. coli. The green fluorescence is detected because quorum sensing molecules released from the red sender cells diffuse into the wells containing receiver cells.

compound. Fig. 5 is a composite image of red fluorescence and green fluorescence. It demonstrates that QS molecules diffuse from the red cells to the neighboring wells. When the neighboring wells contain receiver cells, GFP is expressed and green fluorescence is detected.

CONCLUSIONS

In conclusion, we have presented a method of replica molding to create a microfluidic system to cultivate cells and study chemical communication. The replica molding combines PDMS soft lithography and HEMA-EDMA photopolymerization in a simple and scalable process. Using HEMA-EDMA micro-well arrays, we demonstrate that bacterial species can be physically isolated during growth while QS communication can be retained. We envision that this method can be used to engineer complex microenvironments to quantitatively study microbial interactions for both fundamental and industrial purposes. Examples include the study of synthetic microbial communities and the search for previously uncultivated microbes from natural habitats including the soil and the gut microbiome.

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50 nm THICK ALUMINUM NITRIDE PIEZOELECTRIC NANO-PLATE RESONANT THERMAL DETECTORS

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ABSTRACT

This paper reports on the first demonstration of ultra-fast (thermal time constant, $\tau < 10 \ \mu s$) and high resolution (noise equivalent power, *NEP* ~4 *nW/Hz*^{1/2}) thermal detectors based on 50 nm thick Aluminum Nitride (AlN) piezoelectric resonant nanoplates. Efficient piezoelectric transduction of a high frequency (~1 *GHz*) lateral-extensional mode of vibration in a 50 nm thick AlN nano-plate is demonstrated for the first time and exploited for the implementation of the smallest volume (~87 μm^3) laterally vibrating piezoelectric NEMS resonators reported to date. The effects of volume scaling on quality factor, *Q*, temperature coefficient of frequency, *TCF*, and associated detector *NEP* and τ are experimentally investigated.

INTRODUCTION

One of the most important requirements for the implementation of a high resolution and simultaneously ultra-fast MEMS/NEMS resonant infrared (IR) detector is the scaling of the resonator thickness [1]. In fact, the scaling of the device thickness, t, while maintaining the resonator static electrical capacitance, C_0 , constant to a value larger than the substrate parasitic capacitance, inevitably yields an improved IR detector figure of merit: $FoM = 1/(NEP \cdot \tau) \propto (\eta \cdot TCF \cdot Q)/(C_0 \cdot t^2)$, where η is the IR absorption coefficient of the structure. AlN resonant thermal detectors as thin as 500 nm have been recently demonstrated showing excellent thermal detection capabilities (NEP ~0.4 $nW/Hz^{1/2}$, $\tau \sim 20$ ms, FoM ~ 125 $nW^{1}s^{-3/2}$) [2]. Nevertheless, despite such a high performance (comparable to the one of other existing uncooled IR detector technologies) the size of the demonstrated MEMS resonant IR detectors is ~ 1 order of magnitude larger than the one of commercially available microbolometers. This relatively large detector size has fundamentally prevented this technology from addressing the growing needs of small pixel size IR cameras for both military and civilian applications. In this work, we show that, by scaling the resonator thickness to 50 nm it is possible to implement IR detectors with reduced lateral dimensions (down to $32 \times 28 \ \mu m^2$), comparable to the ones of commercially available microbolometers, and characterized by a $FoM \sim 2$ orders of magnitude higher than what previously achieved by MEMS/NEMS resonant thermal detector technologies.

EXPERIMENTAL RESULTS

In this work an ultra-thin (50 nm) and high quality (FWHM $<3^{\circ}$) AlN piezoelectric layer is employed to form the resonant bodies of lateral-extensional mode resonators (Fig. 1) with 5 different lateral dimensions ranging from 60 µm to 28 µm (Fig. 2). Moreover, thicker devices (200 nm and 500 nm) for each size are built as references to prove the effectiveness of volume scaling in improving device performance. The electromechanical performance of both the 50 nm thick devices and reference devices were characterized (Fig. 3, 4), demonstrating that the scaling of the device thickness down to 50 nm allows to maintain sufficiently high values of device capacitance (low impedance) while simultaneously guaranteeing the achievement of high electromechanical performance $(f \cdot Q \sim 10^{12} \text{ and } k_t^2 \cdot Q \sim 10)$ comparable to that of 10X thicker devices. Furthermore, the experiments demonstrate that the use of a 50 nm thick AlN layer

yields higher Q values when the lateral dimensions of the devices are reduced below 50 μm .



Figure 1: 3D schematic representation of the AlN nano-plate resonant thermal detector.



Figure 2: (a) to (c) SEM images of fabricated 50 nm AlN nano plate resonators with various lateral dimensions. Totally 5 different sizes were designed ($60 \times 68 \ \mu m^2$, $59 \times 52 \ \mu m^2$, $50 \times 44 \ \mu m^2$, $41 \times 36 \ \mu m^2$ and $28 \times 32 \ \mu m^2$). The pitch, W_0 , of the top IDE is fixed at 4 μm for all devices. $t_{Al} = 50$ nm, $t_{Pt} = 25/50/100$ nm for 50/200/500 nm AlN and $t_{Au} = 100$ nm. (d) A cross sectional view highlighting the 50 nm AlN thin film deposited on the Si substrate.



Figure 3: Q factor values extracted from MBVD fitting of the fabricated scaled devices with various thicknesses of the AlN nano plate. For lateral dimensions below 50 μ m, the 50 nm thick devices show higher Q than the thicker ones. Low device capacitance and spurious modes prevent measurements of Q values for the smallest 500 nm and 200 nm AlN devices. The extracted k_t^2 (without deembedding) of these devices varies between 1.1% and 0.7%.

The thermal capacitances of the fabricated devices were estimated based on their geometries and material properties (Fig. 4) showing an ultra-low thermal time constant, $\tau \sim 2 \mu s$, for the smallest 50 nm thick device. The thermal resistances, R_{th} of the 50 nm, 200 nm and 500 nm thick devices were estimated based on their anchor geometries and material composition (not optimized in this work) and found to be $\sim 1 \times 10^4 \text{ K/W}$, $0.6 \times 10^4 \text{ K/W}$ and 0.4×10^4 K/W, respectively. The TCF of devices with three different thicknesses were characterized (Fig. 5), showing a high temperature sensitivity ~-52.5 ppm/K for the 50 nm devices (due to the more significant contribution of the metal electrodes characterized by a relatively large temperature coefficient of Young's modulus). These unique features were exploited for the first experimental demonstration of ultra-fast and high resolution AlN NEMS resonant IR detectors. The detectors were characterized using a Pendar Technologies 5 µm quantum cascade laser and the experimental setup shown in Fig. 6. The frequency response to a modulated IR radiation of a 50 nm device was measured and compared to that of a 200 nm one (Fig. 7). The intrinsic absorption peaks of both the 50 nm and the 200 nm AlN devices are located at wavelengths much shorter than 5 μm [3]. Therefore, they are both characterized by small and comparable values of IR absorptance, η , at ~5 μm . The measurement results show a 2X improvement in responsivity ($S \propto \eta \cdot R_{th} \cdot TCF$) for the 50 nm device in agreement with the analytical predictions. The frequency noise spectral densities, f_n , of the two devices were extracted from the measurements and the NEP values were then calculated (*NEP* = f_n/S , assuming $\eta = 1$) to be ~4 $nW/Hz^{1/2}$ for the 50 nm device and ~6 $nW/Hz^{1/2}$ for the 200 nm one. It is worth noting that, these values can be easily reduced to the pW range by optimizing the anchor design [2]. The response times of the two devices were estimated ($\tau = C_{th} \cdot R_{th}$) to be ~10 μs for the 50 nm device and $\sim 16 \ \mu s$ for the 200 nm one. These experimental results demonstrate that the scaling of the resonator thickness enables the implementation of resonant IR detectors with largely improved detection capability (FoM ~2.5×10⁴ nW¹s^{-3/2}), smaller form factor and reduced electrical impedance.



Figure 4: Measured device capacitances and estimated thermal capacitances as functions of the device area. When the device capacitance is smaller than 100 fF, it becomes comparable to the substrate parasitic capacitance deteriorating the effective electromechanical coupling of the resonant element (higher electrical impedance and insertion loss). Thermal capacitances are estimated based on the geometry and material properties of each device. C_{th} as low as 0.2 nJ/K is estimated for the smallest 50 nm device, rendering an ultra-low thermal time constant of ~2 μ s.



Figure 5: Measured TCF for devices with different thicknesses.



Figure 6: Experimental setup for infrared sensing measurements.



Figure 7: Measured frequency responses of a 50 nm device and a 200 nm reference with same lateral dimensions $(60 \times 68 \mu m^2)$ when exposed to a 5 μ m IR radiation modulated at 1 Hz by a chopper.

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HELMHOLTZ PMUT: DEMONSTRATING PASSIVE AMPLIFICATION IN MICROFABRICATED ACOUSTIC TRANSDUCERS

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ABSTRACT

This paper reports the modelling, parametric design, fabrication, and measurement of a piezoelectric micromachined ultrasonic transducer (PMUT) exploiting the Helmholtz acoustic resonance effect to acoustically amplify the electrical response. An analytical model based on electroacoustics was developed and verified by acoustic FEM simulation. The model was applied to generate a set of design parameters, and a Helmholtz PMUT was fabricated and characterized. The device's acoustic response was measured showing an improvement in sensitivity by a factor of 2.16, demonstrating the merits of this approach.

INTRODUCTION

Acoustic transducers with high sensitivity are favored in a variety of applications including ranging, ultrasonic imaging, and acoustic signature detection in noisy environments [1]. This paper demonstrates coupling of a mechanical resonator to a resonant air cavity. Such a geometry employs the Helmholtz resonance effect, as a passive mechanism for amplifying desirable characteristics, such as sensitivity and quality factor. The Helmholtz resonance effect arises in geometries containing a narrow neck leading to a larger volume, such as a glass bottle. Due to the relative compliance of the air in the volume and the incompressibility of the air in the neck, a resonance effect is exhibited where the air volume in the cavity is analogous to a mechanical spring, while the air in the neck of the resonator is analogous to a mass.

To the authors' best knowledge, it is the first demonstration of a fully microfabricated acoustic transducer utilizing the Helmholtz resonance effect for sensitivity enhancement. The addition of an acoustic cavity can be simply and inexpensively implemented to greatly improve the performance of similar acoustic transducers. Packaging is also simplified, since resonances within the packaging become less significant.

DESIGN

The device presented in this paper features a silicon cantilever, with sputter-deposited zinc oxide for piezoelectric sensing, coupled to a microfabricated air cavity. The coupled airacoustic microsystem exhibits acoustic resonance, effectively amplifying the pressure wave which is formed across the cantilever. A schematic representation of the device is shown in Figure 1.



Figure 1: The device schematic with design parameters (a) crosssection view, and (b) top-down view.



Figure 2: Simulated prediction of the acoustic amplification effect, with pressure amplification factor of 3.35.

A parametric model was developed to accurately predict the performance and resonance of such a device. This electroacoustic model, first presented in [2], gives an acoustic transfer function of $\Delta P(\omega) = \frac{R_c + i\omega L_c}{R_c + i\omega L_c}$

$$H(\omega) = \frac{M(\omega)}{P_{in}(\omega)} = \frac{R_G + j\omega L_G}{R_G + j\omega (L_G + L_N) + 1/j\omega C_V}$$
(1)

Where the electroacoustic parameters are related to the geometric parameters by,

$$C_{V} = \frac{V}{\rho c^{2}} \qquad \qquad L_{N} = \frac{\rho(L + \Delta L)}{A_{0}A_{t}}$$
(2,3)

$$R_{G} = \frac{12\eta c}{d^{3}(3D+2d)}, \qquad L_{G} = \frac{0\eta c}{5d(3D+2d)}$$
(4,5)

A multiphysics finite element model (FEM) based on the COMSOL acoustics module was developed for this geometry. An optimized design was derived for the targeted 26 kHz resonance frequency of the air-acoustic system, with the selected dimensions shown in Table 1.

The analytical model and simulation results closely agree in their prediction of resonance frequency, giving a value of 25.8 kHz. The FEM simulation predicts that a sensitivity improvement by a factor of 3.35 can be achieved with the addition of an acoustic cavity (Fig. 2).

Although the analytical model considers volume of the cavity independently from the actual geometry, simulation results indicated that a shallower volume produces a larger Q resonance whose center frequency aligns more accurately with analytical predictions. Figure 3 shows that it is easy to tune the acoustic resonance frequency by adjusting the dimension of the air cavity. In this way we can create arrays of acoustic transducers with varying resonance frequencies. This enables summation of the outputs for increased overall bandwidth or measurement from individual elements for use as a bank of notch filters.

 Table 1: Summary of the selected geometric design parameters.

Parameter	A ₀	t	L	D	d	С	Н
Value (mm)	0.363	0.002	0.4	0.363	0.02	2.7	0.3

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Figure 3: Acoustic frequency response of the device with varying square cavity dimensions.

FABRICATION

The Helmholtz PMUT is fabricated using a two-wafer approach. An SOI wafer with 2 μ m thick device layer (Fig. 4i) is used for fabrication of the cantilever-based piezoelectric sensing element. The SOI's buried oxide layer serves as an etch-stop for KOH backside etching (Fig. 4ii). The sensing layers consist of evaporated Al electrodes, sputter-deposited ZnO, and PECVD SiN for electrical isolation. DRIE is used for the final release of the paddles (Fig. 4ii).

The Helmholtz resonance chamber is fabricated on a second wafer (Fig. 4iv). Silicon nitride serves as an etch mask for KOH anisotropic wet etching of silicon. Upon removing the nitride masking layer, bonding is performed using adhesive SU-8 bonding as described in [3]. It is worth noting that a number of bonding methods can be employed, including eutectic bonding, anodic bonding, thermocompressive bonding or glass frit bonding. The completed wafers are diced, wire-bonded, and packaged (Fig. 5).



Figure 4: Description of two-wafer fabrication process, consisting of (i) conformal nitride deposition (ii) KOH etching, (iii) sensing element deposition and patterning, (iv) cavity etching and bonding.

RESULTS AND DISCUSSION

Device measurements were performed in an anechoic chamber with a GRAS 40AO calibrated microphone serving as a pressure reference. The PMUT output is connected with an Op-Amp based preamplifier circuit and enclosed in an aluminum box for electromagnetic shielding.



Figure 5: Completed device array (left) and bonded device (right).

The sensitivity of the fabricated PMUT was measured both before and after bonding of the Helmholtz air cavity (Fig. 6). The unamplified sensitivity of the PMUT alone is measured to be 0.297 mV/Pa (at 29 kHz with Q of 34.9) and the sensitivity after bonding of the second wafer was 0.64 mV/Pa (at 29 kHz with Q of 73.0). This corresponds to an improvement in sensitivity by a factor of 2.16 with the use of a matched resonant air cavity.



Figure 6: Measured device sensitivity demonstrating amplified sensitivity by a factor of 2.16.

The presented PMUT can be improved further with additional studies into parameter optimization, as preliminary measurements indicated the potential for sensitivity enhancement of up to two orders of magnitude [2].

CONCLUSION

These results support the use of Helmholtz resonance as a mechanism for improving sensitivity of microfabricated acoustic sensors. The amplified sensitivity is achieved with minimal additional cost and processing steps, and can be immediately adopted in a variety of existing fabrication flows of acoustic transducers.

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SCALABLE LIQUID METAL THIN LINE PATTERNING FOR PASSIVE ELECTRONIC COMPONENTS USING SOFT LITHOGRAPHY

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ABSTRACT

This paper presents a size-scalable and residue-free liquid metal patterning technique for all-soft passive electronic components using soft lithography and gallium-based liquid metal (EGaIn, Eutectic Gallium-Indium Alloy). Using the proposed technique, uniform and smooth EGaIn lines with widths ranging from single micrometers to several millimeters are patterned on a PDMS mold at room temperature in ambient pressure. To highlight the process capabilities, the electrical characteristics of fabricated passive components, such as resistors, interdigitated capacitors, as well as vertical interconnects using flexible-through-PDMS vias (fTPVs) are investigated under bending and twisting deformation.

INTRODUCTION

Light-weight, flexible and stretchable electronics have gained significant attention with possible epidermal electronic applications for human health and performance monitoring [1]. One approach to fabricate stretchable electronics is to combine soft substrate materials with conventional metal conductors using wave-like patterns to improve stretchability [1-2].

An alternative approach is to use stretchable conductor materials, such as conducting liquids, to open the path for all-soft, reconfigurable and self-healing electronics [3-4]. Among available liquid conductors, EGaIn is of particular interest due to its excellent electrical and mechanical properties, including high electrical conductivity (3.4×10^6 S/m), low melting temperature (15° C), and low toxicity [4-5]. However, scalable EGaIn patterning ranging from the µm-mm range remains a challenge [4], especially with commonly used techniques such as microfluidic injection, stencil or inkjet printing. Microtransfer molding has recently been demonstrated as promising technology to fabricate micron-scale EGaIn lines [5].

This paper advances microtransfer molding of liquid metals by demonstrating size-scalable, uniform and residue-free EGaIn patterning, and its application to all-soft passive electronic components and circuits (Figure 1).



Figure 1: Concept of all-soft (flexible and stretchable) passive electronic components using gallium-based liquid metal (Eutectic Gallium-Indium Alloy, EGaIn) and PDMS as soft materials.



Figure 2: Scalable thin liquid metal line fabrication process: i) chemical surface modification of PDMS mold, ii)-iii) microtransfer molding of gallium-based liquid metal into PDMS mold, and iv) PDMS cover with contact and flexible through-PDMS via.



Figure 3: Scalable and uniform liquid metal lines patterned using microtransfer molding: (a) cross-sectional view of the EGaIn molded microchannel with 50 μ m width (top) and enlarged view of the molded EGaIn line with 300 nm thickness (bottom), and (b) molded liquid metal lines with widths from 2μ m to 200 μ m.

DESIGN AND FABRICATION

Figure 2 shows the EGaIn patterning process, which utilizes a PDMS mold with embedded microchannels. The surface of the PDMS mold is first selectively chemically modified using toluene for selective wetting. Then, the PDMS mold is pressed onto a donor substrate coated with the liquid metal film, and separated from it. Liquid metal residue on the PDMS mold outside of the channels can now be effectively transferred to a sacrificial PDMS layer. Finally, the PDMS mold with liquid-metal-filled channels is covered with an additional PDMS layer, which has embedded liquid-metal-filled through-PDMS vias for vertical interconnection.

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Using this improved process sequence, EGaIn lines with widths ranging from $2\mu m$ to $200\mu m$ were patterned simultaneously, showing uniform and smooth EGaIn films inside of the PDMS (Figure 3(a)-(b)) and almost no residues between EGaIn features.

RESULT AND DISCUSSION

Figure 4(a) shows an embedded resistor, formed by a resistor line with 5μ m width, connected with 50μ m wide conductor lines. For high-density integration, vertical integration was demonstrated using fTPVs (aspect ratio=1.7-5.7, Figure 4(b)). Figure 4(c) and (d) compare the calculated and measured resistances of the resistors and fTPVs, respectively, with <4% deviation between the calculated and measured values. Figure 5(a) and (b) show an all-soft interdigitated capacitor, having 30 interdigitated electrodes (IDEs), and the measured capacitance as a function of number of IDEs, which agrees well with the simulated values (<6% deviation).

To demonstrate bendability and stretchability, we investigated the electrical characteristics of the embedded resistor by applying bending and torsional forces (Figure 6(a)-(b)). While the resistance increases if the resistor is bend in length-direction, it decreases if bend in width-direction. For a minimal radius of 13.5mm, the measured relative resistance changes were <13%. Upon twisting, relative resistance changes were <3%, indicating that twisting forces have a minor effect because the resistor shape can be maintained.

Finally, a commercial LED was integrated on top of a soft circuit and subjected to bending and torsional forces (Figure 6(c)), demonstrating its electrical functionality under deformation.

CONCLUSION

A size-scalable EGaIn patterning and vertical integration technique is demonstrated to fabricate all-soft passive components and circuits for wearable electronics applications.

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Figure 4: (a) Fabricated soft circuit with serpentine-shaped resistor $(5\mu m)$ and conductor $(50\mu m)$ lines; (b) vertical circuit integration using flexible through-PDMS vias (fTPVs); calculated and measured resistance of (c) conductor line as a function of line length, and (d) fTPV as a function of via diameter.



Figure 5: (a) Liquid-metal-embedded all-soft interdigitated capacitor and (b) simulated and measured capacitance as a function of number of interdigitated electrodes (IDEs).



Figure 6: Resistance change of the embedded resistor soft-circuit to (a) bending and (b) torsional forces, and (c) system-level flexibility demonstration by applying bending and twisting forces to LED-integrated soft circuit.

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TAILORING RADIO-WAVES, LIGHT AND SOUND WITH METAMATERIALS

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ABSTRACT

Metamaterials are artificial materials with unusual bulk properties, based on suitably designed arrays of subwavelength inclusions. They have properties not available in their constituent elements, nor in natural materials at the frequency of interest. These unusual responses are not associated with mixing rules, but they emerge from strong wave-matter interactions, based on carefully engineered meta-atoms and their relative arrangements. In the past decade, metamaterials have opened several exciting directions for basic science, first started in electromagnetics and optics, then expanded into other fields, including acoustics and mechanics. In this paper, I briefly review some recent applications of metamaterials and their potential impact on different engineering frontiers, overcoming long-standing challenges for technology.

INTRODUCTION

The field of metamaterials has seen a continuous and steady growth in the past fifteen years. After initial pioneering work aimed at demonstrating the possibility of negative index of refraction at microwave frequencies [1], resulting from a composite medium with negative real parts of its effective permittivity and permeability, also known as double-negative (DNG) material, the interest has rapidly expanded to the unprecedented electromagnetic properties of these materials, and their potential applications in different engineering fields. These have included the possibility of building ultrathin lenses with subdiffractive resolution properties [2], ultrathin cavity resonators [3] and waveguides [4], and scatterers with unusually large [5] or small [6] cross-section. With the realization of these unusual electromagnetic responses, the interest has rapidly shifted to other fascinating applications, including the possibility of cloaking objects from the incoming radiation [7]-[8], and advancing antenna technology [9]. While the first demonstrations of these unusual concepts were limited to microwave frequencies, the interest in metamaterials expanded in following years to optics, acoustics, and more recently even to other types of waves, including seismic and matter waves.

Among the unusual electromagnetic properties available with metamaterials, also "single-negative (SNG)" materials are popular. These are media for which only one of the material parameters, not both, has a negative real value. This implies that the metamaterial can be properly homogenized and described as a bulk material, which often times requires careful homogenization procedures [10]. SNG media exhibit interesting properties when they are paired in a conjugate manner. These media include epsilonnegative (ENG) media, in which the real part of permittivity is negative but the real part of permeability is positive, and munegative (MNG) media, in which the real part of permeability is negative but the real part of permittivity is positive. When paired together unusual resonances and tunneling arises [11]. The idea of constructing an effective DNG medium by having layers of SNG media has been originally explored by Fredkin and Ron in [12]. Since then, several exciting applications of SNG and DNG materials have been explored in different contexts [13].

In the following, we review a few applications of interest that my group has explored in recent years. I will describe in more detail the impact of these concepts for basic science and engineering applications during my presentation at the conference.

CLOAKING AND RADIO-TRANSPARENT ANTENNAS

Invisibility has drawn the imagination of mankind for several centuries. With the recent developments in metamaterial technology, the possibility of cloaking objects to incoming electromagnetic radiation has been getting close to a technological reality [6]-[8]. While the cloaking concepts keep inspiring scientists and laymen for their fascinating potential, actually cloaked objects can be of importance also in several practical fields of engineering, including radar-camouflaging, radio-transparent devices, and non-invasive biomedical sensing [14]. At the same time, it should be realized that it is inherently challenging to significantly suppress the scattering of objects many wavelengths large with a passive coating [15], as there are fundamental constraints dictated by causality that limit the overall scattering reduction achievable over a certain bandwidth, as a function of the electrical size of the object. In this regard, one of the most viable and realistic application of cloaking technology focuses in the area of radio-frequency cloaked antennas or radiators, whose size is comparable to the wavelength, and therefore it allows significant scattering suppression over large bandwidths. Our group has pioneered this area, proposing several radio-wave antenna cloaking applications [16]-[18], including the possibility of largely suppressing the electromagnetic signature of conventional antennas using suitably designed metasurfaces, while preserving the possibility of transmitting and receiving signals over large bandwidths. Figure 1, as an example, shows a cloaked dipole antenna for cellular communications, highlighting significantly suppressed radar cross section, spanning a large angular range, while maintaining the capability of transmitting and receiving, with the same performance metrics of a bare dipole antenna in the cellular band.



Figure 1: (Adapted from [18]-[19]) A cloaked dipole antenna, designed to transmit and receive radio-frequency signals, while being radio-transparent when excited by an arbitrary external field. On the right: measured scattering gain, defined as the ratio (in dB) between a bare dipole and a cloaked one.

AN INVISIBLE ACOUSTIC SENSOR

Cloaking a sensor or a receiving antenna is an interesting

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application of metamaterials, not only for its impact on engineering applications, but also because it represents a fundamental challenge for basic science. The same action of sensing, in fact, requires extracting a portion of the impinging signal, and therefore it is expected to create a shadow [20]. A way around this fundamental limitation can be realized by considering active metamaterials, or artificial materials that involve active inclusions that can draw energy by a reservoir.

Inspired by recent advances in quantum mechanics in the area of parity-time (PT) symmetry, which studies systems whose Hamiltonian is symmetric under a concurrent parity and timereversal operation, we have recently shown that it is possible to realize a fully invisible acoustic sensor with strong absorption cross-section. The concept was based on pairing a resonant absorbing sensor with its time-reversed image (Fig. 2) [21]. In this device, whose loss and gain are balanced in space, an incoming signal, independent of its amplitude and phase, can be fully absorbed in the first element, yet at the same time the second element produces an exact replica of the incoming signal, eliminating shadows and reflections. The realized system was based on two identical loudspeakers loaded by circuit elements with conjugate impedances. The first loudspeaker was passive, and was designed to convert the impinging sound into a voltage across a resistor. At the same time, the second element was active, with the time reversed impedance of the first element, whose role was to emit a signal in sync with the impinging one, that would suppress the shadow created by absorption. Interestingly, the functionality of this pair of devices may be at the basis of a new class of metamaterials with PT-symmetric properties. We have envisioned fascinating applications of these metamaterials in acoustics and electromagnetics, including the possibility of realizing loss-free negative index, aberration-free planar lenses [22]-[23] and a new generation of advanced cloaking devices that go beyond the limitations of passive cloaks [24].



Figure 2: (Adapted from [19],[21]) An invisible acoustic sensor based on PT-symmetric loudspeakers achieved by loading them with conjugate impedances. This system enables a shadow-free efficient acoustic sensor that can pave the way to loss-free negative-index lenses and active cloaks that overcome the limitations of conventional cloaking technology.

NON-RECIPROCAL MAGNETIC-FREE DEVICES

Sound, radio-waves and light typically travel in space in a symmetric fashion: if we can send a signal from point A to point B,

we can expect to be able to send it back from B to A with equal strength. This inherent symmetry stems directly from Lorentz reciprocity theorem, and it is directly associated with the timereversal symmetry of wave propagation in conventional media. Reciprocity is not necessarily desirable, however, especially when the aim is to isolate a source from its echo, or separate signal flows traveling in opposite directions. An important application of nonreciprocal propagation is for full-duplex communications, i.e., the possibility of transmitting and receiving signals from the same antenna on the same frequency channel, leading to a more efficient use of the radio-wave communication spectrum, as well as better radar systems and ultrasound imaging devices.

The most common way to break reciprocity is based on breaking time-reversal symmetry by biasing ferromagnetic materials with a constant magnetic bias. This approach presents however several challenges: it requires the use of scarce materials, which also lead to heavy and pricy devices, and it is difficult to integrate them on-chip. Recently we have shown that reciprocity can be broken without the need of magnetic bias, replacing it with an angular-momentum bias properly applied to a metamaterial cavity. In our first proof-of-concept demonstration, we applied an angular momentum bias in the form of a slow motion of fluid, to a circularly symmetric acoustic cavity, enabling a first-of-its-kind circulator, or a three-port non-reciprocal device that imparts a preferred sense of circulation to the incoming signal, for acoustic waves (Fig. 3) [25].

The system is a basic three-port device that allows one-way rotation of the input signals, rotating from port 1 to 2, from 2 to 3, and from 3 to 1, while it prevents transmission in the opposite circulation direction. Based on this simple system, we were able to measure very large isolation (over 40 dB) for airborne acoustic waves. The subwavelength acoustic ring cavity shown in Fig. 3 was biased by moving air rotated simply using fans, and the cavity was symmetrically coupled to three acoustic waveguides, which formed the input and output channels of the device.

While it is interesting to see how such a basic component can dramatically modify the way in which sound propagates, not always a mechanical motion of the filling material may be convenient or practical, especially when translating these effects to other types of waves, such as radio signals, which travel much faster than sound. In [26]-[27] these concepts were extended to an equivalent meta-device in which fluid motion was replaced by a suitable form of spatio-temporal modulation of three strongly coupled resonators, which emulates an angular-momentum bias, enabling the realization of magnetic-free circulators for radio and ultrasound waves. We also extended these concepts to light, by considering a microring resonator biased by spatio-temporal modulation imparted via three p-i-n junctions. Using realistic parameters, we were able to design an integrated nanophotonic circulator for light tuned around telecom wavelengths [28].



Figure 3: (Adapted from [25]) A first-of-its-kind circulator for acoustic waves, which enables large isolation and time-reversal symmetry breaking by applying an angular momentum bias to a circularly symmetric cavity. The cavity was loaded with three CPU fans that move air at a moderate velocity, fast enough to break reciprocity and enable large isolation. The cavity is connected to three acoustic waveguides carrying acoustic air-borne signals.

CONCLUSIONS

The field of metamaterials has opened exciting directions in basic research, with a direct impact on several applications, spanning electromagnetics, acoustics, nano-optics, and beyond. Metamaterials have become an important platform for applied technology, with implications at the frontiers of several engineering fields, opening exciting new directions and overcoming several of the conventional limitations of technology that stem from the use of conventional materials.

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EXTREME HOTSPOT HEAT FLUX THERMAL MANAGEMENT VIA THIN-FILM EVAPORATION FROM MICROSTRUCTURED SURFACES

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ABSTRACT

We demonstrate an extreme heat flux thermal management solution targeted towards cooling hotspots with $640 \times 620 \ \mu\text{m}^2$ footprint. Our heat dissipation strategy utilizes thin-film evaporation by incorporating micropillar wicks that allow passive fluidic transport *via* capillarity in addition to maximizing the evaporation area by extending the three-phase contact line. With our wick design, we dissipated $\approx 5.8 \ \text{kW/cm}^2$, the largest heat flux reported to date when compared to past thin-film evaporation studies with similar size hotspots. Our experimental results indicate that thin-film evaporation is a promising thermal management strategy for the next generation microprocessors, power amplifiers and radio-frequency devices, where cooling hotspots is a significant challenge.

INTRODUCTION

Thermal management is a primary design concern for numerous power dense equipment such as microelectronic devices, power amplifiers, electric vehicles, solar energy convertors, and radar and advanced military avionics [1]. These devices generate large amounts of waste heat from sub-millimeter areas during operation. The heat generation can exceed 1 kW/cm² [2, 3] causing hotspots which are localized regions with extreme heat flux and exceedingly high temperature [3]. Furthermore, the heat generation is spatially and temporally non-uniform [4], making the task of removing the waste heat from the device footprint a challenging task for thermal engineers using existing cooling technology [5]. More importantly, the heat generation and the associated rise in junction temperature can adversely impact device performance and reliability [6, 7]. Consequently, many cutting-edge technologies are becoming increasingly dependent upon the ability to efficiently cool hotspots [2, 3].

Traditional air and single-phase liquid cooling solutions use stacks of highly conductive solid-state thermal spreaders such as copper to distribute the heat load over a larger area before ultimately dissipating it *via* convection [8-11]. However, convective air cooling is fundamentally limited by the inferior thermophysical properties of air [1] while single-phase liquid cooling is limited by the low coefficient of performance (COP) due to high pumping power that is required for transporting the working fluid. Furthermore, these systems are not well suited to address extreme heat fluxes (>1 kW/cm²). Consequently, a new paradigm in extreme heat flux hotspot thermal management is required for the advancement of the microelectronic industry.

An efficient strategy to remove chip-level heat loads that exceed 1 kW/cm² for high performance systems is to utilize a phase-change-based approach where the high latent heat of vaporization can be harnessed when the working fluid changes phase from liquid-to-vapor. Furthermore, phase-change-based thermal solutions reduce the size, weight, and overall power consumption making them suitable for microelectronic devices. Such solutions including microchannel flow boiling [7], jet impingement [12], and spray cooling [13], however, are fundamentally limited by instabilities [14]. Accordingly, flow restrictors have been proposed for microchannel flow boiling [15], but lead to significant increase in pumping power which can limit their appeal as a thermal management strategy. A potential phase-change-based cooling strategy for high heat flux devices is thin-film evaporation from enhanced surfaces. Thin-film evaporation [16, 17] has received significant interest in the last few decades due to recent advances in microfabrication technology which facilitated the use of engineered surfaces for heat transfer applications. Thin-film evaporation relies on the phase-change of a thin liquid film from an extended meniscus near the three-phase contact line. The extended meniscus increases the evaporation area, decreases the conduction resistance, and improves the thermal and fluidic transport near the thin-film region [18]. By controlling the thin-film region and the solid-liquid interaction, ultra-high heat fluxes can be dissipated *via* thin-film evaporation.

The present work is aimed at using thin-film evaporation based devices with microstructured wicks for extreme heat flux thermal management. We designed and fabricated test devices that manipulate the solid-liquid interface and the liquid-to-vapor phase transition. The test devices were also designed to closely emulate high performance electronic devices by (a) incorporating multiple hotspots on the same device, and (b) superposing hotspots with background heating. To dissipate this heat, the microstructured wicks relied on capillarity for fluidic transport, *i.e.*, the working fluid was transported to the evaporative area by creating a pressure gradient along the flow direction. The pressures gradient was in direct response to the variation in the meniscus shape along the flow direction and therefore no external pumping power was required for operation. Lastly, by integrating the cooling solution directly on our device, we were able to combine the heat spreader and the heat dissipation technology into a single chip in direct contact with the heat source.

DEVICE DESIGN

Our design leverages microstructured wicks with well-defined silicon micropillar arrays in a square pattern. The overall dimension of the test device was 3×3 cm² with the center 1×1 cm² microstructured area for evaporation (Fig. 1a). We fabricated these devices from a silicon wafer using contact photolithography and deep-reactive-ion-etching. We varied the diameters (D), heights (H), and center-to-center spacings (L) of the micropillars (scanning electron micrograph, Fig. 1b) from 5-12, 40-100, and 12-20 µm, respectively for systematic characterization of fluidic and thermal transport within the micropillar wick. In this work we discuss results for one set of geometry ($D=8 \mu m$, $H=84 \mu m$, and $L=20 \mu m$), however, similar trends were observed across the entire parametric space. The microstructured surface $(1 \times 1 \text{ cm}^2)$ was surrounded by a 6 mm water reservoir (Fig. 1c). The working fluid, de-ionized (DI) water, was passively transported from the reservoir to the evaporative area due to the variation in the shape of the liquid meniscus (i.e., capillary pressure) along the flow direction (Fig. 1c). On the back side of each test device, we incorporated thin-film (100 nm) platinum lines which served both as heaters and temperature sensors (Fig. 1d). The heaters/sensors were calibrated in a convection oven prior to the experiments. The resistance and temperature were linearly correlated with <1% RMS error. To emulate realistic operating conditions in microelectronic devices, each test device incorporated a background heater (H0) which

978-1-940470-02-3/HH2016/\$25©2016TRF DOI 10.31438/trf.hh2016.20 provided uniform heating for the 1×1 cm² microstructured area and three local hotspot heaters (H1, H2, and H3) which provided concentrated heat load over a 640×620 μ m² area (Fig. 1d).



Figure 1: (a) Image of front side of the device. The square and the three circles represent the back side location of the background and hotspot heaters, respectively. (b) Scanning electron micrograph of the microstructured surface. Inset showing pillar diameter (D), height (H), and center-to-center spacing (L). (c) Schematic cross-section A-A showing operating principle of our device. The pressure gradient from the edge to the center of the microstructured area was utilized for fluidic transport (i.e., capillary-wicking). (d) Back side of the device showing thin-film (100 nm) heaters/sensors. The three circles indicate the location of the three hotspot heaters (H1, H2, and H3, each $640 \times 620 \ \mu m^2$) while the square indicates the location of the background heater (H0, 1×1 cm²). The location of the background heater on the back side matches the location of the microstructured area on the front side. The hotspot heaters were located 3 mm apart along the longitudinal centerline. All temperatures were measured at the back side of the test device.

EXPERIMENTAL SETUP

Samples were wet cleaned (acetone, methanol, isopropanol, and DI water), dried using compressed nitrogen gas, and plasma treated (PDC-001, Harrick Plasma) in an oxygen environment for 30 min before each experiment. All experiments were conducted at saturated conditions (\approx 24 °C and \approx 3 kPa) inside a stainless steel environmental chamber ($16 \times 16 \times 16$ in³, Fig. 2). After attaching the different feedthroughs (electrical, thermocouple, and liquid), the system was pumped down using a vacuum pump (Pascal 2010 SD, Adixen) to remove noncondensable gases. The chamber was then allowed to reach saturated conditions by injecting degassed DI water into the chamber via a liquid feedthrough. The chamber temperature was measured by inserting a calibrated J-type thermocouple via a thermocouple feedthrough. All thermocouples were calibrated prior to experiment using a refrigerated water bath heat exchanger (Lauda RE200, Lauda-Brinkmann). A capacitance manometer (740C Baratron, MKS Instruments, Inc.) was used to measure the chamber pressure. High purity DI water (Chromasolv 34877, Sigma-Aldrich) was used for all experiments. The DI water which was degassed via boiling before the experiment was stored in a stainless steel canister outside the environmental chamber. During the experiments, the water inside the canister was maintained at ≈60 °C and ≈20 kPa using a closed-loop temperature feedback system which used a PID controller (CN7800, Omega Engineering). The pressure difference

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between the canister and the chamber (≈17 kPa) was utilized to transport water from the canister to the environmental chamber by overcoming the hydrostatic head and the viscous losses in the valve and tubing. The flow rate of the incoming water to the chamber was measured using a liquid flow meter (L-50CCM-D, Alicat Scientific). The incoming water was actively cooled to ≈ 24 °C before entering the environmental chamber using a water bath heat exchanger (Lauda RE200, Lauda-Brinkmann). During the experiments, the evaporating water vapor was allowed to condense on the internal walls of the environmental chamber. The condensate was removed from the chamber via a drain valve at the end of each experiment. The chamber has visual access and images were acquired at 1000 frames per second using a high speed camera (Phantom v7.1, Vision Research). Data was collected for analysis using a data acquisition system (cDAQ-9174, National Instruments). A DC power source was used to supply the voltage required for heating and temperature sensing via an electrical feedthrough. The electrical voltage and current were measured using a digital multimeter (Model 2000, Keithley Instruments).



Figure 2: Schematic (not-to-scale) experimental setup. The working fluid, DI water, was stored outside the stainless steel environmental chamber in a canister at 60 °C and 20 kPa. The incoming water to the chamber was cooled to the chamber temperature (\approx 24 °C) using a water bath heat exchanger. The T, P, and V indicate the thermocouple, pressure gage, and liquid flow meter, respectively. During the experiments, the water vapor was allowed to condense on the internal walls of the environmental chamber. At the end of each experiment, the condensate was removed via the drain valve and the chamber was made ready for the next experiment.

RESULTS

We experimentally characterized capillary-limited thin-film evaporation with our test devices and experimental setup. We first created a single hotspot using the thin-film heater H2 and simultaneously measured the temperature. The local temperatures 3 mm away from the hotspot were also measured using H1 and H3, while the background temperature (over the microstructured area) was measured using H0. During the experiments, the heat flux was increased in an incremental fashion. Each heat flux was maintained for ≈ 100 s (Fig. 3a). The temporal data was time-averaged to obtain a single data point for the subsequent figures shown here. The heat flux was increased until the hotspot dried out due to liquid starvation (*i.e.*, when the viscous losses exceed capillary pressure). We dissipated $\approx 5.8 \text{ kW/cm}^2$ when the hotspot temperature was $\approx 260 \text{ }^\circ\text{C}$ (Fig. 3a-b). This heat flux is an order of magnitude higher when compared to past thin-film evaporation studies with similar size hotspots [18, 19]. More importantly, the background temperature and local temperatures 3 mm away from the hotspot were <50 °C (Fig. 3b). The measurement uncertainty in our experiments for temperature (±2.5 °C) and heat flux (±0.3 kW/cm²) was estimated *via* standard error propagation analysis by combining the random (statistical, one standard deviation) and system errors [20].

Without background heating, we created multiple hotspots on our test devices and examined the hotspot dryout heat flux. Two hotspots were created by activating H1 and H3 while three hotspots were created by activating H1, H2, and H3. The hotspot temperatures were measured at the location where the heat was generated. For example, when H1 and H3 were active hotspots, the hotspot temperatures at the respective locations were measured using H1 and H3. Our experimental results show that the hotspot dryout heat flux decreased monotonically when multiple hotspots were created on the microstructured surface (Fig. 3c-d). The dryout heat flux which was $\approx 5.8 \text{ kW/cm}^2$ when a single hotspot (H2) was active (red, Fig. 3c), decreased to $\approx 4.2 \text{ kW/cm}^2$ per heater when two hotspots (H1, H3) were active simultaneously (blue, Fig. 3c). This dryout heat flux decreased further to $\approx 2.9 \text{ kW/cm}^2$ per heater when three hotspots (H1, H2, H3) were active simultaneously (black, Fig. 3c). Furthermore, when examining the heat flux as a function of hotspot temperature (Fig, 3c), the results followed a similar trend when one (H2), two (H1, H3), and three (H1, H2, H3) hotspots were active at the same time. For example, when the heat flux was \approx 2.9 kW/cm², the hotspot temperature remained \approx 132 °C (Fig, 3c), whether we activated a single hotspot (H2), two hotspots (H1, H3), or three hotspots (H1, H2, H3). Unlike the hotspot dryout heat flux which progressively decreased as the number of active hotspots increased on the microstructured surface (blue, left ordinate, Fig. 3d), the total heating power increased from 23 W to 35 W (red, right ordinate Fig. 3d) for one hotspot (H2) and three hotspots (H1, H2, H3), respectively.

In actual microelectronic devices, hotspots coexist with background heating. Therefore, we characterized the effect of background heating on the hotspot dryout heat flux (Fig. 3e-f). A single hotspot was created at the center of the microstructured surface by activating H2 while superposing a uniform background heat flux. Background heat flux was provided by uniformly heating the entire 1×1 cm² microstructured area using thin-film heater H0. During experiments, the background heat flux was kept constant and the hotspot dryout heat flux was increased in a stepwise manner until the microstructured surface dried out due to liquid starvation. This procedure was repeated for different background heat fluxes. We observed that, the hotspot dryout heat flux decreased monotonically as background heat flux increased (Fig. 3f). The hotspot dryout heat flux which was $\approx 5.8 \text{ kW/cm}^2$ without background heating (red, Fig. 3e) decreased to $\approx 4.0 \text{ kW/cm}^2$ (blue, Fig. 3e) and $\approx 2.9 \text{ kW/cm}^2$ (black, Fig. 3e) by superposing a 10 W/cm² and 20 W/cm² background heat flux, respectively. Unlike the hotspot dryout heat flux which decreased with background heating (blue, left ordinate, Fig. 3f), the total heating power from the microstructured surface increased from 23 W to 31 W (red, right ordinate, Fig. 3f), when background heat flux increased from 0 and 20 W/cm².

We attribute the high heat fluxes in our study to the micropillar wick design which separates the vapor transport from the liquid transport. Our test devices allowed unimpeded vapor transport by allowing the phase change to occur at the top of the liquid-vapor interface. Furthermore, the micropillar wick surrounding the hotspot region assisted liquid transport by generating capillary pressure owing to the meniscus shape. The capillary pressure was utilized for passive fluidic transport which avoided the need for an external pump. Moreover, we eliminated conventional heat spreaders by incorporating the cooling solution directly on our devices. Even higher heat fluxes can be achieved by optimizing the dimensions of the micropillars, indicating that thin-film evaporation from engineered surfaces is a promising strategy for cooling hotspots.



Figure 3: (a) Temperature as a function of time for a typical experiment. The heat flux was increased in a stepwise manner until dryout. (b) Heat flux as a function of temperature as measured by the different sensors (H0, H1, H2, and H3). A dry island formed at the hotspot H2 (inset, scale bar = 10 mm) when viscous losses exceed capillary pressure in which case the rate of mass loss via evaporation exceeded the rate at which liquid was delivered to the evaporative area. (c) Heat flux as a function of hotspot temperature when one (H2), two (H1, H3) and three (H1, H2, H3) hotspots were activated concurrently. The arrows indicate the hotspot dryout heat flux. (d) The presence of concurrent multiple hotspots decreased the hotspot dryout heat flux (blue, left ordinate), but increased the total heating power (red, right ordinate). (e) Heat flux as a function of hotspot temperature when the hotspot (H2) was superposed with uniform background heat flux. The arrows indicate the hotspot dryout heat flux. (f) When the hotspot (H2) was assisted by a 10 W/cm² and 20 W/cm² uniform background heat flux, the hotspot dryout heat flux decreased (blue, left ordinate), but the total heating power increased (red, right ordinate).

CONCLUSIONS

We experimentally characterized hotspot cooling via thin-film evaporation using well-defined silicon micropillar wicks. Our test devices were designed to supplement spatially distributed hotspots with background heating to closely resemble actual microelectronic devices during operation. Fluidic transport was implemented via capillarity where the working fluid, DI water, was transported due to the pressure gradient along the flow direction. This was facilitated by using microstructured surfaces. Furthermore, the thermal transport near the thin-film region was enhanced by extending the liquid meniscus and three-phase contact line. With our devices, we dissipated $\approx 5.8 \text{ kW/cm}^2$ from a 640×620 µm² hotspot area at $\approx 260 \,^{\circ}\text{C}$ hotspot temperature. More importantly, the average temperature over the entire 1×1 cm² evaporative area and the local temperatures 3 mm away from the hotspot were significantly low (<50 °C). The high heat fluxes attained in our experiments are attributed to the capillarity generated by our tailored microstructured surfaces. The micropillar arrays can be optimized to achieve higher heat fluxes. Our experimental results show that thin-film evaporation is a promising on-chip thermal management approach for cooling hotspots in high performance systems which generate heat loads in excess of 1 kW/cm².

The hotspot dryout heat flux decreased by concurrently creating multiple hotspots which were spatially distributed over the $1 \times 1 \text{ cm}^2$ microstructured surface. Similarly, the hotspot dryout heat flux decreased by assisting the hotspot with background heating. Despite the decrease in the hotspot dryout heat flux, the total heating power increased by spatially distributing the hotspots over the microstructured surface as well as by assisting the hotspot with a uniform background heat flux. The results of this study provide insights into the design of high performance evaporator wicks which are capable of dissipating extreme heat fluxes (>1 kW/cm²). The understanding can be used to improve the design of wicking structures which are commonly used in two-phase thermal management devices such as heat pipes and vapor chambers.

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MULTILAYER ELECTRODEPOSITION FROM MICROMACHINED TEMPLATES: A CONTROLLABLE, SCALABLE ROUTE TO HIERARCHICAL SUPERHYDROPHOBIC SURFACES

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ABSTRACT

We present multilayer electrodeposition guided by micromachined templates as a route to controllable, scalable realization of superhydrophobic surfaces. Sequential electrodeposition of nickel/copper multilayers, followed by a timed, selective copper etching creates metallic, dual-scale hierarchical surface structures, in which microscale pillar structures are defined by lithographically-patterned templates, and nanostructures (50-500 nm) are defined by deposition process parameters. Two types of superhydrophobic surfaces are realized: (1) Metallic surfaces with silane-modified surfaces; (2) and elastomeric surfaces based on polydimethylsiloxane (PDMS) replica molding. Both surfaces exhibit strong non-wetting properties characterized by high static water contact angle reaching over 160° and low contact angle hysteresis (<10°) due to their structural hierarchy. Self-cleaning properties of the fabricated surfaces are demonstrated.

INTRODUCTION

Superhydrophobic surfaces are essential for the survival of many biological systems, such as lotus leaf [1] and water strider [2]. Such non-wetting surfaces are usually characterized by an exceptionally high water contact angle, low contact angle hysteresis, and low sliding angle, i.e., the angle at which a droplet of a certain volume begins to "roll off" from the surface. Superhydrophobicity is useful for many MEMS applications, e.g., improved cooling based on enhanced nucleate boiling [3].

To fabricate superhydrophobic surfaces, applying materials with low intrinsic surface energy, as well as controlling their structural textures are important; even with the materials with extremely low surface energy (e.g., TeflonTM), superhydrophobicity is not achieved if the material is perfectly smooth. Hydrophobic materials with multi-scale structural textures, comprising microscale (1-100 μ m) structures decorated with submicrometer-scale protrusions, form minimized solid-to-water contact area, and thereby exhibit superhydrophobic properties that cannot be achieved from either smooth or textured surfaces with single-scale micro- or nanostructures [1].

Fabrication approaches for hierarchical, superhydrophobic surfaces are usually categorized into three types: bottom-up, topdown, and hybrid. Bottom-up approaches are typically based on colloidal synthesis [4] or sequential material growth/removal [5]. The major advantage of these approaches lies in their simplicity; however, it is relatively difficult to achieve designed wetting properties due to the spontaneous nature of the processes. On the contrary, both micro- and nanostructures can be precisely defined by top-down processes. Sequential lithography steps or pattern transfer processes (e.g., imprinting) are employed to create hierarchical surfaces with designed wettabilities such as surfaces with varying wettability in different orientations (i.e., anisotropic wettability [6]), or at different locations (i.e., surface wettability gradient [7]). However, realization of the nanostructures often requires the use of relatively intensive fabrication techniques, such as electron-beam lithography.

The present approach can be categorized as a hybrid approach, as it combines top-down microlithography and bottom-up multilayer electrodeposition to create structural hierarchy. Lithographically-defined templates are utilized to guide multilayer deposition, thereby creating metallic structures comprised of microscale pillars decorated with concentric ring-like nanostructures, the size scales of which are defined by the individual layer thicknesses of nickel and copper. Surface modification based on silane vaporization, or PDMS replica molding, leads to the realization of metallic or elastomeric hydrophobic surfaces, respectively.

Compared to other hybrid approaches [8, 9], the multilayerbased approach has notable advantages. First, the orientation, location, and geometries of nanostructures are relatively well controlled compared to other bottom-up nanostructure synthesis approaches. Second, controlled nanostructures of high aspect ratios can be formed on the sidewalls of the microstructures, where even the state-of-the-art nanolithography-based approaches (e.g., focused ion-beam lithography, nanoimprinting) usually fail to form desired patterns. Sidewall features can be achieved through deep reactive ion etching of silicon; however, the aspect-ratios of such "scalloping" structures are limited. It is known that well-defined, sidewall nanostructures may substantially improve the robustness (or stability) of non-wetting state of structured surfaces [10]. Third, the present approach provides a route to metallic surfaces with controlled micro/nano features. The relatively robust nature of metal may lead to the use of these surfaces in harsh environments, such as underwater.

FABRICATION APPROACH

The present approach begins with the fabrication of a template comprising a 600 nm-thick sputtered copper seed layer on a sacrificial-layer-coated silicon wafer, and a photoresist mold patterned on the seed (Figure 1(a)). A sequential dual-bath electrodeposition of nickel and copper follows (Figure 1(b)) to form multilayers. Individual layer thicknesses are dictated by control of deposition time, while the deposition currents in the baths are fixed. Typically, current densities of 12 mA/cm² are utilized in both nickel and copper deposition baths, resulting in deposition rates of 2.7 nm/s and 3.3 nm/s, respectively. Further details regarding the deposition setup, multilayer growth modeling, and parameter studies (e.g., deposition characteristics of different types of deposition baths, spatial non-uniformity of the deposition) can be found in [11].

Initially, the multilayer growth is vertically confined within the photoresist mold. After the mold is filled, the growth progresses in three dimensions; the deposition is continued until the deposit is sufficiently thick to cover the photoresist. The layout is properly designed so that the current densities (and corresponding metal deposition rates) do not decrease more than 10% during this second stage of the deposition. The multilayer is mechanically delaminated from the silicon substrate (Figure 1(c)) using tweezers; the copper seed layer is easily detached from the sacrificial polymethylmethacrylate (PMMA) layer due to relatively weak polymer-to-metal adhesion.

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Figure 1. Fabrication procedure. Note the schematics are flipped upside down starting with step (c).

The seed and photoresist layers are sequentially removed in phosphorous-based etchant (Aluminum etchant Type A, Transene) and acetone, respectively. After the template is completely removed from the deposited multilayer, the multilayer "growth rings" are exposed where the concentric, surface relief nanostructures of designed nickel elevation (with respect to copper) are formed after a selective copper etch (Figure 1(d)). A thiourea-based etchant (1M of thiourea dissolved in water, pH tuned to 1 by hydrochloric acid) is used for the etching. The widths of protrusions and valleys of the nanostructures correspond to the thicknesses of nickel and copper, respectively. Large areas of surface relief structures through a single deposition process are achieved; fabricated samples were as large as 20 cm².

It is important to note that both copper and nickel growths are isotropic throughout the deposition. As a result, the orientation of the resulting nanostructure protrusions after the copper etch is nearly normal to the surface (unless an extended period of etching is performed). This is confirmed by a cross-sectional image shown in Figure 2; the individual layers of the multilayer and the patterned photoresist were forming right angles prior to the delamination from



Figure 2. Cross-sectional view of a multilayer prior to the delamination. Growth of the layers is nearly normal to the surface of the photoresist mold. The periodicity of nickel and copper is approximately 600 nm for this sample. Figures (b) and (c) are magnified views of respective sites in (a).

the substrate. Such well-defined surface relief structures enabled PDMS replica molding without de-molding issues (e.g., damaging either metal surface relief or cured PDMS nanostructures), leading to a single-step reproduction of hierarchical, non-wetting surfaces.

Two superhydrophobic surface types are fabricated from the relief structures that are originally hydrophilic. First, metallic superhydrophobic structures are formed by vacuum-assisted deposition of a silane monolayer on the multilayers (Figure 1(e)). Second, PDMS superhydrophobic structures are formed by replica molding using the silane-treated surface relief structures as master molds (Figure 1(f)). Experimentally, we found that PDMS with relatively high modulus should be employed to replicate both microand nanoscale features without de-molding issues. This is achieved by employing either (1) copolymer-based h-PDMS (tensile modulus ~9 MPa)[12], or (2) conventional Sylgard 184 (Dow Corning) with high crosslinker-to-prepolymer weight ratio (e.g., 1:4).

DESIGNED AND FABRICATED SUPERHYDROPHOBIC SURFACES

Hierarchical structures are properly designed and fabricated to realize superhydrophobic surfaces. Among various designs, a pillartype geometry is chosen for the microstructures, in order to minimize the areal fraction of solid-to-water contact. The designed heights of the microstructures (h_{micro}) are larger than half their width (w_{micro}) so that water has more of a chance to form a stable solidwater-air interface "on" the pillars [13]. Two types of structures were fabricated: microstructures alone (designated 'M'), and microstructures decorated with nanostructures (designated 'MN'). The fabricated metallic hierarchical surfaces (Metal, MN) are comprised of micropillars with valleys textured on the nanoscale (Figure 3, Figure 4 (a)-(c)), while the PDMS structures (PDMS, MN) have nanostructures present on the pillar protrusions (Figure 3, Figure 4(a),(d),(e)). Although relatively small periodicities of metallic nanostructures (~ 200 nm) can be achieved (Figure 4(c)), the minimum achievable nanostructure periodicity of replicated PDMS surfaces is approximately 1 micrometer (with moderate aspect ratios ranging from 1:1 to 2:1) due to the difficulties of demolding. Corresponding single-scale microstructures (Metal, M and PDMS, M) are also fabricated for comparison.



Figure 3. Schematic view of designed metal (left) and PDMS (right) hierarchical structures.



Figure 4. (a) An optical image of fabricated metal and PDMS non-wetting surfaces. Low and high magnification scanning microscope images of (b),(c) metal ($w_{Nano}=50 \text{ nm}$, $g_{Nano}=125 \text{ nm}$) and (d),(e) PDMS ($w_{Nano}=500 \text{ nm}$, $g_{Nano}=500 \text{ nm}$) structures.

CHARACTERIZATION

Characterization of the fabricated surfaces is performed based on contact angle analysis. Both static contact angle (SCA) and contact angle hysteresis (CAH, difference between advancing and receding angle) are measured using a goniometer (Model 200, Rame-Hart). Lower SCA and CAH represent smaller energy required to change the position of a liquid droplet, and thus, represents decreased solid-to-liquid friction [14]; this required energy can be characterized by measuring the sliding angle. Superhydrophobicity is often characterized by SCA larger than 150°, CAH smaller than 10° and sliding angle smaller than 10°.



Figure 5. Magnified view of droplets (5 μ L) on respective PDMS surface structures for static contact angle measurements. The "Planar" sample is cured PDMS without any surface textures. The cartoon describes a droplet supported by the micro/nanostructures, forming a Cassie state.



Figure 6. Measured static contact angle (SCA) and hysteresis (CAH) for metal (left) and PDMS (right). Calculated static angles based on Cassie and Wenzel model using equation (1) and (2) are plotted together. Sample dimensions: Metal, M: $w_{Micro}=20 \ \mu m, g_{Micro}=40 \ \mu m, h_{Micro}=18 \ \mu m, PDMS, M: w_{Micro}=15 \ \mu m, g_{Micro}=500 \ nm, g_{Nano}=500 \ nm, h_{Nano}=450 \ nm.$

The static contact angle of a droplet on a textured surface can be modeled using a Cassie (θ^c) or Wenzel (θ^w) model depending on whether air voids are created beneath a droplet, achieving a stable solid-water-air interface, or not (i.e., formation of a complete solidwater interface). The modeled contact angles are calculated using the equations shown below:

Cassie model:
$$\cos\theta^{c} = f(\cos\theta_{0} + 1) - 1$$
 (1)
Wenzel model: $\cos\theta^{w} = r(\cos\theta_{0})$ (2)

where θ_i is Young's contact angle defined on the smooth surface of a material (i.e., measured from a silane-deposited, smooth nickel surface, or a cured PDMS surface (Figure 6)), *r* is the actual, total surface area divided by the apparent surface area, and *f* is the solidliquid contact area divided by the surface area underneath the droplet.

Achieving a stable Cassie state is very important to realize superhydrophobic surfaces, since the reduced water-solid contact area leads to the reduced solid-water friction; consequently, there is more chance for a water droplet to roll off from the surface, resulting in useful properties such as self-cleaning. Based on a thermodynamic analysis, it can be shown that the Cassie state is more likely to be energetically favorable compared to the Wenzel state, and therefore, thermodynamically stable, for surface structures with (1) smaller periodicities and (2) higher aspect ratios [13]; hierarchical surfaces bearing such nanostructures on the microstructures are even more desirable since the water-solid contact fraction is drastically decreased due to the presence of the microstructures while high Cassie state stability is still achieved by the nanostructures [1].

This is confirmed by comparing the measurement and calculation results (Figures 5,6). For both metal and PDMS surfaces, the SCA on the single-scale microstructures falls within the contact angles predicted by the Wenzel and Cassie models. This means a stable Cassie state is not achieved with the single-scale structures even at a low liquid pressure (~100 Pa) induced by a droplet with a small volume (5 μ L). On the other hand, the high SCA of 160° measured from the hierarchical structures corresponds to the Cassie model. The measured CAH from the hierarchical surfaces are much lower than the measurements from the single-scale structures (>80°). The high SCA and low CAH of hierarchical surfaces at which a droplet of a given volume, 10 μ L, begins to roll off from the substrate: 9°-12°) compared to the measurements from the single-

scale structures (>90°, droplets not rolling off even from verticallyoriented surfaces). In summary, both metal and PDMS hierarchical surfaces exhibit superhydrophobicity due to their multiscale architectures.

The utility of the fabricated superhydrophobic surfaces is demonstrated by a droplet impact test and a self-cleaning experiment. Droplets are dispensed from a pipette toward a horizontal, hierarchical surface from a distance (~1.5 cm). Figure 7 captures a single droplet after impact bouncing from the PDMS surface while its spherical shape is preserved. Due to the strong nonwetting nature of the patterned region, the bounced droplet moves toward the edge of the sample where no surface textures are present. Self-cleaning of the hierarchical surfaces is demonstrated by using iron powder (100-200 mesh) "contaminants." After the powders are sprayed on the angled surfaces (Figure 8(a),(b), inserts), water droplets are gently placed on the surfaces. The droplets quickly roll off from the surfaces, removing the contaminants and leaving the clean traces behind (Figure 8(a),(b)). A drastic particle density contrast between the "cleaned" and "uncleaned" areas is observed in the magnified images (Figure 8(c),(d)).



Figure 7. A droplet bouncing on a hierarchical PDMS surface. Note that the droplet shape remains spherical after impact (at 250 ms).



Figure 8. (a) Metallic and (b) PDMS surfaces after water droplets are dispensed and rolled off from the surfaces (Arrows). Figures (c), (d) are magnified images near the edges of the "cleaned" areas.

CONCLUSION

Multilayer electrodeposition guided by micromachined templates was presented as a scalable route to superhydrophobic surface structures. The surface chemistry of the etched multilayers was modified (i.e., silanization) to create metallic superhydrophobic surfaces. Such surfaces were utilized as master molds to produce PDMS superhydrophobic surfaces via replica molding. Both surfaces showed characteristics representative of superhydrophobic surfaces: high static contact angle reaching 160°, small contact angle hysteresis less then 10°, and low sliding angle. Since the geometry of the microstructures, density or aspect ratios of nanostructures, and surface chemistry are widely controllable, this process could be extended to multiple applications, such as spatially anisotropic wettabilities for directional fluid control, or extreme hydrophilicities for fluid wicking.

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FACILE FABRICATION OF FLEXIBLE ELECTRONICS VIA DIRECT LASER WRITING OF CARBON-SILVER NANOCOMPOSITE

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ABSTRACT

In this paper, we demonstrate a facile method for the fabrication of porous-carbon/silver nanocomposites using direct laser writing on polymeric substrates. Our technique uses a combination of CO₂ laser-induced carbonization and selective silver deposition on a polyimide sheet to create flexible highly conductive traces. The localized laser irradiation selectively converts the polyimide to a highly porous and conductive carbonized film with superhydrophilic wettability. The resulting pattern allows for selective trapping of aqueous silver ionic ink solutions into the carbonized regions that are subsequently converted to Ag/C nanocomposite with sheet resistance as low as 0.02 Ω/\Box , upon annealing. The resulting patterns are flexible and electromechanically robust with less than 0.6 Ω variation in resistance after > 15000 flexion bending cycles. Furthermore, using this technique, we demonstrate the fabrication of a wireless resonant pressure sensor capable of detecting pressures ranging from 0 to 97 kPa with an average sensitivity of -26 kHz/kPa.

INTRODUCTION

Flexible and wearable electronics is an emerging field with a wide range of versatile applications, including flexible displays, health monitoring devices, and smart clothing [1]–[4]. Despite the high performance of conventional photo-masking and vacuum deposition processes, limitations such as small printing area, high cost, and high processing temperatures have restricted their use on flexible polymeric substrates [5], [6]. In recent years, many cost-effective direct-writing technologies such as micro contact printing, screen printing, laser-induced forward transfer, inkjet writing, and selective laser sintering (SLS) have been explored as alternative approaches to conventional clean-room processes. These provide attractive features including reduced material wastage and scalable manufacturing.

Among these techniques, inkjet printing and laser-assisted sintering have emerged as preferable approaches. Inkjet printing technology provides a unique non-contact and mask-less patterning process that has been widely used for depositing an extensive number of materials in solution form; these include conductive polymers, CNT, and various metallic nanoparticles [7], [8]. Despite its promise for fabricating flexible electronics, there are still important fabrication challenges associated with synthesis and printing conductive inks. Currently, most conductive inks are based on the suspension of metallic nanoparticles that often exhibit good levels of conductivity only after multiple printing cycles accompanied by high sintering temperatures at 200–350 °C [9], [10]. In addition, suspended nanoparticles tend to agglomerate over time, influencing the print quality and clogging of the equipment nozzle.

The SLS process of conductive metal nanoparticles (NP) has been explored as an alternative approach to inkjet printing. The NP sintering for typical inkjet printing processes is usually implemented by exposing the whole printed substrate to elevated temperatures; SLS is used to selectively localize the heat, thus enhancing the printing resolution and minimizing the substrate thermal exposure [11], [12]. Nonetheless, this method still faces challenges associated with formulation of the binder materials and NP synthesis. Localized high temperatures induced by laser irradiation has also been utilized to selectively convert organic thermoset polymers into conductive carbon materials. This process can generate unique conductive carbon micro-patterns with highly porous micro/nano structures [13], [14]. The high porosity of the carbonized layer can be used to selectively trap precursor aqueous solutions. Despite the low cost of fabrication and stability of the laser carbonization process, previous reports have shown high electrical sheet resistance (typically between 15 Ω/\Box to $1000 \Omega/\Box$) and sensitivity to mechanical deformations [15], limiting its use in flexible electronics, which often require highly conductive and robust interconnections.

In order to further advance the direct-writing lasercarbonization technique and overcome some of the abovementioned shortcomings, we have developed a simple method to utilize the super-hydrophilicity of the carbon traces to selectively synthesize and trap silver nanoparticles into the carbonized regions, significantly enhancing the electrical conductivity and robustness of the flexible interconnects.

FABRICATION AND CHARACTERIZATION

The presented patterning technique relies on the laser pyrolysis of a thermoset polymeric substrate serving as the flexible support to create highly porous network of conductive carbon traces. These traces are later selectively coated with an aqueous reactive silver ink, as shown in Figure 1a-c. In this process, commercially available polyimide (PI) sheets (Kapton[®] HN, with a thickness of 60 μ m) are used as the substrate for carbonization. The process begins by laser writing of highly porous carbon patterns into the surface of the PI sheet using a computercontrolled CO₂ laser cutting and engraving system (PLS6MW,



Figure 1: Fabrication process of carbon/silver nanocomposite using laser pyrolization and selective silver trapping.

Universal Lasers, Inc., Scottsdale, AZ). For maximum conductivity of the carbonized patterns, the laser is operated at 6.75 W with a scanning speed of 1.3 m/s. The laser modifies the surface of the polymer and creates porous super hydrophilic carbon patterns. The selective coating is carried out by loading the porous carbon traces with a reactive silver aqueous solution as shown in Figure 1b, followed by an annealing step at 90 °C for 10 min, Figure 1c.

Due to the hydrophilicity and wicking capability of the highly porous carbonized areas, the solution is only retained in the laserablated regions and rolls way from other areas on the polyimide substrate. The ionic silver ink is prepared by mixing 4 g of silver acetate with 10 mL of ammonium hydroxide. Next, under continuous magnetic stirring condition, 1 mL of formic acid is dropwise titrated into the mixture at room temperature. During the titration process, the solutions color changes from light yellow to a clear. Owing to low viscosity of the silver ink and the strong capillary force of the laser-carbonized traces the ionic silver ink can easily penetrate into the carbon network, allowing for the formation of a homogenous silver layer. Figure 2a-c shows examples of different carbon patterns before and after silver coating. Figure 2d shows an example of a simple flexible circuit made by conductive Ag/C-PI lines and used to light an array of LEDs.

Qualitative characterization

Optical and SEM images were used to evaluate the morphology of laser carbonized microstructures before and after silver deposition, Figure 3. Microscopic images show a clear change in the color from black to silver after the selective silver coating, Figure 3a,b. The SEM image shows the creation of a highly-porous network of carbon micro/nano structures generated by laser carbonization of the polyimide substrate. The alignment of



Figure 2: (a) laser carbonized traces before and after decorating with silver nanoparticles: (i) silver ionic solution, (ii) pristine carbonized trace, (iii) carbon/silver nanocomposite; (b, c) flexible traces before and after silver deposition; (d) array of lit LEDs with flexible interconnect. All scale bars: 1 cm.



Figure 3: Magnified optical (a, b) and SEM (c, d) of carbonized polyimide and carbon-silver nanocomposite. All scale bars: 100 µm.

the carbon network is due to the scanning motion of the laser across the sample, Figure 3c. Figure 3d shows the top SEM image of the carbon trace after the precipitation of silver, illustrating a uniform deposition, forming a highly conductive silver/carbon nanocomposite.

Electrical characterization

Figure 4a shows the sheet resistance of the pristine carbonized polyimide and carbon-silver nanocomposite as a function of the annealing temperatures for samples of 3 cm length and 500 µm width (annealing time was fixed at 10 min). The pristine carbonized polyimide exhibits a maximum sheet resistance of 52 Ω /sq, which is consistent with previous laser carbonization reports [15]. As anticipated, the resistance of the silver-coated carbon traces decreases with increasing annealing temperatures. After drying the Ag/C nanocomposite at room temperature, the sheet resistance decreases slightly to 40 Ω /sq. Increasing the annealing temperature up to 90 °C improves the conductivity, with the sheet resistance decreasing to 0.026 Ω /sq. For temperatures above 90 °C, the effect of annealing on the conductivity is not significant (reactive silver ink is completely converted to bulk silver at 90 °C). Figure 4b, shows the change in the resistance of laser carbonized traces before and an after silver coating at as a function of trace widths ranging from 200 µm to 2 mm at fixed annealing temperatures and times of 90 °C and 10 min. As can be seen, the composites traces show a resistance approximately three orders of magnitude lower than that of the pristine carbonized polyimide traces.

The electrical conductivity between the two ends of the carbonized PI is provided by the connection between carbon particles throughout the material. Mechanical deformation separates the connection points, resulting in an increase in resistance. However, the Ag/C nanocomposite has highly conductive Ag nanoparticles distributed in the carbon network, resulting in a higher conductivity and fewer disconnections between the particles in response to mechanical deformation. To assess the mechanical stability of the conductive traces upon bending, samples with different widths ranging from 200 μ m to 2 mm were subjected to flexion at curvature radii (CR) of 50 mm to 2 mm. The mechanical stability of the conductive composite was characterized with samples subjected to the optimal annealing temperature of 90 °C. Figure 5a shows the change in the carbonized polyimide resistance as a function of CR. As can be



Figure 4: (a) Electrical sheet resistance of carbon-silver composite after annealing at different temperatures, (b) carbonized polyimide and carbon-silver nanocomposite resistances as a function of line width

seen, for small traces (200 μ m), the change in a resistance starts at 30 mm CR with a relatively linear sensitivity coefficient of 0.35 (R/Ro) per degree radius of curvature. However, with increasing width, the starting threshold decreases. For instance carbon traces 2 mm wide exhibit a resistance change starting at 10 mm of CR with a sensitivity of 0.04 (R/Ro) per degree radius of curvature. This observation can be explained by the fact that wider traces have more connecting points throughout the network, resulting in a higher conductivity and fewer disconnections between the particles in response to mechanical deformation.

In addition to a significantly lower resistance, the carbonsilver nanocomposite shows greater robustness and lower sensitivity to mechanical deformation, Figure 5b. For instance, after silver coating, a 200 µm-wide trace with an initial resistance of 7.5 Ω shows less than 12 % change in response to CR as small as 2 mm; this translates to a 900 % decrease in sensitivity to mechanical deformation as compared to a similar carbonized polyimide trace. Furthermore, for traces wider than 400 µm, carbon-silver nanocomposites have a high level of conductivity, with resistance smaller than 3 Ω and less than 9 % change in resistance at the maximum bending angles. This extreme level of conductivity and mechanical stability can be explained by the highly conductive Ag nanoparticles distributed throughout the carbon backbone structure. This structure provides multiple parallel electrical connections between particles, compensating for the change in the resistance of carbonized polyimide at high mechanical deformations.



Figure 5: Change in resistance as a function of radii of curvature for (a) carbonized polyimide and (b) carbon-silver nanocomposite with widths ranging between 0.2mm - 2mm.

WIRELESS PRESSURE SENSING APPLICATION

The described direct writing technology can be used to fabricate low-cost flexible sensors and actuators. As a proof-ofconcept demonstration, we designed and fabricated a flexible passive wireless pressure sensor that can be used for wearable and implantable applications. Figure 6 shows the fabrication process and working mechanism of the LC passive pressure sensor. The device consists of a pressure-sensitive capacitor with a variable spacing between its two electrodes that is connected in series with a planar spiral inductor. In this design, the coil provides an inductively-coupled link between the reader antenna and the LC passive pressure sensor, allowing wireless powering and information transfer. When pressure is applied to the LC resonant circuit the gap between the two electrodes of the capacitor decreases, leading to an increase in the capacitance of the circuit. This change results in a reduction in the resonant frequency of the LC circuit that can be detected wirelessly by an external readout coil. We used a network analyzer to determine the resonant frequency of the sensor at various levels of pressure by measuring the location of the phase-dip in the impedance of the external coil.

Figure 6a shows the fabrication process using the presented conductive printing technique combined with a simple folding approach to make the inductor and capacitor used in the sensor. The inductor coil and the two capacitor electrode patterns were fabricated on a flexible polyimide substrate by using the aforementioned laser carbonized and silver deposition technique.



Figure 6: (a, b) Fabrication process experimental setup of wireless pressure sensor, (c)impedance phase versus frequency for several pressures, (d) sensor resonant frequency vs. pressure.

Next, a 50 µm-thick film of PDMS (Dow Corning Sylgard® 184, 10:1 ratio) was cast onto the conductive patterns and allowed to crosslink at 60 °C for 20 min. To fabricate the cavity needed for the pressure sensitive capacitor, a 300 µm-thick film of PDMS with predefined hole-punched openings was bonded to the PDMS coating. The bonding between the two layers of PDMS was achieved by a standard PDMS-to-PDMS bonding procedure using corona treatment (BD-10A High Frequency Generator, Electro-Technic Products, Inc.) followed by annealing on a hotplate at 90 °C for 2 h. Finally, the substrate was folded in half and bonded to create the final wireless pressure sensor with the PDMS cavity between the two conductive patterns. The change in the resonant frequency was characterized by measuring the dip in the impedance phase for gauge pressures ranging from 0 to 97 kPa, Figure 5c. As can be seen, increasing the pressure results in shifting the position of the dip to lower frequencies. The resonant frequency of the sensor as a function of applied pressure is shown in Figure 5d. As expected, the resonant frequency decreases linearly with pressures up to 97 kPa, with an average sensitivity of -26 kHz/kPa.

CONCLUSION

We have developed a facile method for fabricating highly flexible and robust electrical interconnects using direct writing of a carbon-silver nanocomposite on flexible polyimide (PI) substrates. The laser irradiation selectively carbonizes the surface of the PI substrate and converts it to a super-hydrophilic surface that is used to trap aqueous solutions of precipitating silver, resulting in improved conductivity and mechanical stability/robustness. As a proof-of-concept demonstration of the use of this technology, we demonstrated the fabrication of a flexible wireless LC resonant pressure sensor consisting of a pressure sensitive capacitor connected in series with a planar spiral inductor.

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ALN PIEZOELECTRIC PARAMETRIC OSCILLATORS WITH LOW PHASE NOISE

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ABSTRACT

In this work, integrated parametric oscillators with low phase noise have been developed using geometrically optimized contour mode aluminum nitride (AlN) resonators. These resonators feature dual resonances for orthogonal contour modes at 37.2 and 74.4 MHz respectively. Their comprising parametric oscillators harness the resonantly enhanced parametric amplification in the mechanical domain to perform noise squeezing. The phase noise reduction via the parametric amplification process is analyzed with a numerical method. The measured result has shown a phase noise of -110 dBc/Hz at 1kHz offset for a 37.2 MHz parametric oscillator. Benchmarked to a conventional oscillator based on the identical resonator, the parametric oscillator has demonstrated the anticipated 25 dB reduction in phase noise at 1 kHz offset.

INTRODUCTION

Chip-scale oscillators based on piezoelectric MEMS resonators offer a potential alternative to the quartz-based counterparts as high performance frequency references. Among the various technology platforms, AlN based MEMS resonators are particularly promising due to their integration compatibility with CMOS [1], scalability towards high frequencies [2], and high quality factor (Q) in dry air [3]. Nonetheless, the phase noise performance of the state of the art AlN-based oscillators is still moderate in comparison to the commercial components based on surface acoustic wave (SAW) or quartz crystal resonators. The excessive phase noise originates from the temporal fluctuation in the resonant frequency [4], which is intrinsic to the material and structure of the device, and is challenging to diminish.

Recently, the exploitation of degenerate parametric amplification has attracted significant research interests as it promises a wide range of MEMS-based new applications, ranging from noise squeezing [5] to low power computing [6]. Oscillators and frequency dividers that leverage the parametric amplification either external [7] or intrinsic [8] to a resonator have been demonstrated with improved phase noise performance. These prior works harness the passive and extremely narrowband nature in degenerate parametric amplification to remove or filter/squeeze noise in an oscillatory system. However, an integrated piezoelectric parametric oscillator, in which the fundamental oscillation and parametric amplification are concurrently coupled in the mechanical domain of a resonant device, has never been demonstrated. It is expected that such a microsystem implementation can have higher parametric modulation depth, larger gain, and narrower bandwidth noise filtering for producing stabilized oscillations with more diminished phase noise.

In this work, we have developed parametric oscillators that integrate degenerate parametric amplification in the mechanical domain by utilizing geometrically optimized contour mode AlN resonators [9], and establish the feedback via a frequency doubler and phase shifter. In comparison to a conventional oscillator in which the amplification in the feedback loop is exclusively obtained using solid state devices, the parametric oscillator attributes a fraction of the close loop gain to parametric amplification, and thus benefits from noise squeezing and reduction inherent to a high Q passive amplification. The measured phase noise performance of the 37.2 MHz AlN parametric oscillators has shown a 25 dB improvement at 1 KHz offset over the conventional oscillators.



Figure 1: (a) Mocked up view of the 37 MHz parametric resonator. (b) SEM image of the fabricated device. The dimensions of the resonator are labeled in the figure.



Figure 2: (a) FEM simulated admittance response of both signal and parametric ports. (b) Longitudinal and transverse modes of the parametric resonator.

PARAMETRIC RESONATOR

The heart of our parametric oscillator is a geometrically optimized AlN parametric resonator. The structure and SEM image of a fabricated resonator are presented in Fig. 1. The length of the resonator is set to be 4 times the width so that the resonance of the first order transverse mode [Fig. 2(c)] is twice the resonance of the second order longitudinal mode [Fig. 2(b)]. The orthogonal modes are chosen because their resonances can be independently tuned via geometrically setting the length and width of the resonance. Thus, dual resonances at f and 2f can be readily achieved in fabricated devices. The dual resonances allow for efficient modulation of the stiffness of the parametric resonator with a pump signal at frequency 2f, and resonant coupling of the parametrically amplified vibration at f simultaneously.

The admittance response of a typical parametric resonator is modeled with COMSOL based finite element analysis (FEA). The simulated admittance response of a typical parametric resonator [Fig. 2(a)] confirms the desired dual resonances for the orthogonal modes, which are set to 37.2 and 74.4 MHz respectively. The fabricated parametric resonators were first measured with an Agilent N5230A vector network analyzer (VNA). The measured admittance of a typical parametric resonator is shown in Fig. 3(a). The device dimensions and key parameters are listed in Table I. One of the ports of the resonator is used as the signal port of the resonator, while the other one is used as the parametric port. The parametric amplification in the fabricated resonators was first confirmed by



Figure 3: (a) Measured admittance response of a typical parametric resonator utilized in the oscillator. (b) and (c) Measured Q enhancement excited by a parametric pump on multiple devices.

TABLE I.	DIMENSIONS AND KEY CHARACTERISTICS OF THE
	MEASURED PARAMETRIC RESONATORS

Design Parameter	Symbol	Device 1	Device 2	
Resonator Width (µm)	W_R	200	200	
Resonator Length (µm)	L_R	56.5	58.0	
Electrode Length (µm)	L_E	46.5	48.0	
Electrode Width (µm)	W_E	80	80	
Anchor Length (µm)	L_A	20	30	
Anchor Width (µm)	W_A	20	20	
Top Electrode Thickness (nm)	T_{Top}	80	80	
Bottom Electrode Thickness (nm)	T_{Bot}	80	80	
AlN Thin Film Thickness (nm)	T_{Aln}	500	500	
Longitudinal Resonance (MHz)	f	37.20	36.78	
Transverse Resonance (MHz)	2f	74.42	73.60	
Longitudinal Mode Quality Factor	Q_s	65	78	
Transverse Mode Quality Factor	Q_p	610	570	

* Without prior empirical data on frequency setting, resonators with the same length and slightly different widths were fabricated to ensure the experimental demonstration of devices that can resonantly support the parametric amplification.

measuring the admittance at the signal port while a 74.40 MHz signal is pumped into the parametric port. The pump signal amplifies the fundamental oscillation at 37.20 MHz by periodically modulating the stiffness constants of the AlN thin film in the longitudinal direction via Poisson's effect. As seen in Fig. 3(b, c), the amplification results in a larger admittance at the parametrically excited resonance and enhances the measured Q. Parametric amplification was only observed in devices where the dual resonances in orthogonal directions possess a two-times frequency relation.

PARAMETRIC OSCILLATOR

Using the AIN parametric resonator, a parametric oscillator has been designed as shown in Fig. 4(a). In addition to the AIN parametric resonator, the parts used in the feedback loop are all offthe-shelf components from either Mini-circuit or RF Lambda. In order to establish a self-sustainable oscillation, the 37.2MHz signal



Figure 4. Schematics of the (a) parametric and (b) conventional oscillators.

out of the signal port of the parametric resonator has to be doubled in frequency via a frequency doubler (FK-5-S) before being looped back to the parametric port of the resonator to provide amplification. Following the frequency doubler, a voltage controlled phase shifter (RVPT0117MBC) is inserted in the feedback to provide phase adjustment for satisfying the phase condition required for sustaining the parametric oscillation. The phase shift was fine-tuned during testing to achieve the best oscillator response. Several filters are used in feedback as well. A combination of a low pass (SLP-90+ 81MHz) and high pass filter (ZX75HP-65-S+) is utilized to ensure that only the 74.4 MHz pump signal is sent into the parametric port. An out-of-band rejection of 50 dB is obtained in experiment using these filters. A low pass filter (ZX75LP-40 40MHz) is used to provide 43 dB rejection to the parametric pump signal leaked to the signal port via the feedthrough capacitance of the resonator. It also ensures that only the 37.2 MHz signal is outputted from the signal port and sent to the input of the frequency doubler. Even though the parametric amplification produces a substantial gain (20 dB), it is still insufficient to overcome the conversion loss of the frequency doubler and the cascaded insertion loss of the other components in the loop. More importantly, because the conversion loss of the frequency doubler can be significantly improved by increasing the input signal power, additional solid state amplifiers ((ZKL-1R5+ and ZKL-2+)) have to be added to boost the signal strength to reach the highly nonlinear region of the frequency doubler. This, however, does not degrade phase noise performance of the parametric oscillator since the noise introduced by the solid state amplifiers will be filtered and reduced by the parametric amplification process. At last, in order to perform signal analysis at both ports of the parametric resonator in the closed-loop configuration, two directional couplers (ZX30-9-4+) are incorporated to form ports 1 and 2 of the oscillator.

To highlight the phase noise reduction in a parametric oscillator, we have also constructed a conventional oscillator with its schematics shown in Fig. 4(b) for performance comparison. The frequency doubler and high pass filters are removed from the feedback. Alternatively, the required closed loop gain is provided by the solid state amplifiers. These amplifiers operate in the highly linear region and thus cannot generate a sufficiently large second order harmonic that might accidentally start the parametric



Figure 5. (a) Amplification gain as a function of frequency deviation for various modulation depths. Maximum allowable frequency deviation as a function of modulation depth Γ with respect to (b) Q_s and (c) Q_p .

amplification. To ensure a fair comparison, the same parametric resonator is also used in the conventional oscillator. The measured phase noise of both the parametric and conventional oscillators is compared in the Results section.

NOISE SQUEEZING

The dynamics of the AlN parametric oscillator can be described by a simple one-dimensional model that represents the modulation of the stiffness in the mechanical domain [6]:

$$\ddot{x} + \frac{\omega_r}{Q_s} \dot{x} + \omega_r^2 \left(1 - \chi(n, Q_r, t, \delta) \right) x + \beta x^3$$

= $\Lambda \cos(\omega_r (1+n)t + \varphi)$ (1)

$$\chi(n, Q_p, t, \delta) = 2\gamma A(Q_p)h(n, Q_p)\cos(2\omega_r(1+n)t + \delta)$$
(2)

where x represents the longitudinal-direction displacement of the vibration. The damping in the oscillation is denoted by the second term in Eq. 1, for which ω_r is the signal resonant frequency (i.e. $2\pi f$) and Q_s is the quality factor for the resonance at *f*.

The third term in Eq. 1 captures the effect of the parametrically modulated stiffness. As formulated in Eq. 2, $\chi(n, Q_p, t, \delta)$ represents the pump modulation. γ is the modulation coefficient that measures how effectively the transverse mode displacement changes the stiffness of the AlN thin film in the longitudinal direction. $A(Q_p)$ is the amplitude of the transverse mode vibration excited by the parametric signal (2f). Q_p is the quality factor of the parametric resonance. Naturally, $A(Q_p)$ increases for a larger Q_p . $\gamma A(Q_p)$ is defined as the modulation depth, Γ . *n* is the frequency deviation off ω_r in percentage for a stabilized oscillation. Because the feedback in the parametric oscillator is comprised by a phase shifter and frequency doubler, the pump signal has a phase of δ and the same frequency deviation off $2\omega_r$ as the driving signal off ω_r . h(n, Q) is the normalized displacement amplitude (with respect to the maximum displacement) for a mechanical resonant system with a quality factor of Q. Given a pump signal of frequency $2\omega_r(1+n)$ and coupled to a resonance of quality factor Q_n , h can be expressed as [10]:

$$h(n,Q_p) = 1/\sqrt{1+n+n^2(2+n)^2Q_p}$$
(3)



Figure 6. Typical temporal response of an established parametric amplification process. ($Q_s=65, \omega_r=37.2$ MHz, $\beta=4.85\cdot10^{12}$, $\Gamma=1.435\cdot10^{-2}$, $Q_p=610$, $\Lambda=1.435$ nm).

The fourth term in Eq. 1 entails the nonlinearity in the restoring force. β is the nonlinear elastic coefficient, which has a rather small value for AlN thin films. The term on the right side of Eq. 1 is the oscillator driving/output signal at *f*. Λ and φ are the amplitude and phase of the driving signal, which has a frequency of $\omega_r(1+n)$, To set up parametric amplification, the requirement below must be satisfied [11]:

$$\gamma A(Q_p)h(n,Q_p) = \Gamma h(n,Q_p) > 1/Q_s \tag{4}$$

where Γ is required to be larger than a threshold value for overcoming the loss in the system and sustaining the parametric amplification. Because $h(n, Q_p)$ maximizes at n=0, a driving signal with a non-zero frequency deviation would impose a high threshold value of Γ . Increasing *n* will eventually result in a cut-off of the parametric amplification as seen in Fig. 5(a). Consequently, the phase noise at offsets beyond a maximum allowable frequency deviation is significantly reduced in such a system. The dependence of the maximum allowable frequency deviation on both Q_p and Q_s is shown in Fig. 5(b) and (c) for device 1 in Table I. A O_s of 65 and ω_r of 37.2 MHz are used in the simulation. It is worth noting that, in addition to the maximum allowable frequency deviation, the parametric gain [Fig. 5(a)] also influences the outcome of noise squeezing. It directly determines the magnitude of phase noise in contrast to the carrier for offsets beyond the maximum allowable frequency deviation. A higher Q_p leads to an increase in Γ and consequently a harder noise squeezing effect.

An established parametric amplification process can be numerically modeled by the steady state response of Eq. 1. As shown in Fig. 6, the input/driving oscillation is amplified by the parametric pump signal. The phase noise squeezing in the oscillator can be qualitatively explained as parametric amplification selectively enhances the signal around ω_r and thus reduces sideband phase noise. Different from prior work, this work harnesses the noise squeezing effect that collectively derives from the high Q_p resonant coupling of the pump signal and the intrinsic narrowband nature of parametric amplification.

EXPERIMENTAL RESULTS

Two parametric oscillators have been constructed using devices 1 and 2 in Table I. Their conventional counterparts as seen in Fig. 4 have also been built for performance comparison. The signals from ports 1 and 2 of the parametric oscillator based device 1 were first characterized in the time domain with an Agilent DSO54855A oscilloscope and then in frequency domain with an Agilent E445A FSA spectrum analyzer. The measured results are presented in Fig. 7. We can observe that an *f* oscillation is sustained by a 2f input signal coupled to the geometrically optimized AlN resonator. A 20 dB amplification of the *f* signal is induced by the 2f signal via parametric amplification. Additional amplification of 54 dB in the feedback loop is provided by the solid state amplifiers.



Figure 7. Parametrically excited oscillations measured (a) in time domain and (b) in frequency domain. The oscillation (port 2 signal) at f is amplified and established by pumping a 2f with an optimal phase into port 1.



Figure 8. Comparison of the measured phase noise performance between the parametric and conventional oscillators.

As discussed in the last section, the phase noise reduction will be more prominent for offsets beyond the maximum allowable frequency deviation. The maximum phase noise reduction is predicted to be more than 20 dB based on the noise squeezing model, material properties of AlN, and the experimentally Q values of the AlN parametric resonators.

To characterize the phase noise performance, the signals from ports 1 and 2 of both the parametric and conventional oscillators were measured with an Agilent E5052A signal source analyzer. The results are shown in Fig. 8. A phase noise of -110 dBc/Hz at 1kHz offset has been demonstrated for the parametric oscillators, 25 dB lower than the conventional oscillators based on the same resonators. The measurements have confirmed the noise filtering effect predicted by the model for such parametric oscillators.

CONCLUSIONS

In this work, parametric oscillators with low phase noise have

been implemented with geometrically optimized contour mode AIN resonators. The noise squeezing effect induced by parametric amplification has been theoretically analyzed and predicted to enable low phase noise performance. The implemented parametric oscillators have consequently demonstrated a 25 dB lower phase noise at 1kHz offset than that of the conventional oscillators, thus promising the use of integrated AlN parametric oscillators as low phase noise frequency references.

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SYNCHRONIZATION IN MICROMECHANICAL GYROSCOPES

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ABSTRACT

In this paper, we study the occurrence of synchronization between the two degenerate resonance modes of a micro disk resonator gyroscope. Recently, schemes involving the simultaneous actuation of the two vibration modes of the gyroscope have been implemented as a promising new method to increase their performance. However, this strategy might result in synchronization between the two modes, which would maintain frequency mode-matching but also may produce problems, such as degrading stability and sensitivity. Here, we demonstrate for the first time synchronization between the degenerate modes of a micro-gyroscope and show that synchronization dramatically reduces frequency instability at the cost of increased amplitude instability.

INTRODUCTION

Synchronization describes the tendency of weakly-coupled oscillators to track each other's frequency. This phenomenon has been observed for centuries in various fields in science, describing human's internal clock modulated by daylight periodicity [1], the behavior of grasshoppers singing in harmony [2] or fireflies' flashes emitted at once and with the same period [3]. Nowadays, synchronization is implemented in technologies, providing beat stability in dysfunctional hearts thanks to pacemakers [4] and enhancing telecommunication systems [5].

With the development of micro-technologies, synchronization has also been studied in Micro-ElectroMechanical Systems (MEMS), locking the micro-oscillator's frequency to an external generator [6] or to another MEMS [7] in order to reduce their frequency fluctuations, one of the main sources of noise in microsensors. More specifically in MEMS Gyroscopes, frequency fluctuations are intensively investigated since they directly affect the stability of the gyroscope [8]. Different methods have been suggested in order to reduce these fluctuations [9], but most of them rely on active feedback while synchronization passively fixes the resonance frequency.

This paper focuses on the implementation of synchronization in a Disk Resonator Gyroscope (DRG) and the resulting outcomes of this technique. In the first part, we will describe the device and the gyroscope's working regime. We will then explain the synchronization implementation between one mode of the gyroscope and an external oscillator. In the following part, we will present the implementation of mutual synchronization between the two modes of the gyroscope. In the last part, we will discuss the performance of the gyroscope operated with these techniques.

THE DISK RESONATOR GYROSCOPE

The micro-gyroscope is a silicon disk with a diameter of 600μ m and a thickness of 40μ m (Fig. 1), first time presented in [10], made through the *epi-seal* process [11]. The device is anchored at the center and is surrounded by electrodes. As we apply a voltage difference between the electrodes and the device, we can drive the resonant modes of the disk, tune the resonance frequency [10], control the nonlinearity [12] and detect the vibration amplitude. In our disk, the gyroscope operation is based on the actuation of the two in-plane 20 wineglass modes (described in the following as X and Y modes), the dynamics of which are described by the following equations:

$$\dot{A_X} + \Delta \omega_X \, \dot{A_X} + \omega_X^2 \, A_X + q_X \, A_Y + \frac{8 \, \omega_X}{3} \, \alpha_X \, A_X^3 \\ = F_X + 2 \, \Omega \, k \, \dot{A_Y} \quad (1) \\ \dot{A_Y} + \Delta \omega_Y \, \dot{A_Y} + \omega_Y^2 \, A_Y + q_Y \, A_X + \frac{8 \, \omega_Y}{3} \alpha_Y \, A_Y^3 \\ = F_Y - 2 \, \Omega \, k \, \dot{A_X} \quad (2)$$

where $A_{X,Y}$, $\Delta \omega_{X,Y}$, $\omega_{X,Y}$, $q_{X,Y}$, $\alpha_{X,Y}$, k, and $F_{X,Y}$ represent the amplitude, bandwidth, resonance frequency, stiffness (or quadrature) coupling coefficient, Duffing coefficient, Coriolis coupling coefficient (or angular gain), and force applied for respectively the X and Y modes. Due to both the fabrication process and the symmetry of the device, the two modes present very similar parameters, especially bandwidth $\Delta f = \Delta \omega/2\pi$ and natural resonance frequency $f_0 = \omega/2\pi$ of respectively 3 Hz and 250 kHz. The coupling coefficient q, often introduced as quadrature, describes the linear coupling of one mode on the other and can be controlled by electrostatic means [13]. The Duffing coefficient α affects the nonlinearity of the system and mainly transforms the resonance frequency into an amplitude-dependent parameter [12].



Figure 1: Schematic of the DRG and the shapes of the two modes X and Y used in the synchronization process.

When the device is operated as a gyroscope, one mode is driven as an oscillator (the drive mode, X), while $F_Y = 0$. As a rotation Ω is applied to the device, energy is transferred to the second mode (the sense mode, Y) due to Coriolis coupling through the angular gain k. This energy transfer takes place at the resonance frequency of the driven mode, and in the simplest case where nonlinearity and mode coupling are negligible, and the resonance frequencies of the two modes are near matching, the amplitude of the sense mode is described by:

$$A_{Y} = -\frac{k \Omega F_{X}}{\omega_{Y} \Delta \omega} \frac{1}{\omega_{Y} - \omega_{X} + i \frac{\Delta \omega}{2}}$$
(3)

The amplitude of the sense mode directly provides a measurement of the rate Ω applied to the gyroscope, and maximum sensitivity is obtained when the two modes' resonance frequencies match ($\omega_Y = \omega_X$), a condition known as mode-matching. Neglecting the fluctuation of the dissipation $\Delta \omega$, the performance of the gyroscope relies then on the frequency stabilization of the modes.

As the gyroscope experiences temperature changes, its stiffness is altered as well, resulting in a temperature coefficient of frequency (TCF) for the X and Y modes of respectively 14.9

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ppm/°C and 15.5 ppm/°C. Due to this asymmetry of 0.6 ppm/°C, temperature drifts lead to variations in the frequency difference between the two modes. According to Eq.(3), this variation results in a amplitude fluctuations of Y which directly affects the stability of the gyroscope's bias and scale factor. In the following, we present the effects of the synchronization of the gyroscope's modes on the frequency and amplitude stabilization with two different schemes.

SYNCHRONIZATION TO AN EXTERNAL SOURCE

When an oscillator experiences a perturbation at a frequency close enough to its resonance frequency, the oscillator will tend to match its own frequency to that of the perturbation [1], which is defined as the synchronization process. Investigating this phenomenon in our gyroscope requires a specific setup, presented in Fig. 2.



Figure 2: Setup for studying synchronization between the X mode and an external tone.

First, the resonance (here the X mode) needs to be driven as an oscillator, which is performed thanks to a Phase-Locked Loop (PLL), maintaining oscillation at the resonance frequency f_X . Note that the natural frequency $f_{0,X}$ of X does not always coincide with the resonance frequency f_X , a fact which will be at the core of the following parts.

In a second step, we send an external tone to the system at f_e , provided by a temperature-stabilized quartz oscillator, inducing an additional force F_e applied to the mode. As we sweep the frequency of the external tone within few bandwidths from the natural frequency of the X mode, we observe a region where synchronization takes place (Fig. 3), in which the mode tracks the frequency of the external tone.



Figure 3: Frequency of the X mode f_X as the frequency of the external tone f_e is swept upwards (orange) and downwards (green). f_X follows f_e within the synchronization range, which increases quadratically with the amplitude of X (inset), as predicted from the theory.

The range of this synchronization regime $\delta \omega_X$ is described by:

$$\delta\omega_X = \frac{F_e}{F_X} \sqrt{\Delta\omega^2 + 16 \,\alpha_X^2 \,A_X^4} \tag{4}$$

As long as the external tone's frequency f_e is not further than $\delta \omega_X/2$ from the natural frequency $f_{0,X}$ of the oscillator, the oscillator's frequency f_X is locked to f_e . Because of temperature variations, the natural frequency of the oscillator is fluctuating as well, such that steady synchronization can only occur for a synchronization range larger than the frequency fluctuations. Considering the DRG's TCF, $f_{0,X}$ fluctuates by 3.7 Hz/°C, which is larger than the bandwidth of our system at small amplitude.

From Eq.(4), in order to increase the synchronization range we can either increase the force of the external tone F_e or the nonlinearity of the system $\alpha_X A_X^2$. In order to have synchronization, the range of F_e is limited by F_X , restricting the synchronization range to be smaller than the bandwidth of the system. On the other hand, entering the nonlinear regime allows a larger synchronization range to be reached (inset of Fig. 3 shows ranges up to five bandwidths). Thanks to this nonlinear regime, we kept the mode X synchronized to the external tone over 4 °C of temperature change.

For a fixed frequency f_e , the synchronized oscillator's frequency f_X remains fixed and is locked to f_e even in the presence of fluctuations of the DRG's natural frequency $f_{0,X}$. In order to quantitatively observe the improvement of the frequency stability in the synchronization regime, we present in Fig. 4 the Allan deviation of the resonance frequency of the oscillator with and without the implementation of synchronization. The Allan deviation describes the precision of a parameter (here the resonance frequency) as we average it over time. Due to temperature fluctuations, the Allan deviation of the unsynchronized DRG's oscillation frequency increases for averaging times greater than 0.1s. On the other hand, in the synchronization regime, the DRG's frequency stability is determined by the stability of the external temperature-stabilized quartz oscillator. However, to improve mode matching in a gyroscope, one needs to reduce the fluctuations of the frequency difference between the drive and the sense modes, involving a more complex implementation of synchronization.



Figure 4: Allan deviation of f_X with and without synchronization. At 100s averaging time, the Allan deviation is reduced by 3 orders of magnitude in the synchronization regime.
MUTUAL SYNCHRONIZATION IN THE DRG

Starting from the previous section, one possible way to synchronize both modes of the gyroscope would be to duplicate the setup presented in Fig. 2 for both the X and Y modes. In such a scenario, each mode's frequency would be locked to the external tone's frequency, providing mode-matching at f_e . However, a less demanding implementation of synchronization can be performed, keeping the vibrating mode in the linear regime while enabling a passive frequency mode-match between the two modes that rejects temperature variations.

As discussed previously, synchronization only occurs with oscillators, thus both the X and Y modes need to be driven with separate PLLs (see Fig. 5). Then, instead of implementing an external tone to which the modes would be synchronized, we exploit the mechanical (quadrature) coupling existing between the two modes.



Figure 5. Setup for the mutual synchronization of the X and Y modes through natural mode coupling (curved arrows).

In gyroscopes, this coupling is usually cancelled out to reduce bias and bias instability, however for the purpose of this technique, we enable a small coupling to remain (typically 5% to 10% of the mode's amplitude) which we control by electrostatic means [13]. Due to this coupling, the actuation of one mode affects the other one, which induces a perturbation acting like the external tone in the previous section. As we drive both X and Y, each mode locks its frequency to the other one, entering in mutual synchronization. Thus, as one mode's frequency drifts, the other one follows, as long as the difference between their natural frequencies is sufficiently small. To first approximation, the maximum difference is determined by:

$$f_{0,X} - f_{0,Y} \leq (5)$$

$$\sqrt{\left(\frac{\Delta\omega}{2}\right)^{2} \left(\frac{q_{X} A_{Y}}{F_{X}} - \frac{q_{Y} A_{X}}{F_{Y}}\right)^{2} + 4 \left(\frac{q_{X} A_{Y}}{F_{X}} \alpha_{X} A_{X}^{2} + \frac{q_{Y} A_{X}}{F_{Y}} \alpha_{Y} A_{Y}^{2}\right)^{2}}$$

Note that Eq.(5) is equivalent to Eq.(4) if one of the mode's coupling is negligible compared to the other one, in which case the synchronization acts only in one way. On the other hand, if both modes are operated at the same amplitude with the same coupling, the linear terms in Eq. (5) cancel and synchronization occurs solely due to nonlinearity.

As discussed previously, tuning the nonlinearity enables a larger synchronization range than linear coupling. However, in this particular scheme, the fluctuations that must be handled to perform synchronization are much smaller than in the case with only one mode. Because the TCFs of the two modes are closely matched, the frequency difference between the two modes remains small even though the absolute frequency of each mode shifts by 3.7 Hz/°C. Thus, the linear coupling by itself can handle small temperature variations (according to Eq.(5), up to 2 °C for a coupling of 10%). In this configuration, the Allan deviation of the resonance frequencies' difference (see Fig. 6) shows a stability similar as in Fig. 5, demonstrating mode-matched operation at long integration time. However, as presented in Eq. (3), the impact of the synchronization phenomenon on the performance of the gyroscope can only be determined by considering the amplitude stability of the sense mode.



Figure 6. Allan deviation of the frequency difference between X and Y $(f_X - f_Y)$ with synchronization. Inset: fluctuations of the resonance frequencies of X and Y in mutual synchronization in the time domain.

DISCUSSION ON THE AMPLITUDE STABILITY

By using the linear coupling between the two modes, the gyroscope is operated in a perfect frequency mode-matched regime, the frequency fluctuations being cancelled through the synchronization process. However, these frequency fluctuations are converted into amplitude fluctuations (Fig. 7), the magnitude of which is directly related to the synchronization parameters.

In the linear limit of synchronization between X and Y, the coupling needed to compensate frequency fluctuations will induce amplitude fluctuations in the same proportion. If 10% coupling enables synchronization, amplitude fluctuations of up to 10% may be induced. In Fig.7, we observe frequency fluctuations of 0.4 ppm between the X and Y modes without synchronization, which correspond to 0.1 Hz. Considering our bandwidth of 3 Hz, we need a linear coupling of at least 3.3% of the mode's amplitude, which should induce amplitude fluctuations of the same amount, very close to our measurement of 3%. Because of these induced amplitude fluctuations, the gyroscope performance decreases in the synchronization regime with the current configuration. This result is generic to any gyroscope wherein 1) the modes are operated in a frequency mode-matched regime, 2) the modes are coupled (which is induced at least by the Coriolis force), 3) the modes are driven simultaneously as separate oscillators.



Figure 7. Phase spaces resulting from the two synchronization setups presented (left: X synchronized with an external tone in the nonlinear regime, right: synchronization between X and Y in the linear limit, both graphs using the same scales) and compared with the non-synchronized scenario. These plots reveal that the frequency fluctuations vanish in the synchronization regime at the cost of greater amplitude noise.

In parallel, recent work has shown promising results by driving both modes of a gyroscope simultaneously [14,15]. The results presented here suggest that in such a configuration, care should be taken to avoid accidental synchronization, which could decrease gyroscope performance. To prevent synchronization, the mode-matched regime can be avoided, a phase difference may be fixed between the two modes [14] (which are no longer separate oscillators), only one PLL may be used for the two modes [15] (which are no longer driven as oscillators), or by any means which violates one of the three statements of the previous paragraph.

CONCLUSION

We demonstrated that the synchronization of a mechanical oscillator enables stabilization of its resonance frequency in the presence of temperature variations. By driving both modes of a DRG, we used the coupling between the modes to perform mutual synchronization, wherein each mode tracks the other one's frequency. This configuration enables a perfect passive frequency mode-match between the two modes, robust over temperature variation. However, we demonstrated that this technique converts the frequency fluctuations into amplitude fluctuations, which decreases the gyroscope performance.

This work highlights the importance of avoiding synchronization in gyro control schemes where both modes are simultaneously excited as the synchronization may induce amplitude instability.

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VERTICAL ACOUSTIC CONFINEMENT FOR HIGH-Q FULLY-DIFFERENTIAL CMOS-RBTS

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ABSTRACT

This work demonstrates the use of phononic crystals (PnCs) to create solid-state phononic waveguides in standard CMOS technology. Such waveguides confine vibrational energy in CMOS frontend-of-line (FEOL) layers. Fully-differential driving ensures wave guiding and lowers scattering. Unreleased CMOS resonant body transistors (RBTs) based on these waveguides were implemented in IBM 32nm SOI technology. Quality factors exceeding 13,000 are achieved at 3.185GHz for an $f_0 \cdot Q$ of 4.2×10^{13} , marking the highest $f_0 \cdot Q$ RBT and highest $f_0 \cdot Q$ unreleased MEMS resonator to date.

INTRODUCTION

High-*Q* resonators are crucial components for filters and low phase noise oscillators, which are essential for RF, analog and digital circuits. With the ever expanding demands for higher communication data rates and fast data processing, it is desirable for resonators to scale to GHz-frequencies and beyond. Wearable electronics and medical implants are examples of critical battery-operated systems that necessitate minimization of power consumption, size and cost. Micro-Electro-Mechanical (MEM) resonators are an attractive solution for today's demands, with $Q > 10^4$ at GHz-frequencies and footprint on the order of $50\mu m^2$. Their monolithic integration with CMOS has been the subject of many research studies, with MEMS-first, MEMS-last and CMOS-MEMS being the most notable approaches [1].

The authors have previously demonstrated a new class of *unreleased* CMOS MEMS resonators, the Resonant Body Transistor (RBT), fabricated in the same process and side-by-side with standard CMOS transistors, without the need for any post-processing or packaging [2]. These RBTs rely on *phononic crystals* constructed in the CMOS back-end-of-line (BEOL) layers to achieve elastic energy confinement. Acoustic resonance is detected by active FET sensing, where the stress in the channel modulates the mobility of the sensing transistor [6]. MOS capacitors (MOSCAPs) are used for electrostatic actuation. It is also important to notice that scaling to GHz-frequencies has the advantage of making Landau-Rumer regime the dominant phonon relaxation process. This allows $f_0 \cdot Q$ to scale linearly with f_o , enabling higher Q devices.

In contrast, this work demonstrates *phononic waveguides* in standard CMOS technology, that confine the elastic vibrational energy vertically to the CMOS front-end-of-line (FEOL) layers. Confinement is achieved by reflection from the top by using a BEOL PnC as in [2], and from the bulk wafer by virtue of the faster sound velocity therein. This is a phenomenon similar to index guiding in photonic waveguides [3]. For CMOS RBTs, fully-differential driving is proven to be essential to ensure wave guiding and to minimize scattering. This wave guiding allows for > $50 \times$ increase in quality factor of unreleased CMOS MEMS resonators. RBTs based on these phononic waveguides with fully-differential drive and sense are implemented in IBM 32nm SOI technology as shown in Fig. 1.

DEVICE DESIGN

BEOL Phononic Crystal

PnCs are periodic structures composed of materials with contrasting acoustic impedance, which are characterized by the



Figure 1: SEM micrograph showing (a) the full RBT structure (b) sensing FETs with isolation gates and (c) the termination waveguide section.

presence of *bandgaps* in their dispersion relation. Such bandgaps prohibit wave propagation in at least one direction, within the corresponding frequency range. Table 1 shows the mechanical properties of common CMOS BEOL materials. Large acoustic impedance contrast can be observed, especially between copper and the low-k SiCOH dielectric. This enables the realization of BEOL PnCs with large bandgaps. The latter are highly desirable to minimize the effect of CMOS process variations and to ensure large reflections with small number of PnC layers [2].

	Table 1:	Common	CMOS	BEOL	material	propertie
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	ρ	c_{11}	Z_{II}	C44	Z_{44}
Material	(kg/m^3)	(GPa)	(MRayl)	(GPa)	(MRayl)
SiO2	2200	75.2	12.9	29.9	8.1
Tungsten	17600	525.5	96.2	160.5	53.1
SiCOH	1060	3.96	2.05	1.32	1.18
Copper	8700	176.5	39.1	40.7	18.8

Fig. 2 shows an example of a BEOL PnC implemented in IBM 32nm SOI technology using copper metal line in a background of SiCOH dielectric. This particular structure, with 154nm metal line width spaced by 66nm, demonstrates a 3GHz (71%) complete bandgap. COMSOL FEM simulation with periodic boundary conditions was used to obtain the PnC dispersion relation. This PnC can be used as a strong reflector to provide good energy confinement in unreleased CMOS RBTs as demonstrated in [2]. While it provides high reflection from the top of the RBT cavity, it cannot prevent acoustic wave radiation into the CMOS die substrate [3]. A phononic waveguide must instead be implemented to achieve full vertical energy confinement.



Figure 2: (a) Phononic Crystal (PnC) 2D unit cell using BEOL metals with periodic boundary conditions in x and y. Cu metal width is 154nm spaced by 66nm. (b) Reciprocal k-space and Irreducible Brillouin Zone (IBZ). (c) Dispersion relation showing 3GHz (71%) bandgap.

Phononic Waveguides

Considering the BEOL PnC of the previous section, on top of the FEOL layers and bulk wafer, it is possible to construct a phononic *waveguide* that can fully confine elastic waves to the CMOS FEOL layers. In particular, consider the structure of Fig. 3(a), where FEOL transistor gates are included at double the PnC horizontal period, together with the active area tungsten contacts. The resulting structure is periodic in the x-direction. The x-component k_x of the wave vector \vec{k} is conserved throughout the structure by virtue of the translational symmetry [3].

COMSOL FEM simulation of one horizontal period was used to obtain the dispersion relation of the phononic waveguide, with Floquet periodic boundary conditions (PBC) in x, parameterized by k_x . The resulting dispersion relation is shown in Fig. 3(b). Five types of modes can be observed in this dispersion relation. (i) PnC modes: these are modes corresponding to the PnC bands (band 1 and 2) that can propagate upwards in the PnC. (ii) Modes inside the PnC bandgap: these are evanescent waves that cannot propagate upwards in the PnC. These are not shown in the dispersion relation of Fig. 2(c) as they are not supported by infinitely periodic PnC structures. (iii) Bulk propagating modes: these are plane waves in the bulk wafer with dispersion relation $\omega = c |\vec{k}|$, or $\omega > c k_x$ for a given k_x , with c the corresponding sound velocity. These modes form the continuum referred to as the sound cone in Fig. 3(b), which is bounded by the sound line $\omega = ck_x$. (iv) Evanescent modes in the bulk wafer: these are modes below the sound line with $k_x > \omega/c$, yielding an imaginary k_y that is characteristic of evanescent modes. (v) Localized or guided modes: these modes are both inside the PnC bandgap and below the sound line. They are evanescent waves both in the PnC and the bulk wafer and can not propagate in either. These modes are vertically confined to the FEOL layers, creating a horizontal waveguide in the x-direction. Due to the energy confinement, the resulting spectrum is discrete as in Fig. 3(b). Such modes are ideal candidates for unreleased CMOS RBTs as they provide the necessary energy confinement to achieve a high-Q resonator, as well as sufficient frequency separation for spurious mode reduction.

It is also important to note that the reflection achieved from the bulk wafer is mainly due to the higher sound velocity in the bulk silicon. This phenomenon is similar to index guiding in photonics (or total internal reflection from Snell's law). The SOI buried oxide



Figure 3: (a)1D CMOS waveguide unit cell (one horizontal period) with the PnC of Fig. 2 and FEOL MOSCAP transducers (b) Dispersion relation for the horizontal waveguide showing vertically confined RBT modes.

(BOX) layer does not play any role in the energy confinement and this wave guiding is applicable in bulk CMOS technologies.

Fully-Differential Driving

While the proposed phononic waveguides offer a solution for elastic wave confinement in RBTs, wave guiding is restricted to a certain range of k_x values. Fig. 3(b) indicates that for a given frequency ω , wave guiding only happens for $k_x \in \left(\frac{\omega}{c}, \frac{\pi}{a}\right]$. RBTs must operate in this particular k_x range.

Furthermore, one must consider the effect of perturbations in the design of the waveguide. Random perturbations arise from stochastic process variations, which are prominent in modern CMOS. Systematic perturbations arise when the waveguide characteristics are changed either from systematic process variations (e.g. overetching) or by design (e.g. to implement a termination). Perturbations to the waveguide structure will potentially result in scattering, which is the coupling of the main waveguide mode to other undesired modes. Scattering has adverse effects on the resonator: (i) in the case of scattering to a radiation mode, the scattering process represents energy losses that reduce the quality factor of the resonator; whereas (ii) scattering to guided modes can potentially lead to spurious modes appearing at different frequencies. As highlighted in [7], to a first order, scattering amplitude is inversely proportional to the difference in k_x between the modes under consideration. Scattering at a given frequency corresponds to modes on a horizontal line in Fig. 3(b). For the guided modes, the sound line represents the nearest mode in \vec{k} -space.

To reduce the effect of scattering, it is important to operate as far as possible from the sound line, or at $k_x = \pi/a$. This also has the advantage of yielding the fastest evanescent decay for elastic waves in the bulk wafer, as the imaginary *y*-component of \vec{k} is given by

$$k_{y} = i \sqrt{\left(\frac{\pi}{a}\right)^{2} - \left(\frac{\omega}{c}\right)^{2}}.$$
 (1)

Faster evanescent decay corresponds to higher stress at the driving and sensing transistor gates.



Figure 4: Spatial distribution of actuation stress for (a) in-phase and (b) fully-differential driving, along with (c) a comparison between the k-space components of fully-differential driving for finite driving transducer with 2,4 and 10 periods.

Specific k_x values can be enforced by the spatial distribution of the RBT drive transducer. Operating at $k_x = \pi/a$ simply corresponds to neighboring periods of the waveguide being 180° out-ofphase. This can be formally proven by considering the Fourier series expansion of the driving stress waveform in \vec{k} -space. Consider the in-phase driving of Fig. 4(a). The transducer has a period $\Lambda = a$, where the stress T_o is constant over the length *b*. Fourier series expansion for the stress is given by

$$T(x) = \sum_{n} \frac{\overline{T}_{ob}}{a} \operatorname{sinc}\left(\frac{n\pi b}{a}\right) \exp\left(i\frac{2\pi n}{a}x\right).$$
(2)

The components fall entirely in the sound cone at $k_x = 2\pi n/a$, $\forall n \in \mathbb{N}$. This corresponds to plane wave radiation normal to the FEOL layers into the bulk wafer. On the other hand, the fully-differential driving of Fig. 4(b) has a period $\Lambda = 2a$ and a Fourier series expansion given by

$$T(x) = -\sum_{n} i^{3n+1} T_{\circ} \operatorname{sinc}\left(\frac{n\pi}{2}\right) \operatorname{sin}\left(\frac{n\pi b}{2a}\right) \exp\left(i\frac{\pi n}{a}x\right).$$
 (3)
Fully-differential driving shows components at $k_x = n\pi/a, \forall n \in \mathbb{N}$, which are non-trivial only for odd *n*. This can be intuitively explained by considering the radiation field in the bulk wafer far away from the driving transducers. At a distance $l \gg a$ in the bulk, out-of-phase radiated fields from neighboring transducers destructively interfere together, resulting in a negligible radiation field.

The above analysis is for ideal infinitely periodic transducers. However, real resonators incorporate finite transducers. Fourier transform should be used instead of Fourier series and a continuous spectrum is obtained in \vec{k} -space. The larger the number of the transducer periods the narrower the spectrum in \vec{k} -space will be, which helps in lowering scattering. This is highlighted in Fig. 4(c), where the spectrum of finite transducers is compared for different number of fully-differential periods ($\Lambda = 2a$). Scattering reduction favors a large number of driving periods, however, this comes at the cost of resonator footprint, input capacitance, as well as increased random process variations. This represents a design trade-off.

Full RBT Structure

An RBT based on phononic waveguides is implemented in IBM 32nm SOI technology. The full RBT structure is shown in Fig. 5. Two MOSFETs are used for fully-differential active sensing in the middle of the RBT structure. Eight MOSCAPs (driven 180° out-of-phase) are used for fully-differential electrostatic internal dielectric drive on each side. This enforces the condition $k_x = \pi/a$ for



Figure 5: Cross section of the vertically confined RBT design (half structure) with differential capacitive actuation and transistor sensing.



Figure 6: Simulated stresses at sensing FET in the resonator assuming bulk material properties.



Figure 7: Simulated y-stress of the waveguided RBT mode at 3.155GHz resonance.

reduced scattering with sufficient mode uniformity, while maintaining relatively small resonator footprint. Phononic waveguides with mismatched characteristics have been used at both ends to terminate the cavity. Shallow-trench isolation (STI) is used to separate the driving MOSCAPs from the sensing MOSFETs for better electrical isolation. As seen in Fig. 5, the STI isolation in this technology is sufficiently shallow, causing small perturbation in the waveguide structure. However, numerical optimization for the full RBT structure is required to match the different waveguide sections and avoid scattering [8]. The SOI BOX layer further contributes to the electrical isolation.

FEM frequency domain simulation is used to assess the performance of the fully-optimized RBT. The average stresses along the channel length of the sense MOSFET are shown in Fig. 6. The ystress for the resonance mode at 3.155GHz is shown in Fig. 7. The mode is confined to the FEOL layers, with evanescent decay in the bulk wafer, BEOL PnC, as well as the termination.

EXPERIMENTAL RESULTS

Waveguided RBTs in this work were fabricated in IBM 32nm SOI technology. SEM micrographs of the RBT are shown in Fig. 1, highlighting the phononic crystal, sensing FETs, as well as the termination waveguides. Fabricated resonators occupy an area of $4\mu m \times 13\mu m$.

Fig. 8 shows an optical micrograph of the RBT probing pads, along with the DC biasing connection. Fully-differential RF GSSG



Figure 8: (a) Optical micrograph of the resonator showing probing pads and (b) zoomed-in top-view of the RBT.

probes were used for driving and sensing ports of the RBT. The driving MOSCAPs was biased in strong inversion with $V_p = 1V$. The sensing FETs were biased in linear regime with $V_{gs} =$ 0.7V and $V_{ds} = 85$ mV to maximize the sensitivity of the drain current to channel mobility modulation. Drain current for a single sensing FET was $115\mu A$. RF testing power was limited to -20dBm, which corresponds to a peak voltage of 60mV on the MOSCAPs, in order not to perturb the DC operating point.

An Agilent N5225A PNA was used for RF characterization, which was performed at room temperature in air. The front-panel couplers on the PNA were reversed for a 15dB enhancement in sensitivity. As a result, the PNA transmitted power was reduced by the same factor. To compensate for this loss, the testing power was increased to -5dBm. Full 4-port S-parameters measurements were performed for the RBTs. On-chip open structures were used for deembedding. An electromechanical transconductance g_{em} was extracted from the resulting Y-parameters as $g_{em} = Y_{2l}-Y_{12}$, according to the standard transistor π -model. An IF bandwidth of 3kHz, along with 30-point average were used. A dwell time of 20µs was selected to allow for resonance build-up. Additionally, a smoothing filter with a 9-point aperture was used to reduce the peak-to-peak noise.

The measured fully-differential g_{em} of one RBT is shown in Fig. 9, demonstrating resonance at 3.1885GHz with a FWHM quality factor $Q\sim13,300$ for an $f_o \cdot Q$ of 4.2×10^{13} . This marks the highest $f_o \cdot Q$ RBT and highest $f_o \cdot Q$ unreleased MEMS resonator to date.

CONCLUSION

Phononic crystals in CMOS BEOL layers were used to realize unreleased phononic waveguides that confine elastic vibrations in the CMOS FEOL layers. Reflections from the bulk substrate are achieved by a phenomenon similar to index-guiding in photonics. Such phononic waveguides can be used as the basis for a resonance cavity of unreleased CMOS RBTs, mechanical coupling thereof, or phononic networks for advanced signal processing. Fully-differential driving is essential for wave guiding and scattering reduction.



Figure 9: Measured electro-mechanical transconductance (g_{em}) of the RBT, with record breaking $f_o Q$, in close agreement with simulation.

RBTs implemented in IBM 32nm SOI achieve $Q\sim13,300$ at 3.19GHz, for an $f_o Q$ of 4.2×10^{13} , marking 56× increase over previous design. The solid-state nature of these devices and small feature sizes available in advanced CMOS nodes enable high-Q GHz-frequency operation, with relaxed packaging and operating voltage constraints.

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FUTURE OPPORTUNITIES ARISING FROM IMPLICATIONS OF MOORE'S LAW

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The semiconductor device world has been fueled for the past 60 or more years by Moore's Law and its implications on semiconductor device technologies, by driving industry to develop eversmaller gate sizes on an annual basis, accompanied by corresponding increased performance of the associated electronics. On one hand, it has been obvious for many years that a limit to Moore's law is inevitable, when the gate size reaches the single molecule length scale. At present we are reaching the 10-nm length scale, so that discussions of the end of Moore's law are becoming more widespread, active, and serious in the nano electronics sector and at international conferences involving nanoscale materials and applications.

Concurrently, electronic devices continue to increase in functionality and energy efficiency. New start-up companies continue to be launched and young people at universities continue to be attracted to this research field and to commit much of their effort into coupling advances in hardware with computer science into start-up companies in the electronics industry. Creativity and inventiveness remain at center stage.

Silicon, along with carbon, is in Column IV of the period table, and is an Earth-abundant material that has made a big mark on modern electronics. Even though new materials are finding niche applications in modern device technology, silicon is expected to remain the material of choice to support much of industrial semiconductor device technology for the foreseeable future, because of its special electronic properties, and because of the huge effort and investment that industry has already made to achieve silicon's present key role in present-day electronics.

However, the research community has had to put significant effort into planning for the future and particularly to what happens when Moore's law comes to an end. This has become an increasingly important issue for public discussion at conferences and in the research literature for the past decade. Scientific progress has in recent years been made faster and faster and planning for the future has been more organized. Combining this societal aspect with activities occurring in organizations and sponsored by governments, studies of research opportunities in the next decade have become more common and have had more impact.

Historically, what has happened in the past when a particular technology stops meeting societal expectations for advance is that competing emergent technologies get a chance to blossom and major advances in technology occur and the standard of living rises as a result. This is what happened with the adoption of electricity as a general energy source just before we entered the 1900s, soon followed by the adoption of AC rather than DC power, and after several decades the appearance of computers and the start of the digital world.

Several new research directions have already emerged as paving the way for reaching center stage when the era of Moore's law, as we know it, ends. Two clear developments that have clearly taken hold is the development of new research directions for electronics research, along with the discovery of new science associated with the new materials. Firstly, in the United States, the funding agencies have been very helpful in organizing studies with wide participation motivated by the research community through the decadal studies by the National Academies of Science and Engineering, which were effectively extended by more detailed studies organized by the Department of Energy's Office of Science and other governmental agencies and implemented by providing group grants to promote collaborative interdisciplinary studies. In Europe, there were regional and topical programs, such as the flagship program on graphene and nanoscience which was soon extended to other popular layered materials with bandgaps. Individual counties also were motivated to start local programs around a theme and these countries started to award group grants promoting collaborative teams to solve larger scale scientific problems using teamwork approaches. The approaches in time became more popular following international trends.

Now let me address some specifics about my own research field in quite general terms. In the field of materials physics, threats of bringing Moore's law to smaller and smaller length scales has resulted in a revolution supporting the research on new materials that had not been much studied before at the nanoscale and the study of the new science brought by the studies of these materials.

The new materials under study nowadays fall into different categories, which can be classified in different ways, such as their dimensionally, their special properties, their method of preparation, their potential practical use, their potential scientific use, and their potential in serving as a standard reference material. Let us now consider some of these categories further.

Under the category of dimensionality, in the past my group considered bulk or three-dimensional (3D) materials almost exclusively, since these were the only materials that we could then make in the laboratory. We, of course, have considered lower-dimensionality materials abstractly in our imagination since the beginning of my independent career in1960, and we studied low-dimensional materials to some extent in actuality. I for one was attracted to study carbon-based materials because they were abundant on our planet and they had properties different than silicon, which was a highly popular material. I recognized in my early career that silicon along with carbon are both in Column IV of the period table, and are Earthabundant materials. Silicon, however, had made a big mark on modern electronics and carbon had not in my early career. Even though new materials are finding niche applications in modern device technology, silicon is expected to remain the material of choice to support much of industrial semiconductor device technology for the foreseeable future, because of its special electronic properties, and because of the huge effort and investment that industry has already made to achieve its present key role in present-day electronics.

In 1992 my group wrote a paper saying that it would be interesting to study carbon nanotubes because they were a new type of one-dimensional-like material. Without changing its chemical composition, carbon could be either semiconducting or metallic in the form of single-wall nanotubes, depending on their chirality. Interestingly, it took six years more before we learned how to prepare carbon nanotubes well enough to verify that this concept was indeed correct, but once the preparation method was demonstrated in a simple way in our 1998 paper, it soon became easy enough to demonstrate by high school students in their own classrooms.

Another characteristic of nanoscale materials like carbon nanotubes is that they can be doped with certain chemical species to give them other interesting properties, such as various types of magnetism or superconductivity which would be different from the properties of their bulk counterparts relative to critical field, and critical temperature.

978-1-940470-02-3/HH2016/\$25©2016TRF DOI 10.31438/trf.hh2016.26 Yet another type of different property of the low-dimensional form of carbon relative to its bulk counterpart would be in the formation of nanotube ensembles whereby the nanotubes could be aligned, showing different properties from the individual tubes, with different properties found if all the tubes in the ensemble were of the same chirality, or were all metallic or all semiconducting. Different properties could also be achieved by ensembles of ordered vs. disordered nanotubes.

Almost every category of property mentioned above has had some scientific interest from a metrological viewpoint, and the metrology of low-dimensional systems is a topic of current interest that has entered the intellectual property world with products associated with some of them. The simple (5,4) semiconducting nanotube may become a simple standard because people can easily make it and use it for semiconducting applications. Nanotubes are 1D systems that are likely to depend on their preparation method, and materials synthesis and preparation are other topics worthy of discussion relative to what may happen in the era of beyond Moore's law. In recent years the nanoscience research community increased rapidly. Carbon nanotubes became popular in the early 1990s, but the associated research community and the resulting commercial world kept growing, especially after the discovery of graphene in 2004, which fueled another large expansion ready to exploit the new layered materials now under development. These new materials include nanoscale forms of transition metal dichalcogenides of many kind with a large variety of properties, which are getting ready for exploitation by the next wave of young people looking for new opportunities in the nanoworld.

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UNPRECEDENTED FIGURE OF MERIT IN EXCESS OF 108 IN 920 MHz ALUMINUM NITRIDE CROSS-SECTIONAL LAMÉ MODE RESONATORS SHOWING k² IN EXCESS OF 6.2%

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ABSTRACT

In this work we demonstrate a 900 MHz cross-sectional Lamémode resonator showing a k_t^2 in excess of 6.2% and a loaded quality factor in excess of 1750. Such a high k_t^2 , which closely matches that Finite-Element-Methods (FEM) predicted value, is attained through the use of a 3-finger CLMR and a thickness-fieldexcitation (TFE) scheme. This device not only shows a high k_t^2 comparable to the one attained by film-bulk-acoustic resonators, FBARs, but is also characterized by an unprecedented figure-ofmerit (FoM \sim 108) that, to the best of the authors' knowledge, is the highest demonstrated in AlN resonators operating at ~1 GHz.

INTRODUCTION

Micro Electro Mechanical (MEM) resonators are key enablers for the development of miniaturized and low-power multi-band radiofrequency (RF) systems capable of operating in the extremely crowded modern commercial and military spectral environment.

For years, MEM resonators have been researched for their ability to attain high quality factors (Q) and large electromechanical coupling coefficients (k_t^2) in small volumes. Recently, the complete maturation of the Aluminum Nitride (AlN) Film-Bulk-Acoustic resonator (FBAR) technology [1] has allowed the replacement of off-chip surface acoustic wave (SAW) devices in commercial products, hence enabling better performance in a miniaturized form-factor.

The AlN FBAR technology relies mainly on the e33 piezoelectric coefficient of AlN to transduce resonant vibration along the thickness of an AlN plate. As the device resonance frequency (f_r) is set by the thickness of the AlN plate (T_{AIN}), it cannot be tuned lithographically. As a consequence, the integration of multi-frequency FBAR-based filters on the same chip can only be attained through an increase of the fabrication complexity (i.e. by mass-loading or trimming). Such a limitation has been overcome by the AlN contour-mode resonator (CMR) technology [2]. In fact, AlN CMRs rely mainly on the e_{31} piezoelectric coefficient of AlN to transduce resonant vibration along an inplane direction of an AlN plate (e.g., width extensional or length extensional motion). Therefore, the lithographically set lateral dimension of the device determines its resonance frequency, enabling the fabrication of CMRs operating in the Ultra-High-(UHF), Very-High- (VHF) and Super-High- (SHF) frequency ranges on the same chip [3],[4]. Although multi-frequency AlN CMRs can be readily integrated on the same chip, their k_t^2 is lower than that of FBARs, due to the intrinsically lower amplitude of the e_{31} compared to the e_{33} . For this reason FBAR-based filters have been preferred to the CMR-based ones for the implementation of low insertion loss and wideband passive filtering networks.

More recently, AlN cross-sectional Lamé-mode resonators were demonstrated (CLMRs) [5],[6]. CLMRs are piezoelectric resonators formed by two metallic IDTs sandwiching an AlN plate. They rely on the combined use of both the e_{31} and the e_{33} piezoelectric coefficients of AlN to transduce a Lamé-mode in the cross-section of an AlN plate through. Thanks to this special feature, CLMRs can simultaneously achieve high- k_r^2 and a lithographic definition of their resonance frequency (f_{res}), enabling

the implementation of lithographically defined integrated contiguous and not-contiguous pre-select filters for platforms adopting carrier-aggregation (CA).

In this work we demonstrate 1-port CLMRs simultaneously showing a record-high k_t^2 in excess of 6.2%, a quality factor (*Q*) in excess of 1750 and a Figure of Merit (*i.e.* FoM= $Q^*k_t^2$) in excess of 108. To the best of the authors' knowledge, such a high FoM-value is the highest ever demonstrated in AlN resonators operating in the same frequency range.

CROSS-SECTIONAL LAMÉ MODE RESONATORS

AlN CLMRs are piezoelectric resonators capable of transducing a Lamé-mode in the cross-section of AlN plates through the coherent combination of the e_{31} and e_{33} piezoelectric coefficients of AlN (Fig. 1). Thanks to the opposite sign of these two piezoelectric coefficients, in-phase charge components are generated by vibration along both the cross-sectional directions (thickness and width) of the plate. As a consequence, the k_1^2 attained by AlN CLMRs is function of both the e_{31} and e_{33} piezoelectric coefficients of AlN, and it is higher than the k_1^2 achieved by conventional laterally vibrating AlN resonators such as CMRs.



Figure 1: schematic-view of a 3-finger CLMR. The device is formed by two IDTs sandwiching an AlN film. The pitch of the IDTs (W) is selected to be similar to the thickness of the AlN layer (T_{AIN}). Such choice enables the excitation of high- k_t^2 degenerate or nondegenerate Lamé-modes in plates. The mode-shape relative to the total displacement of the same device, when exciting a nondegenerate Lamé mode, is also reported.

Similarly to CMRs, CLMRs can be excited through either a Lateral-Field-Excitation (LFE) [7] or a thickness-field-excitation (TFE) approach [6]. LFE CLMRs are formed by one set of IDT patterned on either the top or the bottom surface of an AlN layer. In contrast, TFE CLMRs are formed by two interdigital-metal electrodes sandwiching an AlN film. The IDTs, in both TFE and LFE CLMRs, are needed to produce the excitation of the electric field in the cross-section of the AlN layer.

As demonstrated in [5], CLMRs achieve maximum k_t^2 -value when the pitch of the IDTs (W) is set to a specific value (W^{opt}) similar to thickness (T_{AIN}) of the AlN plate. In fact, in this scenario, a nondegenerate Lamé-mode is excited in the cross-section of the AlN plate. However, due to the capability of exciting

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high- k_t^2 degenerate cross-sectional Lamé modes [8] in plates, CLMRs can attain high k_t^2 also when W is slightly different from W^{opt}. In addition, since f_{res} depends on W, the transduction of such degenerate modes also enables a significant lithographic tunability of the device operating frequency. This feature is crucial for the implementation of multi-frequency resonators and filters monolithically integrated on the same chip with minimal fabrication complexity. In this work, a non-degenerate CLMR (adopting the optimum IDT geometry, *i.e.* W=W^{opt}) showing a record k_t^2 in excess of 6.2% is experimentally demonstrated for the first time.

Fabrication Process

The CLMRs presented in this work are formed by a 4 μ m thick AlN layer and two 0.1 μ m thick platinum IDTs placed on the top and bottom surfaces of the AlN film. The choice of using platinum for the bottom IDT was dictated by the need of growing a high quality AlN film. Platinum was also used for the top IDT in order to preserve high acoustic symmetry in the device cross-section.



Figure 2: Microfabrication process of TFE CLMRs: (a) Pt film was deposited on top of Silicon, through a 10 nm thick Ti layer used as adhesion layer, and patterned through lift-off process; (b) AlN film was deposited on top of the Pt film and vias in the AlN were formed; (c) Pt film was deposited on top of Si and patterned through lift-off process; (d) AlN film was etched through the use of a SiO₂ hard mask that was preferred to traditional photoresist mask to attain steeper AlN sidewalls; (e) Si substrate was released by XeF₂ isotropic etching.

The devices were fabricated using a four-mask microfabrication process (Figure 2): 10/100 nm of Ti/Pt was deposited and patterned on top of a high resistivity silicon wafer to form the bottom IDT. Next, a 4 μ m thick AlN film was sputter-deposited. Then, we etched AlN through wet etching to form the vias. Next, 10/100 nm of Ti/Pt was deposited and patterned to form the top IDT. Then, the AlN film was etched by ICP in Cl₂ based chemistry to define the width of the AlN plate. This was done through the use of a hard mask made out of 2 μ m of SiO₂ so as to attain steep AlN sidewall angles (>75°). Finally, the Silicon substrate underneath the resonator was released through XeF₂ isotropic etching.

High-FoM exceeding 108 in AlN CLMRs

3-fingers 920 MHz TFE CLMRs were fabricated. These devices are formed by two 100-nm thick platinum IDTs sandwiching a 4- μ m thick AlN-plate. The pitch of their IDTs was optimized (*i.e.* W was set to 5 μ m), through simulation, in order to maximize the

simulated k_t^2 value. A Scanned Electron Microscope (SEM) picture of one of the fabricated CLMRs is shown in Figure 3.

Two quarter-wave acoustic transformers were designed at the edges of the device [9] in order to minimize the loss of acoustic energy through the anchors (*i.e.* known as anchor-losses) [10],[11]. As verified through 3D-FEA (Figure 4), this technique, which was originally developed for AlN CMRs, is also effective in minimizing the displacement in the inactive-regions of CLMRs, thus enabling a reduction of anchor-losses and, consequently, a larger mechanical quality factor (Q_m). Both the 3D-FEA simulated and measured admittance (Y) curves relative to the best fabricated CLMRs (Figure 3) are reported in Figure 5.

As evident, a k_i^2 value in excess of 6.2% was extracted through MBVD-fitting [12] of the measured response. Such value matches closely the predicted value (~6.6%) found through FEA, thus confirming its validity. In addition, the measured response showed a loaded quality-factor, Q_{load} , (extracted from the measured device 3dB- bandwidth), in excess of 1750. Such high Q_{load} and k_i^2 values allowed to achieve a measured FoM in excess of 108. To the best of the authors' knowledge, such a large FoM is the highest ever demonstrated in AlN resonators operating in the same frequency range.



Figure 3: Scanned-Electron-Microscope picture of a fabricated CLMR.



Figure 4: left) Simulated modal-distribution of the device displacement, through 3D-FEA. As evident, the use of the acoustic quarter-wave transformers enables a significant reduction of the magnitude of the total-displacement reaching the resonator inactive-regions; right) 3D-FEA simulated cross-sectional modal distribution along a cut-plane (AA') at the resonator center. As evident this modal-distribution matches closely what found with the 2D-FEA analysis reported in Figure 1.

In addition, several spurious-modes, which could not be found through 2D-FEA (Figure 5), were instead detected in both the measured and the 3D-simulated admittance responses (Y). Although their origin is still under investigation, these spurious modes are likely due to the transduction of low-coupling transverse modes propagating along the resonator length, hence not being detectable through 2D-FEA. Strategies to suppress such undesired modes of vibration remain under investigations.

CONCLUSION

This paper reports on crucial advances in the development of monolithic integrated RF-passive components based on the recently developed Aluminum Nitride (AlN) cross-sectional Lamémode (CLM) piezoelectric technology. In particular, we demonstrated a cross-sectional Lamé-mode resonator (CLMR) operating around 920 MHz and showing k_t^2 and Figure-Of-Merit (FoM) in excess of 6.2% and 108, respectively.



Figure 5: red) Measured admittance response of the best fabricated CLMR. The device was tested, in air, by probing it through Ground-Signal-Ground (GSG) probes. The device response was de-embedded from 10 fF of parasitic capacitance associated with the probing pads; blue) MBVD-model fitting of the measured admittance. The device static capacitance (C_0), motional resistance (R_m), quality factor (Q_{loaded}) and k_r^2 are extracted from the MBVD-model fitting [13]. Orange) 2D-FEA (dashed-line) and 3D-FEA (continuous-line) simulated admittances of the best fabricated CLMR.

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ALUMINUM NITRIDE VICINITY-BASED RESONANT CHARGING LINK

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ABSTRACT

This work reports on a low-cost and CMOS compatible approach for the synthesis of a through package charging and authenticating communication link enabled by GHz-range aluminum nitride (AIN) piezoelectric transducers. The acoustic link is formed by two acoustic transducers that leverage the enhancement of energy transfer at resonance. Power transfer efficiency (PTE) of -12 dB in agreement with the simulation results is measured for a transducer size of $1 \times 100 \times 100 \ \mu\text{m}^2$ through a 700 μ m thick silicon substrate. This work is the first step towards the development of next-generation high-efficiency and micro-scale power transfer units that could energize chips through packages or biomedical implants.

INTRODUCTION

The security and integrity of electronic systems is challenged by the presence of counterfeit integrated circuits (ICs) in the supply chain. Counterfeiters use a variety of easy and inexpensive techniques to recycle discarded ICs, alter them, and reintroduce them to the supply chain for profit. Introducing vicinity-based authenticating communication and charging capability into microscopic-scale chips that can be inserted into temper-resist packaging of ICs can be a viable solution for eliminating the prospect of counterfeiting from the electronic supply chain. Furthermore, near-field wireless powering of microsystems operating in liquid [1] or embedded in hazardous and inaccessible areas such as the human body [2] or sealed vessels [3] could be of great interest as frequent replacement of battery sources in such systems is cumbersome and expensive.

When dealing with metallic packages, the use of acoustic waves to power any IC is the only option as electromagnetic waves would decay exponentially into conductive materials [4]. Moreover, the use of acoustic transducers ensures higher PTE than any other wireless links over a distance that is 10~100 times larger than the acoustic transducer dimensions. Acoustic or ultrasound power transfer has been previously studied mostly for high power industrial applications operating at low frequencies [5]. Chip-scale acoustic resonators, with the same working principle but operating at higher/RF frequencies, have also been extensively researched in the MEMS field for frequency synthesis and selection [6]. However, chip-scale acoustic power transfer is yet to be fully explored. Due to record-breaking f-O products up to 10^{14} , high overtone bulk acoustic resonators (HBARs) [7] represent ideal candidates for the realization of power transfer units whose efficiency is dramatically enhanced by operation at resonance. Since fabrication of such structures requires no releasing step, they represent extremely robust solutions for various applications. Through substrate acoustic links have been previously demonstrated with HBAR devices based on different piezoelectric materials [8], [9], validating the high efficiency and low loss feature of such acoustic links. Hoople et al. [10] also reported the use of aluminum nitride transducers for on chip communication by sending an acoustic pulse from one transducer in an array, reflecting the pulse off the bottom surface of the silicon substrate, and receiving the pulse from a neighboring transducer in the array. Kuo et al [11] then took the concept one step further and used aluminum nitride transducers in a Through-Silicon-Via (TSV)-like communication link. However, none of these device have been applied to through packaging power transfer and charging. In this work, we introduce chip-scale acoustic transducers



Figure 1: Schematic presentation of the HBAR-like acoustic link used for through package power transfer.

in a two-port configuration as promising solutions for throughpackage energy transfer and charging. As a proof of concept, herein, we experimentally validate the acoustic link on double side polished single crystalline silicon substrates and prove, through simulations, how the approach can be extended to other materials.

DEVICE DESCRIPTION AND FABRICATION

The presented platform consists of two identical thin piezoelectric films each sandwiched by two metallic electrodes mounted on the two sides of a substrate (Figure 1). The longitudinal bulk acoustic waves (BAW) that are originated by vibrations of the top piezoelectric acoustic transducer propagate through the acoustic channel (the silicon substrate in this implementation) and are absorbed by an identical transducer on the other side of the package. Similar to FBARs, the individual transducers utilized here can be excited to vibrate across the thickness of the film (see Figure 2). The main piezoelectric coefficient used to excite the thickness mode vibrations is d_{33} . As a result, thickness mode vibrations can result in high electromechanical coupling making the technology suitable for broadband power transfer. The film thickness, *t*, sets the resonance frequency of the device for a given overall acoustic velocity of the material stack, c_{ac} (piezoelectric and metal films).

$$f_0 = \frac{1}{2t} c_{ac} \tag{1}.$$

Aluminum nitride is elected here as a CMOS-compatible and high electromechanical piezoelectric material. The fabrication process also only involves standard deposition and etching steps to ensure low cost. As a proof of concept, the devices are fabricated on a 700 um thick high resistivity double side polished silicon substrate. The process starts with sputtering and patterning of 100 nm thick Pt layers (bottom electrode) on both sides of the wafer using lift-off (Fig. 3a). 1 µm thick aluminum nitride layers are then deposited using sputtering and patterned via Cl2/BCl3/Ar based dry etch to access the bottom electrodes (Fig. 3b). In the next step, another 100 nm thick Pt layer is deposited and patterned on both sides to define the transducers' geometries (Fig. 3c). Finally, the electrical pads are coated with 300 nm thick gold layer (Fig. 3d) to enable flip chip bonding as well as to ensure good contact with the probes. For maximum performance, it is imperative to carefully control the thickness of AlN and Pt films on both sides to match so that the two transducers have close resonance frequencies. For instance, about 50 nm difference in the thicknesses of 3 GHz AlN transducer films results in ~40 MHz difference in their frequencies and ~1 dB degradation in overall PTE. Figure 4 depicts the optical microscopic images of the fabricated top and bottom transducers.

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Figure 2: COMSOL displacement mode shape.



Figure 3: Eight-mask CMOS compatible fabrication process.

The interesting aspect of such platform is that it does not involve any released parts, hence dramatically enhancing yields and making the structure more robust to any environment. Even though the top and bottom transducer geometries are identical, the two structures have different interconnects and traces to enable GSG probing on the top transducer and flip chip bonding on the bottom transducer.

DEVICE MODELING

The structure is formed by two equivalent transducers and is tested in a two-port configuration. The equivalent electromechanical model of the acoustic link can be obtained by means of connecting the output and input ports of the equivalent models of adjacent layers (Fig. 5). Taking advantage of Mason's model, electrode and packaging layers can be represented by two-port circuits, while three-port circuits are used for piezoelectric slabs [12]. As shown in the figure, the structure is modelled as a three electrical terminal device (effectively four, two of which are shorted together to ground), representing input, output, and ground. In Fig. 5, force, F, and acoustic velocity, v, components on the two sides of each layer play a role similar to voltage and current in an electrical circuit. The governing equations between force and velocity components can then be expressed as:

$$\begin{cases} F_{n_{i1}} = a_n v_{n_{i1}} + b_n (v_{n_{i1}} + v_{n_{i2}}) \\ F_{n_{i2}} = a_n v_{n_{i2}} + b_n (v_{n_{i1}} + v_{n_{i2}}) \end{cases} \quad n = p, m, a$$
(2)

where p, m, and a subscripts denote piezoelectric, metal, and packaging media, respectively. a_n and b_n are frequency dependent and can be written as:

$$\begin{cases} a_n = iZ_n \tan(\frac{k_n d_n}{2})\\ b_n = -iZ_n \sin(k_n d_n) \end{cases}$$
(3)

where Z_n is the characteristic acoustic impedance of the layers, d_n is



Figure 4: Optical microscopic images of the top and bottom transducers.

the layer thickness, and k_n is the wave number which is defined as:

$$k_n = \omega / c_n - j\alpha_n \tag{4}$$

where ω , *c*, and α are angular frequency, velocity of sound, and the acoustic attenuation coefficient. Attenuation coefficient lumps all kind of losses in the transducer as well as the packaging material. Since the attenuation coefficient tends to proportionally increase with frequency for most of materials, larger acoustic losses are expected at higher frequencies. However, it should be noted that due to the simplicity of the fab, ~3 GHz resonators employing 1 μ m thick AlN films are demonstrated in this work. Thicker films, up to 6 μ m [13], could be used to lower the device center frequency.

For the piezoelectric slabs a further force term is added to the right side of equation (2):

$$f_{p_i} = hC_0(V_i - \frac{h(v_{p_{i_1}} + v_{p_{i_2}})}{i\omega})$$
(5)

where h and C_0 are the piezoelectric constant and static capacitance of the rigidly attached transducer. When the material is piezoelectric, a third equation should also count for the electrical port:

$$I_i = i\omega C_0 V_i - h C_0 (v_{p_{i1}} + v_{p_{i2}})$$
(6).

When both the source and load are matched with the system impedance (50 Ω in most cases), the power transfer efficiency (PTE) can be calculated using the following equation:

$$G_T = |S_{21}|^2 \tag{7}.$$

By analyzing the equivalent electromechanical model via Advanced Design Systems (ADS) S-parameter simulations and assuming perfect alignment between the two transducers, PTE values for 700 μ m thick channels of different media at the center frequencies of 2.7 GHz and 500 MHz can be predicted as summarized in Table 1. It can be seen from Table 1 that by simply increasing the film thickness, lower operating frequencies and hence better PTEs can be achieved. Clearly, the acoustic link efficiency benefits by using lower center frequency at the expenses of bandwidth. In this manuscript, our goal is to present the general model and validate the principle of operation of the acoustic link. Trade-offs between PTE, bandwidth, and robustness will have to be considered depending on the specific application.



Figure 5: Equivalent electromechanical circuit of the acoustic channel.

Table 1: Simulated PTE prediction for other packaging materials at two different operating frequencies.

Packaging Material (700 μm Thick)	Attenuation Coefficient (α) @2.7 GHz, dB/cm	PTE (dB) for 1 μm AlN, 50 Ohm Load	Attenuation Coefficient (α) @500 MHz, dB/cm	PTE (dB) for 6 μm AlN, 50 Ohm Load
Rexolite [®]	950	-68.0	176	-13.0
Aluminum	55	-18.2	10.2	-3.4
Glass	18	-13.5	3.4	-2.5
Silicon	11	-11.4	2	-2.1

EXPERIMENTS

As a proof of concept and in order to demonstrate the predicted PTE values, identical transducers with less than 1% variations in their thicknesses were realized on double side polished silicon substrate and diced into 3×3 mm² dies. As illustrated in Figure 4, the transducer design was constrained to a maximum size of $100 \times 100 \ \mu\text{m}^2$ because of the specific IC application it targeted. To test the devices in the two-port configuration, the top and bottom transducers were designed for direct GSG probing and flip chip bonding with another silicon carrier chip, respectively. Gold patterned carrier substrate to be bonded with the backside transducers were fabricated on high resistivity silicon substrates using a specifically designed mask. The dimensions of the carrier silicon chips after dicing is 7×7 mm². Flip chip bonding was conducted in house using a Laurier M9 bonder. Direct gold to gold bonds were created through application of 1000 gr force between the two substrates heated up to 200 °C. Such bonds are produced without application of any conductive adhesive material. As it can be seen from Figure 6, to enable simultaneous probing of the bottom along with the top transducer, long gold traces had to be extended from the center of the chips to the open area of the bottom die. To probe the bottom transducers in the two-port configuration, the GSG probes land on the testbed silicon substrate (see Figure 7). In operation, the input RF signal of the network analyzer excites the AlN thin film on the top transducer into the thickness mode vibration creating longitudinal bulk acoustic waves that propagate through the silicon substrate and are received by the bottom transducer. The transducers performed as expected at 3 GHz showing a PTE of -12 dB when terminated with 50 Ω (Figure 8 and 9). As it can be seen from the figures, the experimental results agree well with the predicted values obtained from the analytical model.



Figure 6: Custom-made measurement setup via flip-chip bonding technique.



Figure 7: Custom-made measurement setup via flip-chip bonding technique.

It should be also noted that slightly better PTE values (~ 1 dB) can be achieved if the load and the source are terminated with optimized impedance values ($\sim 30 \Omega$ for this specific implementation).

The periodic nature of the frequency response is due to the packaging substrate that operates as a resonant cavity. Essentially when the substrate is of finite thickness, the resonant response is modulated by the effects of resonance in this cavity and the response of the piezoelectric layer becomes an envelope of all the harmonics due to the substrate. 6 MHz frequency separation of the harmonics is dictated by the velocity of the elastic waves and thickness of the packaging medium as

$$\Delta f = \frac{v_a}{2d_a} \tag{8}.$$

The quality factor, which is extracted from the individual harmonics at resonance, is largely determined by the acoustic losses in the "packaging material" (silicon here) and can be related to attenuation coefficient in the same media:

$$Q = \frac{\pi}{\alpha\lambda} \tag{9}.$$



Figure 8: 2-port PTE measurement of the acoustic link.

As the results suggest, a 6 dB bandwidth of about 1 GHz ensures that the acoustic link can operate efficiently over a broad range of frequencies.

CONCLUSION

This paper demonstrated the use of HBAR-like structures operating in the GHz range for power delivery across a package. ADS analytical simulations were conducted to predict the power gains at different frequencies and various packaging materials and thicknesses. The experimental results on the proof of concept demonstration on 700 µm silicon substrate confirm accuracy of the analytical model and shows an impressive PTE of about -12 dB, which is due to the crystalline structure and low attenuation characteristics of silicon. Although the most frequently used nonhermetic packaging material e.g. epoxy and PDMS, have very large values of attenuation coefficients resulting in poor power gains at GHz range frequencies, simulation results suggest that the state-ofthe-art packaging materials such as Rexolite® can be used to effectively deliver power with acceptable PTE values. Since the attenuation coefficient of the majority of materials drops linearly with frequency, it is also expected to achieve better PTEs for thicker AlN layers (lower frequencies). Future directions include showing operation through metallic packages as well as realization of much thicker AlN films (up to 6µm) for enhancing the acoustic channel PTE.

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Figure 9: 2-port phase measurement of the acoustic link.

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DIFFRACTION BASED TRANSMIT-RECEIVE DELAY ELEMENT WITH ZERO TEMPERATURE COEFFICIENT

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ABSTRACT

In this paper we present a pulse-echo transmit-receive ultrasonic delay element that is stable over time and has a novel diffraction based zero temperature coefficient of delay at two temperatures. The delay element is based on a CMOS compatible aluminum nitride (AIN) piezoelectric transducer requiring no release step, further improving reliability and reducing cost of production. The silicon bulk wave propagation leads to stable operation owing to low loss within silicon and large mode volume. The delay element achieves <6ppm stability and demonstrates zero temperature coefficient at 43° C and 72° C.

INTRODUCTION

Previous work from our group has demonstrated on-chip ultrasonic communications [1], ultrasonic delay line memory [2], and fingerprint sensing [3] (Fig. 1) using transit and receive links consisting of GHz ultrasonic pulses. The scaling of CMOS circuits in size and speed favor direct integration of GHz ultrasonic transducers with CMOS eliminating off-chip electronics or even bonded CMOS-MEMS chips, providing a pathway for a monolithic electronic to ultrasonic interface, eliminating the need for release steps. This paper illustrates a design strategy to achieve zero temperature coefficients at any desired temperature. This could allow one to design a 37°C, the nominal human body temperature, to measure body parameters with the delay as a reference. CMOS integrated delay-elements could be even more stable and programmable, owing to reduced parasitics.

A delay block is a circuit that shifts the input signal in time by a desired amount, and delivers a delayed output similar to the input signal. Delay elements can be used in wide range of applications including phase modulation in clocking systems as well as different digital systems. Furthermore, stable delay elements can be used as a timing reference in delay-locked-loops where the delay can be calibrated across different temperature and process variations. Precision and stability in a delay element block is one of the key specifications that directly impacts all of the aforementioned applications.

Different architectures for on-chip delay elements have been proposed in literature. The most commonly used delay method is an inverter based delay line where delay is proportional to the number of inverter buffers multiplied by the delay of a single buffer. This architecture is sensitive to process and temperature variations as well as supply voltage fluctuations. RC delay elements are also widely used in different delay based applications, however, this approach also suffers from high temperature dependence (2500 ppm/⁰C) [4]. In [5], a thyristor-based delay element is demonstrated. In this circuit, the delay is proportional to the current source as well as the threshold voltage of CMOS transistors, thus minimizing the supply dependence and hence enhancing the supply noise rejection. Minimum delay variation to power supply noise of 9.43% was reported with 314 PPM/⁰C temperature coefficient.

In this paper, a new ultrasonic based delay element is proposed. An ultrasonic wave packet, generated by a 75 μ 75 μ AlN piezoelectric transducers, propagates through the thickness of the silicon wafer (~725 um), where it experiences approximately total reflection at the bottom of the substrate due to the high acoustic



Figure 1: Applications of 3D integrated AlN transducers – reconfigurable phased array communication link, sonic through silicon vias, sonic delay line memories, and integrated impedance reflectometry.

impedance mismatch between silicon and air. Due to reflection of the initial pulse and continuous diffraction of the pulse, part of the ultrasonic wave front can be received by a neighboring AlN transducer on the top of the silicon (Fig. 4). The time for the acoustic wave to be received on this transducer is the delay between the transmitted wave and the first echo. This delay can be used as an on chip stable delay reference for different timing applications.

DIFFRACTION BASED TEMPERATURE COMPENSATION

In order to analyze the diffraction patterns, consider acoustic waves induced by a piezoelectric square transducer. The pressure at an off axis observation point can be written as [8]

$$p(\omega, x_o, y_o, z) = \frac{j\omega p_o}{2\pi v} e^{-j\left(\frac{\omega}{v}\right)\left[z + \frac{x_o^2 + y_o^2}{2z}\right]}$$
$$\iint_{-\infty}^{\infty} \frac{\operatorname{rect}\left(\frac{x_1}{L}\right)\operatorname{rect}\left(\frac{y_1}{L}\right)e^{j\frac{\omega}{vz}(x_ox_1 + y_oy_1)}e^{-j\frac{\omega}{vz}\left(x_1^2 + y_1^2\right)}}{R} dx_1 dy_1 \quad (1)$$

where p_0 is the pressure induced by the piezoelectric transducer at the transducer's surface, ω is the angular frequency of the acoustic waves, v is the speed of sound in silicon, z, x_0 , and y_0 are the Cartesian coordinates of the observation point, and L is the length of the square transducer. This expression is the Fresnel diffraction expression valid for $z^3 \gg \frac{L^4}{\lambda}$ [6].

The AlN pixels used have a resonant frequency of ~ 1.3 GHz, and a lateral size of 75 um, the silicon substrate thickness is 725 um, and the speed of sound in silicon is 8433 m/s. As this thickness is much longer than the Fresnel zone length, and also satisfies the Fraunhofer approximation $(z \gg \frac{L^2}{\lambda})$, equation (1) can be approximated to

$$p(\omega, x_o, y_o, z) = \frac{j p_o \beta L^2}{2\pi R_o} e^{-j\beta \left[z + \frac{x_o^2 + y_o^2}{2z}\right]} sinc\left[\frac{L x_o}{\lambda z}\right] sinc\left[\frac{L y_o}{\lambda z}\right]$$
(2)

where β is the wave number of the acoustic wave $(\beta = \frac{\omega}{v})$. Destructive interference from the infinite point sources at the surface of the piezoelectric transducer results in nulls in the diffraction pattern along the x-axis. This can be seen from the

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locations of the zeros in the function sinc(x), which are located at all values of $x = n \pi$, where n is an integer ($n \neq 0$). Due to symmetry, diffraction patterns along the x-axis and the y-axis are identical. At $y_0 = 0$, the pressure distribution along the x-axis follows the equation

$$p(\omega, x_o, y_o = 0, z) = \frac{j p_o \beta L^2}{2\pi R_o} e^{-j\beta \left[z + \frac{x_o^2}{2z}\right]} sinc\left[\frac{L x_o}{\lambda z}\right]$$
(3)

where
$$p = 0$$
 for $x_o \approx \frac{\lambda Z}{L}, \frac{2\lambda Z}{L}, \frac{3\lambda Z}{L}, \dots \dots$

From the above expressions, it can be seen that the far field diffraction angle is directly proportional to the wavelength of the propagating pulse, and inversely proportional to the size of the input piezoelectric transducer. Fig. 2 shows the plot of the diffraction pattern for 3 different wavelengths at the far field.



Figure 2: Far field diffraction patterns for different wavelengths at z=1.7mm, D=75 um.

In this setup, both the transmitting and the receiving transducers are side by side on the top of the silicon substrate. The transmitting transducer is actuated by an input pulse modulated at the resonant frequency of the transducer. The transmitted pulse is reflected at the bottom of the substrate and received by the receiving transducer. This results in the first echo for the transmitted pulse. Due to acoustic impedance mismatch at the top of the silicon substrate, most of the signal power reflects back to the substrate and takes another round trip through the silicon bulk. Throughout the rest of the paper, the signal received after one round trip will be referred to as the first echo and the signal received after two round trips will be referred to as the second echo. As an approximation, the first echo diffraction pattern will be modeled by substituting Z with double the silicon chip thickness (~ 1.45 mm), similarly, the second echo diffraction pattern will be modelled by using a Z equals to four times the chip thickness (~ 2.9 mm). Fig. 3 is a schematic for the transducers on top of the silicon chip showing the dimensions used and the diffraction patterns for the first and second echoes.

The effect of temperature on the speed of sound in silicon has been discussed in detail in the literature [7]. The acoustic velocity is given by $v = \sqrt{E/\rho}$, E is the stiffness of the material and ρ is its density. According to [7], for acoustic waves propagating perpendicular to the (100) Si wafer plane, the stiffness decreases approximately linearly with the temperature increase with a temperature coefficient of -78.8*10⁻⁶ /°C. Temperature coefficient is calculated by the equation $TC = \Delta E/(E_0\Delta T)$, where ΔE and ΔT are the difference in stiffness and temperature respectively, and E_0 is the stiffness at room temperature.



Figure 3: Schematic for the device used and the two diffraction patterns for the 1st and 2nd echoes.

Temperature impacts the delay of the delay cell in several ways. As temperature increases, stiffness of the delay medium decreases, and as a result, acoustic velocity is reduced. This causes delay to increase linearly with temperature. Also, thickness of silicon increases with temperature through the thermal expansion coefficient of Silicon. The total delay between the two echoes can thus be represented by the equation

$$\Delta t = \delta_2 - \delta_1 + (t_{rise2} - t_{rise1}) \tag{4}$$

$$\delta_i = \frac{l_i}{c} = \frac{1}{c_0(1-|\beta|\Delta T)} \frac{2 i t_w(1+\alpha\Delta T)}{\cos(\theta_i(T))}$$

$$\theta_i = \Gamma_i \lambda / t_w = \Gamma_n c(T) / (ft_w) = \frac{\Gamma_n c_0(1 - |\beta| \Delta T)}{ft_w(1 + \alpha \Delta T)}$$

Where δ_i is the delay time between the initial excitation and the ith echo due to the path length l_i , t_w is the thickness of the silicon, c is the speed of sound, T is the temperature, f is the frequency, θ_i is the diffraction angle corresponding to the appropriate null, as in Fig. 3, and Γ_n is an integer that corresponds to the sinc extremums t_{rise1} and t_{rise2} are the rise times of the first and second echoes, respectively. β is the temperature coefficient by which the speed of sound decreases in silicon (~ -50 ppm/°C). α is the thermal expansion coefficient of Silicon (~ 2.6 ppm/°C). These values indicate that the delay is always a rising function of temperature.

However, as shown in equation (4), additional delay is present due to the circuit effect on measuring the rise time. In Fig. 2, as the wavelength decreases, angle of diffraction decreases. Consequently, the amount of received power changes, resulting in changing the rise time of the received echoes which in turn changes the delay. As an example, in Fig. 3, in the case of the first echo, as temperature increases, the third peak of the diffraction pattern moves away from the receiver transducer range. Consequently, the amount of signal power received at higher temperature is less than the amount received at room temperature. Conversely, for the second echo, as temperature increases, the first peak in the diffraction pattern moves towards the range of the receiver transducer. As a result, the amount of signal power received gets higher. As will be shown later, the rise time of the echo pulse is inversely proportional to the received signal amplitude (total received power). So, trise1 increases and trise2 decreases causing the total delay in equation (4) to decrease. After a certain threshold the behavior will be reversed and the received signal amplitude increases with temperature at the first echo and decreases with temperature at the second echo causing the delay between the first and the second echoes to increase. Fig. 4 shows how the received signal amplitude changes with temperature for the first and the second echoes.

An envelope detector is used to down convert the signal from the resonant frequency of the transducer to baseband. We can model the electronic circuit as a rectifier. The signal is rectified using a half wave rectifier Zener diode, then a capacitor is charged by the rectified wave to track the envelope of the signal. The change of rise time is related to the change in the signal amplitude (received signal power) by the capacitor charging equation

$$t_{rise} = R_{eff}C \ln\left(1 + \frac{V_T}{V_{peak}}\right)$$

$$\Delta t_{rise} = t_{rise2} - t_{rise1} = R_{eff}C \ln\left(\frac{V_{Peak1}(V_{Peak2} + V_T)}{V_{Peak2}(V_{Peak1} + V_T)}\right) \quad (5)$$

trise is the pulse rise time in sec, R_{eff} is the effective resistor charging the capacitor in ohms, C is the capacitance of the capacitor in Farad, V_T is the switching threshold voltage, and V_{peak} is the input signal amplitude. V_{Peak1} and V_{Peak2} are normally very close to each other in value, hence, equation (5) can be approximated by the Taylor expansion of the first term giving

$$\Delta t_{rise} \approx R_{eff} C \left(\frac{V_{Peak1}(V_{Peak2} + V_T)}{V_{Peak2}(V_{Peak1} + V_T)} - 1 \right)$$
$$\Delta t_{rise} \approx \frac{-R_{eff} C V_T \Delta V_{Peak}}{V_{Peak}(V_{Peak} + V_T)}$$
(6)

From (6), as the received power changes (V_{Peak}), the rise time also changes. At small variation in peak voltage, Δt_{rise} changes linearly with ΔV_{peak} and the coefficient of variation is proportional to the envelope detector circuit parameters (R_{eff} and C). This second term has a larger component of delay measurement, and makes the delay sensitive to a resistor and capacitance. Hence, if on-chip resistors and capacitors are used, the delay could be susceptible to on-chip process variations and components aging.



Figure4: Measured change in peak voltage with temperature for the two pulses

EXPERIMENT SETUP

The experiment setup (Fig. 5) comprises an RF signal generator, a pulse generator, an RF switch, the transducers device, a thermocouple to measure the temperature, an oven, an envelope detector, and a frequency counter with high precision to accurately measure the delay. The RF switch is controlled by the pulse generator to create pulses modulated by the high frequency signal generated by the RF signal generator. The pulse width should be less than the time of flight of acoustic waves in silicon so that echoes do not overlap. In our experiment, time of flight for a round trip in silicon is ~175 ns, so, the pulse width was chosen to be 80 ns. The

pulse is then used to actuate the transmit AlN transducer. The wave generated by the piezoelectric transducer of width 75 um propagates through the thickness of a silicon wafer as illustrated in Fig. 4. The envelope detector is then used to down convert the received signal and the frequency counter is used to measure the delay between the first 2 echoes.



MEASUREMENT RESULTS

Fig. 6 shows the measured signals at the output of the amplifier and the output of the envelope detector. The first pulse is due to electrical coupling of the transmit signal to the receive transducer. This is followed by several acoustic echoes. In this paper, the reference delay of interest is the delay between the first and the second echo. However, the same analysis applies to the delay between all consecutive echoes. At room temperature, the delay between each two consecutive pulses is ~175ns. The input frequency to the envelope detector is 1.31 GHz which is the resonance frequency of the piezoelectric transducer.



Figure 6: Experiment waveforms showing the delay of interest.

The Allan deviation curve is calculated for the delay of interest as a metric of stability. The Allan deviation is the square root of the Allan variance, which is half of the average of the squares of the differences between successive delay readings over a certain time period. The Allan deviation allows different types of noise to be distinguished based on the Allan curve slope when plotted on loglog plot. Fig. 7 shows the Allan deviation curve for the delay between the first and the second echoes. The curve starts with a slope of -1/2 due to white noise, then slope starts to decrease until it flattens at the flicker noise floor, where more averaging doesn't help in noise reduction. Unlike the conventional Allan deviation curves, the curve doesn't start with a -1 slope which means that the system doesn't suffer from quantization noise. A minimum standard deviation of 0.87 ps is achieved which corresponds to a delay error of 5 ppm.



Figure 7: Allan deviation of t_{delay} . Averaging delays over 4 seconds results in an Allan deviation of 0.87 ps

Fig 8. shows the measured delay across temperature. The effect of the diffraction pattern change discussed earlier can be clearly seen. The temperature effect on t_{delay} in PPM/⁰C is in Fig. 9. Around 43°C and 72°C, the temperature coefficient is 0. These values can be changed through the experiment setup parameters like the receiver position, the actuation frequency, and the effective resistance and the output capacitance of the envelope detector, allowing one to program to 37°C, body temperature, for human body sensing modalities.



Figure 8: Measured delay between the first and the second echoes across temperature

CONCLUSION

In this paper, a CMOS compatible delay element based on ultrasound pulse packet propagation through the silicon bulk is introduced. The delay element showed high long term stability with an Allan deviation of 0.87 ps (<6ppm). Also, due to the diffraction angle dependence on temperature, a zero temperature coefficient is achieved at 43°C and 72°C. The transmit-receive geometries can be designed with different spacing, higher number of electrodes for differential delay measurement and differential receive electronic characteristics. Further design is needed for delay-locked loops to investigate the stability of frequency sources.



Figure 9: Temperature coefficient in PPM/°C across temperature showing measured data and a fitted curve.

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ELECTROSPUN POLYCAPROLACTONE NANOFIBERS AND MICROBEADS LOADED WITH DEXAMETHASONE FOR EXTENDED OCULAR DRUG DELIVERY

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ABSTRACT

A dexamethasone (DX) loaded electrospun polycaprolactone (PCL) nanofibers/microbeads hybrid drug release system is fabricated and characterized. Drug release profiles change for the different ratio of nanofibers and microbeads, which is controlled by the electrospinning parameters such as the solution viscosity and the tip-to-collector distance. Also, it is observed that the electrospun nanofibers/microbeads show enhanced polymer orientation and crystallinity, resulting in significantly lower diffusivity than that of the spin-cast structures. The hybrid system shows an extended release time longer than a month.

INTRODUCTION

Ocular drug delivery using traditional eye drops suffers from limited absorption due to the barrier of corneal epithelium and rapid precorneal loss with tears [1]. Alternatively, direct intravitreal injection is used, which could cause retinal detachment, hemorrhage, endophthalmitis, and cataract [2]. Controlled release over an extended period of time with reduced injection frequency is desired.

Electrospinning is an economic fabrication method for producing nanofibers [3]. Microbeads are formed when the electrospinning conditions are altered. As the sizes of nanofibers and microbeads are different, the release profiles of embedded drug differ, which can be exploited for controlled release. Figure 1 shows a hybrid release system consisting of nanofibers serving as a fast releasing medium and binders between microbeads, while microbeads release drug much slowly, showing two release profiles. Figure 2 shows the schematic of the electrospinning process and the resultant polymer architecture of nanofibers and mibrobeads. Solution viscosity and tip-to-collector distance (TCD) affect the morphology [4-5]. Microbeads are formed in a low viscous condition due to low surface tension. Small TCD produces excessive residual solvent in fibers, transitioning to microbeads.



Figure 1:Schematic of a hybrid controlled drug release system with two release profile from nanofibers and microbeads.

Polycaprolacton (PCL) is a biodegradable polymer with a long biodegradation profile [6]. Dexamethasone (DX) is known to relieve ocular symptoms caused by infection, allergies and persistent macular edema. In this work, a DX loaded electrospun PCL hybrid nanofiber/microbead system is demonstrated for controlled release. Four samples are fabricated in different polymer viscosity and TCD conditions. Two solvents are used. Composition and operational parameters are summarized in Table 1. An electric field strength of 1 kV/cm is used for electrospinning.



Figure 2: Schematic of the electrospinning setup and polymer parameters for the analytical modeling of diameters.

RESULTS AND DISCUSSIONS

Figure 3 shows SEM images of the fabricated samples. Samples 1 and 2 with different solvents show pure nanofiber morphology with an average diameter of 307.4nm \pm 8.2nm and 669.4nm \pm 342.5nm, respectively. It shows the nanofiber diameter relies on solvent properties. Hybrid nanofibers and microbeads are observed in Samples 3 and 4, where the PCL concentration is diluted. The microbead diameters are 1.56 µm \pm 0.6 µm and 3.68 µm \pm 1.5 µm, respectively. The volume percentage of microbeads increases by decreasing TCD from 40.15% to over 90%.



Figure 3: SEM images of electrospun 16 wt/vol% of PCL in (A) DCM/DMF and (B) acetone/ethanol, and 4 wt/vol% of PCL in DCM/DMF with (C) 12.5 cm and (D) 7.5 cm.



Figure 4: Analytical model prediction and measurement of the microbead radius with varying tip-to-collector distance.

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An analytical model for the dimensions of nanofibers/microbeads is obtained using the volume conservation theory and flow rate equation shown in (1) and (2).

$$\begin{array}{l} 4/3\pi r_{MB}{}^{3} + \pi r_{NF}{}^{2} \, L_{NF} = C \\ Q = (dp/dL) \, A_n \end{array}$$
(2)

where r_{MB} , r_{NF} , L_{NF} , dp/dL, and A_n are the microbead radius, nanofiber radius, nanofiber length, pressure gradient, and cross-section area of the needle tip. Figure 4 shows the microbead radius based on the model prediction and experimental results.



Figure 5: Drug release of nanofiber stacks with different thickness.

The release profiles of nanofiber stacks with different thicknesses are shown in Figure 5. Figure 6 shows the drug release profile of all samples. Samples 1 and 2 have similar profiles within 10 hours. The releasing time of Sample 3 is around 18 days, more than 40 times extended. Sample 4 shows less than 90% of drug released within 1 month.



Figure 6: Release profile of pure nanofibers and hybrid samples.



Figure 7: X-ray diffraction measurement of electrospun PCL nanofibers/microbeads and spin-cast PCL thin film.

Figure 7 shows the X-ray diffraction (XRD) measurements to study the effect of polymer orientation. Electrospun samples are

compared with spin-cast thin film. Full-width-half-maximum (FWHM) extractions show that electrospinning can enhance polymer crystallinity and orientation due to external electrostatic force and stress. The properties of all the samples are summarized in Table 1.

Table 1: Summary of pure nanofiber and hybrid structure.

	Sample 1	Sample 2	Sample 3	Sample 4
Solvent	16 wt/vol% DCM/DMF*	16 wt/vol% Acetone/ ethanol	4 wt/vol% DCM/DMF*	4 wt/vol% DCM/DMF*
TCD (cm)	12.5	12.5	12.5	7.5
Nanofiber diameter (nm)	300	670	108	92
Nanofiber Volume %	100	100	59.85	8.70
Microbead Diameter (nm)	N/A	N/A	1500	3680
Microbead volume %	N/A	N/A	40.15	91.30
Nanofiber diffusivity (10 ⁻¹¹ mm ² /hr)	725.5 ± 111.0	1286.5 ± 77.1	18.07 ± 7.20	2.59 ± 0.48
Microbead diffusivity (10 ⁻¹¹ mm ² /hr)	N/A	N/A	14.27 ± 1.65	97.34 ± 11.53

*DCM/DMF: Dichloromethane/Dimethylformamide

CONCLUSIONS

An electrospun PCL hybrid drug release system consisting of nanofibers and microbeads was developed. It continuously releases DX over a month at room temperature. The hybrid system provides continuous long term delivery and localization of the microbead in the eye. XRD data show the electrospinning process results in lower diffusivity polymer structures. This process could be favorably used as an infrequent treatment alternative.

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FLEXIBLE SYSTEMS FOR WEARABLE PHYSIOLOGICAL MONITORING APPLICATIONS

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ABSTRACT

A flexible-system integration strategy is demonstrated which merges plastic based sensors that interface with the skin with silicon integrated circuits consolidated on a flexible printed circuit board for complex signal processing. As an example application, we present a flexible fully-integrated and wireless perspiration biosensor that accurately and simultaneously measures a panel of electrolytes and metabolites in sweat in real-time while calibrating the sensors' response against the change in skin temperature.

INTRODUCTION

Wearable biosensors have a significant role in the future of the healthcare as they facilitate real-time and non-invasive monitoring of an individual's health state. The implications of such wearable devices are far reaching and include various applications, such as fitness and athletic monitoring, as well as remote health monitoring of toddlers, elderly people and patients with disabilities or conditions that are in continuous need of a caregiver's attention. Currently commercialized wearable sensors are only capable of tracking an individual's physical activities and vital signs and do not provide insightful physiological information at molecular levels.

The advances in the field of flexible electronics have opened up the possibility of developing new classes of sensors that form intimate contact with human body and analyze previously underexplored physiologically rich sources of information (e.g. sweat, skin etc.). For these wearable biosensors to find application in clinical and translational medicine settings, an integrated-system approach must be adopted to ensure that the originated signals at the sensor level are accurately processed, calibrated against other influential parameters, and are seamlessly transferred to a display/storage unit or cloud servers online. Accordingly, on-site analog/digital signal processing and data transmission functionalities are required that interface the sensor unit. To this end, silicon-based integrated-circuit technologies should be utilized as they have already been optimized for low power and wireless consumer applications. The challenge remains in devising an integrated system which meets the mechanical constraints at the sensor level and the electrical constraints at the circuit level while providing a fully flexible solution that can be comfortably worn.

FLEXIBLE INTEGRATED SYSTEMS

Here, we devise a flexible-system integration strategy which merges plastic based sensors that interface with the skin with silicon integrated circuits (ICs) consolidated on a flexible printed circuit board (FPCB) for complex signal processing (Figure 1). This approach decouples the aforementioned stringent mechanical and electrical requirements, and at the same time exploits the strengths of the underlying technologies.

As an example application, we demonstrate a flexible fullyintegrated and wireless perspiration biosensor that accurately and simultaneously measures the main electrolytes (e.g. sodium and potassium) and metabolites (e.g. glucose and lactate) of sweat in real-time while calibrating the sensors' response against the change in skin temperature [1]. In this approach, the sensors are patterned and functionalized on a mechanically flexible polyethylene terephthalate (PET) substrate and the IC chips and other circuit components are assembled on a thin polyimide-based PCB (thickness ~200 μ m). The electrolyte sensors are potentiometric based and are realized with the aid of ion-selective membranes.









Figure 1: (a) Schematic of the sensor array (b) Top-view of a flattened flexible PCB. The PCB amplifies, filters, and calibrates the transduced signals. It then wirelessly transmits the data through Bluetooth to a custom-developed mobile application. (c) The fully flexible integrated platform where plastic-based sensors interface the flexible wireless PCB.

RESULTS AND DISCUSSION

As shown in Figure 2, the electrolyte sensors generate electrical voltage that is logarithmically proportional to the abundance of the respective electrolytes in sweat.

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Figure 2: The potentiometric electrolyte sensor responses to the respective analyte solutions in phosphate-buffered saline (PBS) (a) sodium and (b) potassium sensors.

The metabolite sensors are enzymatic based and are functionalized with glucose oxidase and lactate oxidase (Figures 3a and 3bb).



Figure 3: The enzymatic-based metabolite sensor responses to the respective analyte solutions in phosphate-buffered saline (PBS) (a) glucose and (b) lactate sensors. (c) The resistance response of the temperature sensor to temperature changes in PBS.

These enzymatic sensors generate electrical current that is linearly proportional to the abundance of the respective metabolites in sweat. Here, the temperature sensor information (Figure 3c) not only serves as a useful marker of the thermal state of the individuals, but also is required to calibrate the enzymatic sensor readings against the skin temperature.

The form factor of this flexible platform allows for its placement on various body parts such as forehead and wrist. This platform is used to measure the detailed sweat profile of human subjects engaged in prolonged indoor and outdoor physical activities. Figure 4 illustrates the representative glucose and sodium readings from real-time perspiration monitoring on the forehead of a subject during stationary leg cycling.



Figure 4: On-body real-time perspiration analysis during stationary cycling with varying power output. Representative sweat glucose and sodium readings are plotted (the accuracy of in-situ readings were verified through comparison with sweat samples collected at discrete time points)

We envision that this platform could be exploited or reconfigured for *in-situ* analyses of other biomarkers within sweat and other fluid samples to facilitate personalized and real-time physiological and clinical investigations.

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WEARABLE GRAPHENE SENSORS ON ADHESIVE TAPES

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ABSTRACT

We report a simple, reliable, and cost-effective method of fabricating wearable graphene-based physical sensors on adhesive tapes for detecting mechanical signals, such as pressure, force, and strain. The device fabrication mainly involves two steps, including patterning graphene by repeated "sticking and peeling off" processes with scotch tapes, and then transferring the patterned graphene to another adhesive tape. Once a reusable mold is formed by soft lithography, no further film deposition, lithography or etching processes are required to obtain graphene-based wearable sensors. We demonstrate using the strain and pressure sensors made on polyimide tapes to track body motion, as well as monitor pressure applied to a flexible object.

INTRODUCTION

Wearable sensors have been widely used in real-time motion tracking, human and structural health monitoring, smart prosthetics, and humanized robotic systems. In particular, flexible mechanical sensors with high sensitivity and low cost, such as pressure and strain sensors, have attracted considerable attention. Mechanical flexibility is crucial in developing wearable sensors, which may allow for direct contact between the sensors and sensed surfaces of objects with irregular shapes. Recently many elastomer-based conductive composites have become popular sensor materials for wearable mechanical sensors. They are formed by introducing different conductive nanomaterials (e.g., carbon nanotubes, graphite, etc.) into flexible and stretchable elastomers (e.g., silicone, polyimide, polyethylene, etc.), exhibiting good flexibility and electrical conductivity. Also, due to high piezo resistivity and stretchability, the two-dimensional material, graphene, has become a promising candidate for sensitive material of pressure and strain sensors formed on various flexible and streachable substrates [1, 2].

Here we report a simple, reliable, and cost-effective method of fabricating wearable graphene sensors on adhesive tapes for detecting pressure, force, and strain.

FABRICATION PROCESS

Fig. 1(a) depicts the fabrication process. First, SU-8 mold was obtained by conventional soft photolithography method, then the desired channels (with 0.21mm depth) was obtained on PDMS by pouring, thermally curing, and peeling it off from the SU-8 mold. Then, a graphene dispersion in ethanol and distilled water mixtures (volume ratio of 70:30 [3]) was sonicated for 400 min and drop cast onto a polydimethylsiloxane (PDMS) mold pretreated with oxygen plasma. After drying on a hotplate at 90 °C for 5 min, a graphene film was formed, covering the whole surface of the mold. Subsequently, scotch tapes were used to stick and peel off multiple times until all excess graphene was removed from the top surface of the PDMS mold, leaving behind the desired graphene filled in the channels of the mold. Then, another adhesive tape (here, 25 µm thick polyimide film with adhesive on one side) was laid flat and adhered to the PDMS mold. Afterwards, the whole structure was annealed in an oven at 200 °C for two hours. The annealing treatment allows increasing electrical conductivity of the graphene patterns by about two orders of magnitude and strengthening the adhesion between polyimide and graphene. Lastly, the thin polyimide tape was peeled off from the mold and the conductive graphene patterns were transferred to the polyimide tape. Fig. 1(b)

shows transferring of graphene patterns from microfluidic channels onto a polyimide tape. Fig. 1(c) shows different patterns formed on the tapes. Fig. 2 shows scanning electron microscopy (SEM) images for the multilayer graphene patterns onto the polyimide tape.



Figure 1: (a) Schematic of fabrication processes to make graphene sensors on adhesive tapes. (b) Photos showing how to transfer graphene patterns onto a polyimide tape. Scale bars: 4 mm. (c) Fabricated graphene structures. Scale bars: 1 mm.



Figure2: Scanning electron microscopy of the graphene patterns transferred onto a polyimide substrate.

RESULTS AND DISCUSSION

Fig. 3a shows the relative resistance change of the graphene sensor on the polyimide tape (Fig. 1b) as a function of applied tensile strain along the length of the graphene pattern. The response of sensor to tensile strain is almost linear. The strain sensor is adhered on the middle joint of index finger (Fig. 3c) to track different bending positions of the finger (Fig. 3b). As the finger bends, the resistance of the sensor increases. Once the finger goes back flat from bending position, the resistance follows to go back to its initial value, indicating that the sensor has high reversibility.

As another application of graphene pattern sensor, the sensor is attached to the surface of a balloon to track dynamic changes in tension on the balloon during inflation. (Fig. 4). The pressure of air source is kept at 3 pounds per square inch or PSI during the measurement, while the duration of flowing the air to the balloon changes. Fig. 4 shows that as the balloon is inflated, the resistance of the sensor increases. As the balloon stays at a given inflated state, the resistance becomes stable.

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	Pressure (kPa)					ΔR/R (%)				
	F1	F2	F3	F4	F5	B1	B2	B3	B4	B5
Preparation	0	0	0	0	0	1.7	1.5	1.2	1.3	1.4
Adjustment	4.4	5	6.5	5.3	2.5	3.5	3.4	3.2	3.1	3.5
Catching	420	456	547	467	469	4.9	4.6	4.7	4.3	4.2

Table 1: Relative resistance changes of five pressure sensors (F1-F5) and five strains sensors (B1-B5) attached to fingers during catching a tennis ball



Figure3: (a) Relative resistance change as a function of tensile strain applied along the surface of polyimide tape. (b, c) Demonstration of tracking motion of the index finger.



Figure 4: Demonstration of tracking changes in tension on the balloon during inflation.

The graphene pattern formed on the tape can detect pressure applied to the surface of the pattern. Fig. 5a shows the relative resistance change of the graphene sensor as a function of external pressure applied normal to the surface of the graphene pattern. The mouse clicking pressure is monitored using the sensor attached on the left side of the mouse. When the mouse is clicked, the resistance of the sensor becomes lower and when the clicking pressure increases the relative resistance change becomes more. Fig. 5b tracks how the sensor responds to different clicking pressure and frequency.

Fig. 6 demonstrates using two sets of sensors (total 10) to track bending and pressure levels involved during catching a tennis ball bounced up from the hard floor. Each finger is equipped with a strain sensor at the top of the finger joint and a pressure sensor at the fingertip. Table 1 shows the obtained pressure and strain values at the three states: preparation, adjustment, and catching.



Figure 5: (a) Relative resistance change as a function of pressure applied normal to the surface of the tape. (b) Demonstration of tracking mouse clicking pressure and frequency.



Figure 6: (a, b) Positions of five strain sensors (F1-F5) and five pressure sensors (B1-B5). (c) Demonstration of tracking pressures and strains during catching a tennis ball. The pressure and strain signals are read out at three states: preparation (left), adjustment (middle), and catching (right).

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CAVITY OPTICAL TRANSDUCER PLATFORM WITH INTEGRATED ACTUATION FOR MULTIPLE SENSING APPLICATIONS

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ABSTRACT

We present an on-chip cavity optomechanical transducer platform that combines high measurement bandwidth and very low displacement noise floor with compactness, robustness, small size, and potential for low cost batch fabrication inherent in microelectro- mechanical- systems (MEMS) [1]. Our fiber-pigtailed transducers use surface-micromachined silicon-on-insulator photonic, low-stress silicon nitride structural and metal electrical actuation layers, while front- and backside bulk micromachining defines v-grooves and overhanging cantilevers. The motion of the mechanical devices, such as cantilevers and high mechanical quality factor membrane resonators, is optically measured by integrated silicon micro disk optical cavities. The devices can be actuated electrothermally or electrostatically, and this actuation can also be used to tune readout gain. A displacement noise floor below 10 fm/vHz is achieved for mechanical devices with varying over three orders of magnitude stiffnesses (≈ 0.2 N/m to ≈ 200 N/m). The combination of electrical actuation, low-loss mechanics, and optomechanical readout will enable a wide variety of high-performance on-chip resonant and non-resonant sensors.

INTRODUCTION

The measurement of physical quantities by transducing them to a mechanical motion has a long history. The recent advancements in fabrication of micro- and nanomechanical resonators have continued this trend. Ongoing miniaturization and better process control have enabled high quality factors for both optical and mechanical resonators and therefore more sensitive measurement of microscopic physical phenomena. While micromechanical pressure and acceleration sensors are now ubiquitous in consumer electronics and other products of everyday life, in the physics laboratory the micro- and nanoscale resonators allow measurements with unprecedented degree of precision [2].

One of the most significant obstacles to realizing the full potential of micro- and nanomechanical sensing is the readout of the motion of the small resonator with high sensitivity, high bandwidth, and without excess power dissipation. In the past years numerous methods for the readout of resonator motion have been developed [3]. Electrical readout schemes, such as capacitive, magnetomotive, piezoresistive, and piezoelectric, are convenient but suffer from various combinations of poor scaling with reduced size, power dissipation limitations, magnetic field and material requirements, and thermal Johnson noise in the readout signal. On the other hand, optical readout schemes, such as beam deflection and interferometric, substitute optical shot noise for thermal noise, in principle don't dissipate any power at the transducer, and have a high measurement bandwidth. However, to effectively couple motion to light, most of the off-chip optical methods need a certain minimum moving structure size and reflectivity, which often involves bulky structures or mechanically dissipative reflective coatings.

In nanophotonic optical cavities, the light is trapped in a very small volume and is made to interact for a longer timer and more closely with the mechanical resonator. Typical photonic cavity optical quality factors on the order 10^5 to 10^6 increase the readout signal to noise by the same factor. The readout bandwidth is reduced from ≈ 100 THz optical frequency to about ≈ 100 MHz, still fast enough for most mechanical sensors. Maintaining stable coupling of a microscopic mechanical resonator with an off-chip optical cavity is challenging due to alignment and drift of components with respect to each other. Here this challenge is overcome by integrating the high quality factor optical cavity directly underneath the moving device, allowing strong interaction with the optical near-field of the cavity, while avoiding mechanical This interaction is described by the contact (Fig.1). optomechanical coupling coefficient (g_{OM}) relating the change in optical frequency of the micro disk cavity to the displacement of the mechanical device. This fully integrated stable and practical optomechanical transducer is fiber connectorized and implements the readout of mechanical motion with gigahertz bandwidth.

Low loss, stable and robust fiber coupling of the transducer is essential to allow sensitive and reliable operation. Therefore, the fibers have to be securely attached to the chip without the introduction of high excess losses between the on-chip waveguide and the optical fiber.

This readout approach allows independent tailoring of the various optical and mechanical parts of the transducer. The photonics can be separately optimized for low losses, high quality factor and desired cavity size, while tuning the waveguide-cavity coupling depth and the optomechanical coupling to achieve the optimal readout sensitivity and dynamic range. The mechanical components' size, shape, stiffness, and resonance frequency can be tailored to best address the specific sensing applications. The actuation can be tailored for the needed displacement and force ranges, ideally without introducing mechanical losses, avoiding increased mechanical noise and decreased Q in resonators.



Figure 1: Exemplary schematic of the transducer (not to scale) showing overhung cantilever on a torsional pivot as the mechanical device. SiN is shown in green, Si is shown in blue and grey. The red arrow indicates the direction of movement.

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TRANSDUCER PLATFORM COMPONENTS AND DESIGN

Figure 1 schematically illustrates the arrangement of the different components in our transducer platform, such as the optical fiber, inverse-taper coupler, waveguides, microdisk cavity, and the mechanical (torsional cantilever) structure. Electricallycontrolled actuators (not shown) are also included in the platform to tune the static position and dynamically excite the motion of the mechanical device. The photonic structures for operation in the telecommunication wavelength range are fabricated in the silicon device layer of a silicon-on-insulator (SOI) wafer, because of the outstanding optical and mechanical properties of silicon. The mechanical device is created in silicon nitride (SiN), because it shows good mechanical properties resulting in high quality factor devices, has low optical loss, an index of refraction below that of Si and is compatible with a hydrofluoric (HF) acid release. For the metallization we choose gold with a chromium adhesion layer (Cr/Au), compatible with HF and potassium hydroxide (KOH) etches. Furthermore, the combination of SiN and Cr/Au shows a good thermal bimorph actuation efficiency. Silicon dioxide is used as the sacrificial material.

The sensitivity to motion is proportional to the optical quality factor of the micro disk cavity and quickly increases with decreasing gap between the cavity and the mechanical resonator [1]. It is therefore important to accurately locate the mechanical structure in close proximity to an optical micro disk cavity, while maintaining the high optical quality factor of the micro disk optical mode. In our design, the micro resonator is lithographically aligned to the disk, and completely encloses it, while a sacrificial layer defines the gap in the fabrication process. Dedicated lithography and etch steps are used to reduce the sacrificial layer thickness to a predetermined value at the optomechanical transducer, allowing us to control the gap within tens of nanometers, while keeping a thicker silicon dioxide sacrificial and cladding layer elsewhere

Coupling light from optical fiber to an on-chip waveguide may result in high losses due to mode-size and effective-index mismatch between the optical fiber and the Si waveguide structure, which induce optical scattering and backreflection. Tapering from the waveguide dimensions to the fiber mode dimensions for improving coupling efficiency between optical- fiber and waveguide modes have been suggested [4]. However, to avoid excessive coupling to radiation modes in the taper the required typical taper length must be of the order of a millimeter. Inverse tapers, decreasing the waveguide width at the end accomplish low loss coupling by expanding the waveguide mode to match the fiber mode size. Almeida suggested a micrometer- long nanotaper coupler that converts both the mode size and the effective index of the waveguide to that of the optical fiber [5]. The nanotaper is fabricated in the silicon device layer of a SOI wafer and is butt coupled to the optical fiber. In this coupling scheme, the optical fiber has to be no more than a few micrometer away from the tapered waveguide end. However, it is difficult to fix the optical fiber in the optimum position for the best coupling, without any support structure. Therefore, we etched v-grooves into our chip to be able to actively align and securely attach the optical fibers in the optimum optical coupling position with UV curable epoxy. Since the mode size and effective index of the taper strongly depend on the surroundings of the nanotaper we decided to use an overhanging silicon nanotaper in air, avoiding the possibility of increased losses into the nearby substrate. Simulations were performed to maximize the mode size and effective index overlap and therefore the coupling efficiency. All simulations were performed at λ = 1.55 µm. As input mode reference, a Gaussian beam with the diameter of a single mode optical fiber was used. The waveguide core material was Si (refractive index of silicon used: $n_{Si} = 3.48$). The waveguide height and width were taken as h = 260 nm and $w_w = 500$ nm, respectively. The transmission (S₂₁) has been calculated for TE- and TM- like modes. A parametric sweep has been performed to find the optimal taper tip width, which gives the best optical transmission and can still be fabricated within the presented process without more than 5 % deviation from the ideal geometry. Fig. 2 shows the results for S₂₁ as a function of taper tip width. The maximum transmission occurs at a taper tip width of around ≈ 100 nm, which is also a reasonable size for the electron beam lithography used to define the waveguide taper. The taper nominal length is ≈ 50 µm.

Separating photonic and mechanical layers affords flexibility in the design of the mechanical parts to suit specific sensing applications. We designed cantilever structures, torsional structures, and membranes, on chip structures, and overhanging structures, as well as various types of actuation mechanisms. The membrane structures are designed to have a resonance frequency ranging from \approx 70 kHz up to \approx 1 MHz - an SEM of these sensors is embedded into the setup in Figure 3. The cantilevers are designed to combine small size with high resonance frequencies, with a range between ≈ 50 kHz and ≈ 4 MHz. The integration of an actuator increases the range of possible applications. The builtin static actuation gives the possibility of tuning the transducer gain and measurement range. This is accomplished by changing the static gap size between the mechanical structure and the optical cavity. We decided to develop designs for two actuations schemes, bimorph and electrostatic actuation. Bimorph actuators deliver fast responses and large force. However, the introduction of metal on the mechanical structure creates significant internal losses and therefore reduces the mechanical quality factor drastically. In contrast, electrostatic fringe field actuation doesn't need any metal in contact with the mechanical member, which lets the mechanical member freely oscillate and doesn't affect the mechanical quality factor. However, the maximum forces, as indicated by simulation, are rather small, but still suitable for on-resonance excitation for frequency sensing. Detailed actuator performance is a subject of a future study.



Figure 2: Simulation result of the transmission (S21) as a function of taper tip width. The inverse taper length is 50 μ m.



Figure 3: Schematic of the detection setup with an embedded SEM of the membrane transducer and a cross section schematic for this transducer (dashed box).

DEVICE FABRICATION

The main challenge is to fabricate these diverse optical and mechanical structures in a unified batch fabrication process and a single platform, which can be tailored for specific applications. In the following we will present the process using the overhanging cantilever probe as an example.

The fabrication for the cavity optical transducer is based on double side polished SOI 100 mm (4") wafers with a ≈ 260 nm top silicon layer, with low doping for good optical properties, and a $\approx 2 \,\mu m$ buried oxide (BOX) layer. The process flow is summarized in Figure 4. In the first step, the waveguide taper, waveguide, and micro disk are defined via electron beam lithography and inductively-coupled reactive ion etching down to the BOX layer. The nominal width of the waveguide is ≈ 500 nm and the gap between the waveguide and the disc is defined to be \approx 340 nm. The waveguide is linearly tapered down to a width of 100 nm over the distance of \approx 50 µm at both waveguide ends for low loss coupling to/from optical fibers (Fig. 4 b). The remaining structures are defined by i-line stepper optical lithography unless otherwise noted. A sacrificial silicon oxide layer ($\approx 1 \text{ } \mu\text{m}$) is deposited and defined to create a window to the Si substrate for the later KOH etching as well as a hole in the center of the micro disk, which is used to anchor the micro disk to the bulk silicon with the following SiN layer. The silicon dioxide is thinned down by a CF₄ plasma etch through a lithographically-defined window in photoresist in the region above the micro disk to ensure good optomechanical coupling (Fig. 4 c, d). A low-stress silicon nitride layer (≈ 400 nm) deposited in a low-pressure chemical-vapor deposition furnace acts as a passivation layer in the waveguide region and as structural material for the mechanical structure. Following nitride deposition, a gold layer is deposited and defined in a liftoff process to create a micro heater, electrical connection, and wire bond pads. (For the electrostatically actuated transducer, the micro heater is replaced by electrodes for fringe field actuation). The SiN layer is lithographically patterned (Fig. 4 e), and dry etched to form the SiN cantilever, SiN ring above the micro disk, and SiN anchor to mechanically attach the micro disk to the bulk silicon. The previously defined metal layer is used as a hard mask for SiN, to self-align the SiN structure in critical areas (Fig. 4 e). For front side protection during the later KOH etch, a hafnium oxide (HfO) layer of ≈ 20 nm is deposited via atomic layer deposition.



Figure 4: Representation of the process flow for the transducer with integrated thermal actuation and overhanging tip. The image in the top left shows the whole device. The dashed red in indicates the path for the cross sectional views (a).

In the following, a reactive ion etch (RIE) is used to open up a window in the HfO and SiN for anisotropic etching of the silicon, to form v-grooves for optical fibers. A back to front aligned backside lithography followed by RIE etching is used to form an anisotropic etch window on the backside as well. Both lithographies for the definition of the front and back side etch window for anisotropic etching are defined with contact aligner lithography. During the following anisotropic silicon etch, vgrooves are formed on the front side of the chip and the shape of the cantilever chip is defined by etching through the handle wafer from the backside (Fig. 4 e). (Another approach is the replacement of the backside KOH etch with a ICP etch to create a backside trench with vertical sidewalls. This approach has been used to develop acceleration sensors with large seismic masses made from the handle wafer.) Silicon dioxide layers and HfO are removed by 49 % HF wet etching to undercut and release the movable structures as well as the micro disk, which is anchored to the bulk silicon with a SiN anchor. A critical point drying process is used to avoid stiction between the parts due to capillary forces (Fig. 4 f). At the end of each v-groove the overhanging waveguide inverse tapers are suspended between silicon support structures and coupled to optical fibers, which are placed in the v-groove, actively aligned and glued into place with ultraviolet (UV) light curable epoxy.

MEASUREMENT SETUP

The detection setup used to characterize the device is shown in Figure 3. Light from a tunable laser (1520 nm to 1570 nm) is sent through a polarization controller and coupled into the fiber pigtailed device, allowing for polarization adjustment to maximize the light coupling to the desired micro disk optical mode before recording data. The injected light circulates hundreds or thousands of times (depending on the cavity's finesse) before exiting through the outgoing optical fiber. The output of the fiber is analyzed with a photodetector and either the transmission spectrum of the device is recorded by sweeping the laser wavelength, revealing the spectral location and spectral width of the cavity's optical modes, or modulation of the transmitted intensity as a function of the mechanical motion of the cantilever is measured by fixing the laser wavelength on the shoulder of an optical cavity mode. Motion of the cantilever results in a frequency modulation of the optical cavity modes, which can be translated into an intensity modulation by probing these modes on the side of their resonance minima. The information obtained from the transmission spectra is thus used to determine the laser wavelength for optimal transduction sensitivity. The output signal is intensity-modulated in proportion to the mechanical motion, and is transduced by a photodetector before being sent to an electronic spectrum analyzer to reveal the spectrum of mechanical modes (Fig.5). In electrical actuation experiments a network analyzer is used to measure the device transfer function - a ratio of the optical modulation to the driving force – as a function of the drive frequency.

RESULTS AND DISCUSSION

Figure 5, shows the measured thermal mechanical noise spectral density of a nitride membrane device held at four corners, similar to the one in Figure 3. A clear peak in the noise spectrum occurs at ≈ 668.1 kHz, in good agreement with a finite- element model with a nitride with tensile stress of about ≈ 150 MPa. A high mechanical quality factor of better than 110 000 is evident from the data.

A TE optical mode with an optical quality factor of $\approx 800\ 000$ was used to carry out this measurement at a very low optical power level of approximately $3.16\ \mu$ W (-25 dBm) excitation power, 830 nW (-30.8 dBm) at the sensor and 219 nW (-36.6 dBm) at the photodetector, accounting for an estimated 5.8 dB fiber pigtail coupling losses at each facet. Despite the low power, the signal to noise ratio on resonance is approximately 20 dB. Low power was chosen deliberately to demonstrate the

sensing performance, while also preventing the optical forces from shifting the frequency of the narrow mechanical resonance, an optical spring effect clearly observed at higher optical powers.



Figure 5: Measured mechanical frequency noise spectrum of the membrane transducer. The dotted green line indicates the background noise level. Signal power is reported relative to 1 mW.



Figure 6: Measured mechanical frequency noise spectrum of the cantilever transducer with Lorentzian fit. The dotted green line indicates the background noise level. Signal power is reported relative to 1 mW.

Figure 6, shows the measured thermal mechanical noise power spectral density of a torsional cantilever transducer. The red line shows a Lorentzian fit of the power spectral density in cantilever displacement, calibrated using the equipartition method [6]. The background corresponds to the measurement noise of 9 fm/ $\sqrt{Hz} \pm 0.5$ fm/ \sqrt{Hz} . The uncertainty is one standard deviation. The statistical uncertainty derived from the measurement is small. The main uncertainty is propagated form the Young's modulus used to calculate the spring constant for the displacement sensitivity calculation with the equipartition theorem. The estimated variation of the Young's modulus originating from deposition conditions is about 10 %.

We measured mechanical quality factors between $\approx 50,000$ and $\approx 500\ 000$ for the low-stress silicon nitride membrane transducers and ≈ 50 to $\approx 2\ 000$ for the cantilever transducers which include the metallization for bimorph actuators.

Static and dynamic electrothermal actuation has been characterized. First, the static displacement of the silicon nitride

structure was characterized as a function of voltage with a white light interferometer (Figure 7). The dashed red line shows a 2^{nd} order polynomial fit to indicate the expected displacement/actuation voltage relation.



Figure 7: Relative cantilever displacement with applied DC voltage. The statistical uncertainty based on repeated measurements is smaller than the data markers.



Figure 8: Normalized gain of the optomechanical displacement sensor is decreased by static actuation. The one standard deviation uncertainty based on fitting network analyzer spectrum data is smaller than the data marker size. Inset: Optomechanical signal power as a function of frequency for 8 mV AC and 50 mV DC. Signal power is reported relative to 1 mW (inset).

We then optomechanically measured the dynamic response of the structure to actuation. A small fixed modulation (AC) voltage, swept between 300 kHz and 10 MHz, was added to the actuator static bias (DC) voltage, resulting in a small, known mechanical modulation of the gap. The laser wavelength has been fixed at the steepest slope on the side of the optical resonance line at each applied DC voltage. A network analyzer was used to provide the AC voltage and detect the resulting optical power modulation, which is proportional to the motion amplitude and the optomechanical coupling. A typical optomechanically measured device transfer function is shown on the Figure 8 inset.

The AC bimorph output is proportional to the product of AC and DC voltages, as the actuation force is quadratic in applied voltage. To illustrate the tuning of the optomechanical gain by the actuator, we first normalized the displacement spectra by the DC voltage to account for the stronger drive with larger DC voltage. The resulting normalized displacement transfer function (Figure 8) reveals the gain decreasing with increasing bias as the actuator increases the transducer gap and decreases the gain of the optomechanical sensor.

The results show that the small actuation here is capable of tuning the gain by more than 10 %.

SUMMARY AND CONCLUSIONS

In summary, we have presented an overview of the design, micro- and nanofabrication, and characterization of a novel type of fully-integrated cavity optomechanical transducer platform for measurement of physical quantities by transducing them to mechanical motion. The approach of full silicon integration of all nanophotonic components with mechanically separated highquality-factor movable components creates the opportunity to independently tailor optical, mechanical, and the actuation parts for a variety of MEMS and NEMS sensing applications that require high precision, high bandwidth, and small footprint. Additional benefits of the photonic readout approach are low power dissipation at the sensor, insensitivity to electromagnetic interference and robustness of fiber-connectorized devices. We demonstrate high mechanical and optical performance of platform components by optically detecting thermomechanical fluctuations and actuated motion of mechanical devices while tuning the optical readout gain by electrical actuation.

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RESONANT PIEZOELECTRIC CANTILEVERS AS NANO-PRECISION DISPLACEMENT PROBES

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ABSTRACT

This work presents a new class of highly sensitive displacement probes with sub-nm displacement resolution using a piezoelectric resonator as a frequency output strain gauge. The device is comprised of a length extensional bulk mode thin film piezoelectric on silicon (TPoS) resonator coupled to a V-shaped micro-cantilever. In this manner deflection of the cantilever tip is transferred to the resonating plate causing a change in its resonance frequency. Operating at 8.4MHz, fabricated sensors show sensitivities as high as 1.5 Hz/nm with a potential displacement resolution of 0.5Å. The high sensitivity and fast response time along with a wide range of linearity make such probes suitable for different dynamic measurements in the sub-nm to a few microns range. Such devices can be used in compact fully electronic (non-optical) atomic force microscopes (AFM) or high resolution surface profilometry.

INTRODUCTION

Atomic force microscopes (AFMs) have proven to be invaluable tools in different state of the art micro and nanotechnology research areas with applications in high resolution surface imaging, high density data storage, biosensing and nanolithography [1-4]. At the heart of an AFM is a micro-cantilever that deflects as it scans a surface, typically due to atomic forces, and monitoring of its deflections can provide a high-resolution 3dimentional image of the surface. Different approaches have been sought to measure cantilever tip deflection. Current modulation of a tunneling tip was the first method initially used within the first AFMs to measure the cantilever deflections resulting from atomic forces on the surface of interest [1]. This method was capable of measuring forces in the 10⁻¹⁸N range with displacement resolution of ~1Å. A few years later, optical levers were introduced providing unrivaled measurement resolution in the sub-Å range [5]. Although the optical lever is still the most common approach in commercially available scanning probe microscopes, complexity and bulkiness remain its main challenges, especially when measurements in adverse environments, such as low temperature or ultra-high vacuum conditions, are needed.

Electrically readable integrated force probes with nanometer precision are of great interest as potential replacements for bulky and complex optical levers. Chip-scale MEMS force probes could provide such powerful solutions with limited to no compromise in performance. Embedding piezoresistive strain sensors within microcantilevers is among popular techniques showing sub-nm resolution [2], [6]. Piezoelectric detection is another method explored to measure induced stress along the cantilever due to deflection [7]. However both of such displacement/force measurements happen at DC which necessitates low bandwidth measurement in order to reduce interference of low frequency noise. This constraint in turn limits the response time of the probe. Moreover such techniques require a high precision A/D conversion circuit to measure minuscule voltage or current changes resulting from the week transduction mechanisms. In addition to above-mentioned disadvantages, a piezoresistive strain gauge requires a relatively high DC bias which also causes higher power dissipation and consequently temperature instability [2], [6].

On the contrary, frequency-modulated output at higher frequencies provides a faster response, and is less vulnerable to noise, particularly low frequency noise of the DC measurements. Furthermore, frequency measurements can be performed directly by a digital frequency counter without the need for A/D conversion.

Micro-fabricated capacitive and piezoelectric displacement sensors for frequency output tapping mode microscopy have been reported [8], [9]. Although resolving issues of previous amplitude measurements, such methods require operation in vacuum to avoid performance deterioration due to air damping while resonating at flexural resonance mode. Moreover, capacitive detection needs a small gap and relatively high bias voltage to achieve high displacement sensitivity.

Thin film piezoelectric-on-substrate (TPoS) resonators have been used for a wide range of sensory applications such as high resolution mass sensing [10], and pressure and temperature measurement [11], [12]. Such lateral extensional mode resonant devices can be used in low power self-sustain oscillating circuits providing highly stable frequency sources without operation in vacuum. In this work, for the first time, a TPoS resonator has been coupled to a cantilever. Bending of the cantilever deforms the resonator changing its resonance frequency. Such design can lead to a high throughput displacement probe addressing deficiencies of prior microscale detection mechanisms. Piezoelectric transduction can also bypass temperature stability issues resulting from selfheating (due to the required DC bias) of piezoresistive readout.

OPERATION PRINCIPLES

The proposed displacement probe consists of a rectangular BAW resonator with two triangular cantilever extensions on both ends. The resonator is used as a frequency output strain gauge operating in its first extensional resonance mode as illustrated in Figure 1(a). A piezoelectric transducer was used in order to actuate such resonance, which is made of a 1 μ m thick AlN layer sandwiched between two platinum electrodes.



Figure 1: (a) 8.4MHz first length extensional resonance mode of the resonator coupled with V-shaped cantilevers, (b) static mechanical stress on the cantilever and the resonator resulting from a static force applied to the cantilever tip.

Figure 1(b) shows the stress on the cantilever and the resonator resulting from a static force applied to the tip of the V-shaped cantilever. The mechanical stress modulates the resonance frequency of the resonator. V-shaped cantilevers are added on both

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sides and at the corners of the resonator to maintain symmetry for the resonant structure.

A typical AFM cantilever has a spring constant (K) in the range of 0.01-100 N/m. Depending on the type of microscopy, there is a trade-off between force and displacement sensitivity; a stiffer probe provides a higher displacement sensitivity, while a softer probe provides better force sensitivity. However, low stiffness probes are required for contact AFM to avoid surface damage. Moreover, fabricating the cantilever and resonator from the same silicon device layer, an additional compromise exists between quality factor (Q) of the resonator and stiffness of the cantilever in the design of the proposed structure. A thicker silicon substrate leads to a higher resonator Q, which improves the frequency stability of the TPoS resonator when engaged in an oscillating circuit configuration. In contrast, stiffness is proportional the substrate thickness cubed. Probe stiffness can also be reduced by increasing the length of the cantilever instead of reducing the thickness. Nevertheless, a longer cantilever can potentially disturb the operation of the resonator, and reduce the effective stress resulting from deflection on the resonator, thus decreasing the displacement sensitivity.

On the other hand, the performance of a resonator can be evaluated based on its motional resistance (R_m) [13]. In order to engage the resonator in a single stage self-sustained oscillating circuit, R_m needs to be minimized. The electrode area on the resonator is inversely proportional to the motional resistance. The length of the resonator which determines the resonance frequency is not easily adjustable, and therefore the lateral surface area only can be controlled by the resonator width. Increasing the width of the resonator increases the probe stiffness while decreasing the motional resistance simultaneously. Therefore, a trade-off between the spring constant of the probe and the motional resistance of the resonator should be addressed with a proper selection of lateral surface area.

The structure was fabricated on a 5μ m silicon device layer. Such thickness has been chosen in order to provide a fair performance of the structure as a force/displacement probe and reliable frequency source component at the same time. The stiffness of the triangular cantilever alone with the length of 330μ m was found to be 16.8 N/m based on the static force simulation results. After coupling with the resonator, overall probe stiffness was found to be 9.2 N/m. The resonator horizontal dimensions are chosen to be $420 \times 315 \mu$ m² leading to first lateral resonance mode frequency of 8.4MHz and motional resistance for the resonator that is low enough for operation in a single stage oscillation circuit.

EXPERIMENTAL AND METHOD

Fabrication Process

The resonant piezoelectric probes were fabricated on a SOI substrate with 5µm thick <100> oriented low resistivity device layer. Figure 2 shows the fabrication process flow. Bottom electrodes patterns were first formed on the substrate with negative nLOF2020 photoresist. A 150nm of Pt layer forming the bottom electrodes was then sputtered and lifted-off. A 1µm AlN piezoelectric layer was then deposited (Figure 2(a)) and patterned to provide electrical accesses to the bottom electrode. AlN etch was performed in two steps: a chlorine-based ICP etch to remove the main bulk of the 2µm thick layer followed by a wet-etch step in 2.5% TMAH to remove the remainder of the AlN layer. Similar to the bottom metal electrodes, top platinum electrodes (150nm thick) were formed via sputtering and lift-off (Figure 2(b)). Later a 1.5µm layer of low temperature LPCVD oxide was deposited to be used as a hard mask for the upcoming silicon etch step and to protect the



Figure 2: Fabrication process of resonant piezoelectric cantilevers.

AlN layer during etching and cleaning steps. An 800nm gold layer was deposited and lifted-off on wire-bond pads after etching the oxide layer on the pad areas (Figure 2(c)). After etching the oxide hard mask layer with plasma, silicon was etched via DRIE to form the resonator and cantilever out of the device layer (Figure 2(d)). Finally, to suspend the whole structure, silicon handle-layer and BOX layer were etched via silicon DRIE and fluorine based ICP respectively from backside (Figure 2(f)).

The SEM view of fabricated probe is illustrated in Figure 3.The cantilever arms are merely made of the same silicon device layer. Furthermore, the top platinum layer is divided into actuation and sensing electrodes allowing two-port operation of the resonator.



Figure 3: SEM image of a fabricated resonant piezoelectric cantilever comprised of a first length extensional mode thin film piezoelectric-on-substrate (TPoS) resonator with triangular cantilever extensions.



Characterization

In-situ SEM characterization of the displacement sensors was performed inside a scanning electron microscope (SEM) using a Zyvex S100 Nano-manipulator as shown in Figure 4(a). Input, output and ground electrical connections from network analyzer to the device inside the SEM chamber were provided using three nanomanipulator probes while a forth probe was used to deflect the cantilever. The working distance (WD) of the SEM electromagnetic lens was used to measure the displacement at the cantilever tip (Fig. 3b). The WDs were read at high magnification to maximize reading accuracy. Figure 5 demonstrates the measured frequency responses of the resonator under different cantilever tip displacements. The resonator has a resonance frequency of 8.4MHz with a quality factor of ~900 and motional resistance of $3k\Omega$. Frequency shifts resulting from four different displacements at the free end of the probe are evident in acquired frequency responses. Resonance peaks suggest that deformation resulting from higher deflection of the cantilever slightly reduces the peak amplitude and quality factor due to deformation of the resonator. Device characterization does not show a noticeable change in Q resonator in air and under vacuum.



Figure 5: The resonance frequency of the device changes in response to vertical displacement of cantilever tip.

Figure 6 graphs the measured resonator frequency shifts versus probe tip deflection showing a linear response with sensitivity of ~1.5 Hz/nm for a relatively wide 30μ m range of displacements. The minimum detectable displacement (MDD) of the displacement probe is limited with frequency stability of the resonator. Engaging such resonator in a self-sustained oscillator circuit leads to $10^{-8} df/f_0$ Allan deviation in a 1 minute measurement period with sampling rate of 10Hz. Such frequency stability is associated with 0.08Hz translating to a potential displacement resolution as small as 0.5Å for the fabricated probe. Considering Cantilever stiffness of 9.2N/m obtained from COMSOL finite element analysis a force resolution of 400pN has been achieve for such structure with a 5 μ m thick silicon device layer. Such pico-range force detection limit demonstrates a resolution force sensitivity of the probe along with its high resolution displacement sensitivity.



Figure 6: Resonance frequency shift vs. displacement at the end of cantilever

COMSOL analysis shows that frequency shift is not a function of device/cantilever thickness and therefore the device can be thinned down to increase force sensitivity by orders of magnitude while maintaining the same displacement sensitivity.

Different microscale displacement detection techniques as AFM probes are summarized in Table 1. The proposed displacement probe shows the minimum detectable displacement (MDD) in the same range compared to other approaches. However the current detection approaches do not provide versatile and robust schemes in microscale implementation of AFM probes.

CONCLUSION

A thin film piezoelectric-on-insulator (TPoS) MEMS resonator, vibrating at its first length extensional mode, was used as a frequency output strain gauge coupled with triangular cantilevers. Such structure was used as a displacement sensor showing linear sensitivity of 1.5 Hz/nm over a wide range of cantilever tip

Table 1: MEMS displacement probes comparison

	Detection Mechanism/ Output	MDD^+	Operation Mode	Disadvantages
[2]	Piezoresistive Output/ Voltage Amplitude	0.5Å in 10Hz-1kHz BW	DC measurement	Low frequency noises – Thermal instability – Speed limit
[7]	Piezoelectric Output/ Current Amplitude	0.1-0.3Å in 125Hz BW	DC measurement	Low frequency noise sources – Speed limit
[8]	Capacitive Sensing/ Frequency	0.5Å (2Hz freq. stability)	Flexural resonance mode – 66.5kHz	Small gap – Large DC bias – Air Damping
[9]	Piezoelectric Output/Frequency	1Å (2Hz freq. stability)	Flexural resonance mode – 95kHz	Air Damping – Stiff cantilever
This work	Resonant TPoS/ Frequency	0.5Å (Freq. stability of 10 ⁻² ppm)	Resonance at 1st length extensional - 8.4MHz	

+ MDD: Minimum Detectable Displacement

deflections. The frequency stability of the resonator within the proposed probe demonstrates a potential minimum detectable deflection (MDD) of 0.5Å. This detection limit is comparable with its state of the art counterparts without putting limits on scanning speed of the probe. Such frequency output displacement probes can also address problems associated with some of the previously demonstrated microscale detection mechanisms such as vulnerability to low frequency noise sources, temperature instability and vacuum operation.

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A CONTACTLESS ELECTROCHEMICAL IMPEDANCE MEASUREMENT METHOD

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ABSTRACT

We present the first demonstration of a passive, contactless method to interrogate electrochemical impedance (EI) via a microfabricated MEMS coil. EI measurement was achieved using the principle of reflected impedance between a pair of inductively coupled coils. Ionic concentration and electrolyte temperature were wirelessly transduced from measurement electrodes connected to a microfabricated coil in a flexible thin film substrate. This technique is widely applicable for resistive transduction and obviates the need for soldering discrete electronic components, microcontrollers, batteries, and bulky hermetic packaging. It also features low power consumption (~75 μ W) and all biocompatible construction (only Au, Ti, and Parylene C), making it ideal for use in harsh or *in vivo* environments.

INTRODUCTION

Chronic recording of physiological signals via the use of implanted sensors has been a long-standing goal for the medical and scientific community. Such sensors would enable continuous monitoring to facilitate greater understanding of disease progression as well as inform course of treatment.

The *in vivo* environment poses a formidable challenge for the development of telemetry circuitry used to encode and wirelessly transmit measured signals [1]. Water vapor and ion permeation create undesired paths for electric current and can alter critical bias voltages. The inflammatory response to foreign bodies can subject the implant to a highly oxidative or acidic environment [2]. Blood flow and other involuntary movements can subject the implant to chronic and repeated mechanical strain. Such events can damage sensitive components or introduce errors in signal measurement and transmission.

Naturally, the presence of the implant should also minimize harm to the host environment. The materials selected for the packaged device should be nontoxic and induce minimal immune response. The use of soft, flexible materials (i.e. those mechanically matching tissue properties) and elimination of sharp corners also serves to minimize damage to tissue [3]. Power requirements for electronics should be kept in check to minimize heating to safe levels.

In this work, our goal is to address these requirements with a new method to measure and transmit signals within a simulated *in vivo* environment. Passive wireless capacitive transduction methods have been widely studied and utilized, with notable examples demonstrating successful chronic implantation in humans [4]. However, for resistive transduction, wireless transmission has been limited to the use of active circuitry (analog or digital via microcontrollers). This burdens system design by adding power constraints, thermal budget concerns, and bulk to the overall package. Here, we introduce the use of reflected impedance across a pair of inductively coupled coils to achieve wireless resistive transduction without active circuitry and demonstrated its use for measurement of electrolyte conductivity and temperature.

THEORETICAL CONSIDERATIONS

A pair of inductively coupled coils shares the properties of a transformer. An alternating current source induces a varying

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Figure 1: Schematic of reflected impedance principle, where Z_p represents the complex impedance of the primary circuit resistance and capacitance, L_p and L_s the inductances of coupled coils, and Z_s the complex impedance of the load on the secondary circuit. An AC source (LCR meter) induces current within the secondary circuit across a pair of inductively coupled coils (coupling coefficient M).

To simplify, an equivalent single loop model that lumps the secondary circuit into a complex impedance term may be utilized (Figure 2). Due to conservation of energy, a load that is attached to the secondary coil will result in an apparent load on the primary side, as represented in (1). In this manner, changes in the secondary load may be reflected across the inductively coupled coils and measured as changes on the primary side [5].



Figure 2: Equivalent single loop primary circuit with reflected impedance represented as Z_R .

$$Z_R = \frac{\omega^2 M^2}{Z_s + j\omega L_s} \tag{1}$$

DESIGN

Size of the secondary coil was maximized to optimize coupling with the primary coil. Coupling can also be improved by increasing the number of turns in order to capture increased magnetic flux. However, this can result in a large and unwieldy device footprint for implant applications. Additional improvement in coupling can be achieved through the use of a ferrite core on the primary coil.

All of the materials selected for use have a proven track record for use within the *in vivo* environment. Thin film gold was used for the electrodes due to its inertness and its well-characterized electrochemical properties [6]. Electrode size and spacing were

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based on previous investigations on electrochemical impedance sensing [7]. The low resistivity of gold also serves advantageous for its use as a coil in RF applications. Parylene C was selected as the substrate and to encapsulate the electrodes and coil, due to its excellent insulation properties as well as its amenability to microfabrication processes [8].

METHODS

The fabrication of this thin film polymer device was based on previously reported methods [7]. A pair of Au electrodes (2000 Å thick on 200 Å Ti adhesion layer, $300 \ \mu m \times 300 \ \mu m$, $500 \ \mu m - 5000 \ \mu m$ electrode-to-electrode spacing) attached to a planar coil (30 mm diameter, 1-16 turns) was patterned and electron beam evaporated onto a flexible Parylene C substrate (10 \mu m thick) (Figure 3). Following deposition of another 10 \mu m Parylene insulation layer, electrodes were exposed via O₂ plasma reactive ion etching (Technics RIE, 200 W, 150 mTorr) [9].



Figure 3: Overview of fabrication process. (i) Deposition of $10 \ \mu m$ thick Parylene C substrate. (ii) Patterning and deposition of Ti adhesion layer and Au. (iii) Deposition of $10 \ \mu m$ Parylene insulation layer, reactive ion etching (O₂) to expose electrode sites, release, and cutout from silicon substrate.

After cutout and release (Figure 4), the devices were placed in a polystyrene Petri dish and submerged in phosphate buffered saline (PBS) of various concentrations $(0.5 - 10 \times, 8-150 \text{ mS/cm [10]})$ and temperatures (22°C - 65°C) to mimic a range of typical and abnormal *in vivo* conditions. Deionized water was used to establish a baseline impedance reference.



Figure 4: (Left) Photograph of microfabricated and released thin film Parylene C device held by forceps. Sixteen turn coil shown; one, five, and six turn coils were also fabricated. (Right) Inset of left; micrograph highlighting electrochemical impedance (EI) measurement electrodes ($300 \times 300 \mu m$ exposed area).

The secondary circuit comprises of the Au planar coil ($L = 11.1 \mu$ H, $Q \sim 1.5$ at 2.198 MHz) and electrodes submerged in electrolyte, which can be represented by the Randles circuit model (Figure 5). At resonant frequency, the reactance of the capacitive (C_{dl}) and inductive (thin film coil) elements will be equal in magnitude but opposite in sign. The resulting reflected impedance consists of the remaining resistive element, which is the solution resistance (R_s). The Petri dish (0.8 mm thick base) was placed onto and concentrically aligned with the commercially available primary coil

having a ferrite core (Figure 6, 24 μ H, Q = 180@125 kHz, Wurth Electronics, 50 mm diameter, 22 turns).



Figure 5: Secondary circuit schematic, which comprises of insulated Au coil and Randles circuit model of electrodes submersed in electrolyte.



Figure 6: Primary coil with ferrite core to improve coupling with thin film (secondary) coil.

The primary coil was elevated (onto an additional Petri dish to prevent inadvertent coupling with the benchtop) and attached to an LCR meter (Hewlett Packard E4285A) (Figure 7), which provided the AC signal and recorded the reflected impedance.



Figure 7: Device under test (DUT) was placed within a Petri dish and submerged in PBS (phosphate buffered saline). Primary coil includes ferrite core to improve inductive coupling with thin film (secondary) coil. Not drawn to scale.

The optimal measurement frequency was determined to be 2.198 MHz (Q = 34.3, minimum phase, 1 V_{P-P}) to bypass the double layer capacitance (C_{dl}) in the Randles circuit (Figure 8) [11]. At this measurement frequency, the impedance is dominated by the solution resistance. Reproducibility of coil position was achieved by observing phase angle measurement and adjusting alignment until minimum phase ($\leq \pm 1^{\circ}$) was attained. To assess misalignment performance, coil centers were translated up to 5 mm from initial alignment while measuring reflected impedance.



Figure 8: Frequency response of system comprising of the primary coil coupled with the DUT. The excitation signal on the LCR meter was set to 2.198 MHz (minimum system phase to bypass double layer capacitance C_{dl}) for subsequent testing.

RESULTS

Reflected electrochemical impedance was measured from fabricated devices and utilized to transduce ionic concentration and temperature of the electrolyte into which the devices were submerged (Figure 9). The observed power draw at the primary coil was approximately 75 μ W.



Figure 9: Reflected impedance was observed to be inversely proportional to ionic concentration. Deionized water was used as a baseline reference and indicated using the arrow.

Electrochemical impedance of ionic solutions has previously been shown with a direct wired measurement to be inversely proportional to temperature [12]. This relation was measured wirelessly using this reflected impedance method with $1 \times PBS$ across a range of different temperatures (Figure 10).



Figure 10: Electrochemical impedance of $1 \times PBS$ was observed to be inversely proportional to temperature.

Reflected impedance increased linearly with coil misalignment by approximately 1% per mm of coil center offset. The results were achieved with a 5 turn thin film coil, and preliminary results suggest that altering coil diameter and increasing turn count can reduce misalignment effects on baseline impedance measurement.



Figure 11: Reflected impedance increased with coil misalignment across the range tested, but remained within 5% of baseline measurement.

CONCLUSION

A method to wirelessly measure electrochemical impedance was developed and demonstrated by measuring ionic conductivity (0-150 mS/cm) and solution temperature (22-65°C). The method utilizes inductively coupled coils and requires no active electronic components. Measurement frequency was determined to be 2.198 MHz and performance with suboptimal coil alignment (translation up to 5mm) was evaluated. Featuring all biocompatible material construction, flexible polymer substrate, and low power draw (<100 μ W), the device is well suited for the wet, *in vivo* environment. The overall structure may also lend itself well to use in other harsh environments.

The electrochemical impedance transduction principle utilized here was previously demonstrated in a variety of sensing applications such as sensing of catheter blockage [13], liquid flow measurement [12], contact force [7], and drug delivery volume [10, 14], but with direct wired measurements. We demonstrated that it may be feasible to incorporate a coil and adjust electrode packaging to enable wireless subcutaneous sensing of these aforementioned signals. In addition, coating the electrode surface with antigens [15] or chemically sensitive polymers [16] can add specificity to the electrochemical measurement.

Additional development is planned to refine this method for implant applications. Miniaturization of coil size and further characterization of inductive coupling (foveation, distance, increasing Q factor) will be performed. Performance will be assessed in an *ex vivo* application to measure catheter obstruction.

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A FLUSH-MOUNT SENSOR PACKAGE FOR A MEMS PIEZOELECTRIC MICROPHONE WITH THROUGH-SILICON–VIAS FOR AIRCRAFT FUSELAGE ARRAYS

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ABSTRACT

This paper presents the design, fabrication and preliminary testing of a flush-mount sensor die and associated package for an aluminum nitride (AIN) based piezoelectric MEMS microphone for aircraft fuselage arrays. The optimal microphone design is determined using composite plate theory in combination with lumped element modeling. Through-silicon-vias are incorporated into the fabrication of the sensor thus eliminating front-side wire bonds and enabling an overall flush surface for the packaged sensor that minimizes flow disturbance.

Frequency response measurements show a comparable sensitivity (13.1 μ V/Pa) and significantly higher resonant frequency (194 kHz) than prior piezoelectric aeroacoustic microphones. The developed packaging method for the sensor demonstrates an overall flushness to within 10 μ m, showing substantial improvement from any previously reported efforts. This is the first truly flush-mount piezoelectric MEMS microphone with associated packaging that has been developed.

INTRODUCTION

In their effort to locate, understand and mitigate the impact of noise sources on an aircraft, aeroacoustic researchers are in need of a high performance, low cost microphone to address the increasing noise restrictions on commercial aircraft. Existing commercial sensors, even with their relatively high cost, in some cases constrain the quality and type of measurement that may be achieved.

Previously developed MEMS aeroacoustic microphones [1-10] have failed to address the need for a sensor that can be packaged and installed with a hydraulically smooth front surface to be used for boundary layer measurements in a fuselage array. Protrusions from the surface must be within the viscous sublayer, or smaller than 5 viscous wall units, in order for the flow to see an essentially flat surface [11]. The surface topography of the sensor and associated package that utilizes wire bonds alone is locally limited to ~130 μ m [2], and a required protective encapsulant adds to the surface roughness. Additionally, through-silicon via (TSV) transducer implementation has largely been limited to under-bump metallization attachment techniques [12,13] where sensor flushness is not a priority.

This work addresses the limitations of existing MEMS piezoelectric microphones used in aeroacoustic applications by incorporating through-silicon-vias (TSVs) into the fabrication to eliminate the use of front-side wire bonds that affect the flow field and create an overall flush-mount microphone package. Requirements for this microphone include a low noise floor of < 48 dB SPL, a flat frequency response function over the audio range from 20 Hz- 20 kHz, a linear response up to 172 dB, and the packaged sensor flushness to be within 12.5 μ m.

DESIGN

Structure

The microphone structure includes a circular diaphragm composed of the device layer of a silicon-on-insulator (SOI) wafer, with an annular ring composed of a AlN and molybdenum (Mo)

978-1-940470-02-3/HH2016/\$25©2016TRF DOI 10.31438/trf.hh2016.36 film stack as shown in Figure 1. It also contains a passivation layer to repel moisture, and is front-side vented. The diaphragm displaces under an incident acoustic pressure into the cavity on the backside of the wafer and the resultant strain in the piezoelectric material produces a proportional output voltage across the electrodes; this is the microphone output.



Figure 1: Photograph of an AlN piezoelectric microphone die with TSVs, die size is 2 mm x 2 mm (top) and cross-sectional view of the microphone structure (bottom).

Modeling and Optimization

The goal of the microphone design is to maximize its operation range in terms of pressure and frequency while meeting the demands of the particular application. The main competing requirements are to achieve a high maximum linear pressure, P_{max} , of 172 dB while achieving the lowest possible minimum detectable pressure, *MDP*. This is accomplished through the use of the lumped element modeling technique [14] in combination with a piezocomposite diaphragm model [1]. Both are then integrated into a single-objective optimization algorithm that places constraints on the bandwidth and P_{max} , with the objective to minimize *MDP*, where the design variables include the piezoelectric film stack geometry and diaphragm dimensions. Fabrication constraints are also included. The optimization yields a 6.8 µm thick diaphragm with an outer diameter of 744 µm, a 93 µm wide piezoelectric film stack and an AlN layer thickness of 1 µm.

FABRICATION

Fabrication of the microphones is broken down into three phases, and is outlined in Figure 2. Phase I includes the fabrication of the initial SOI substrate with TSVs and the deposition of the piezoelectric film stack, both of which are performed at external foundries, Icemos Technology Ltd and OEM Group respectively. Phase II is the front-side processing done at the Nanoscale Research Facility (NRF) at the University of Florida. Phase III is the back-side processing steps performed at both NRF and the Lurie Nanofabrication Facility (LNF) at the University of

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Michigan.

Phase I begins with the formation of TSVs on a SOI substrate using an etch, isolation and deposition process [12]. The formation of the TSVs includes the growth of a 1 μ m layer of thermal silicon dioxide passivation layer between the substrate and the doped poly-silicon that forms the TSV. When this is done, oxide is also formed on the front and back surfaces of the wafer. The front layer is retained in diaphragm composition (Figure 2a). Thereafter, the Mo/AIN/Mo piezoelectric film stack is deposited (Figure 2b), and the wafers are ready for Phase II processing, which defines the piezoelectric transducer.

Phase II begins by defining the piezoelectric annular ring. A thin layer of silicon dioxide is deposited, and then a small feature is defined to provide electrical isolation between the two electrodes, and prevent an electrical short when the front side metal contacts are made. After etching a ground strap to the device layer and performing a metal liftoff to connect the electrodes to the TSVs (Figure 2c), a hydrophobic silicon nitride layer is deposited to protect the sensor from moisture, and the front side vent opening is patterned. Contact windows are etched through the nitride layer to expose the metal pads over the TSVs, and allow for front-side probing (Figure 2d).

Phase III first consists of etching a handle layer ground strap, then a back side metallization step to form contact pads to the TSVs. A winding vent channel is defined (Figure 2e) and finally, a deep reactive ion etch process is used to form the cavity and vent opening to the front side (Figure 2f). The resulting sensors are then diced into a final size of 2 mm x 2 mm using a laser dicing method.



Figure 2: Schematic view of the process steps to create an AlN piezoelectric microphone with TSVs.

PACKAGING

The final sensor package is comprised of two main components, the endcap assembly and the laboratory test assembly, necessary for the calibration of the sensors.

The "endcap" assembly consists of the sensor die, a circular printed circuit board (PCB) substrate and a circular metal shim cap that sets the sensor flushness as shown in Figure 3. The sensor and shim cap are attached to the PCB using an epoxy film. This film seals the back cavity by encompassing the edges of the die without clogging the vent structure, which prevents unwanted acoustic leakage paths and thereby controls the low frequency response of the sensor with the combination of the electrical properties of the piezoelectric film and the chosen cavity and vent geometries. The electrical connections to the endcap are recessed into the PCB within the perimeter of the die for protection.

The process for attaching the sensor to the PCB is shown in Figure 4. First, the PCB is prepared by planarizing the front and back surfaces. During the PCB manufacture, the gold-plated through holes, or vias, protrude above the surface and must be polished down to create a smooth surface. Next, a pocket is milled on the back side of the board over the vias to create a recess and holes are milled through the PCB to access the metal pads on the back side of the sensor. The epoxy film is then cut to the desired dimensions using laser machining with a cutout for the die in the center and an overall diameter slightly smaller than the shim cap. The shim cap thickness is chosen such that the combined height of the shim cap and epoxy film is \sim 25 um thicker than the sensor.



Figure 3: Cross-sectional view of the endcap assembly for a flushmount sensor package.

It is at this point that the sensor and shim cap are placed on a layer of heat release tape to protect the diaphragm. The epoxy film is placed on the PCB. A flip chip bonder is then used to align and place the sensor/shim cap combination on the PCB. Pressure is applied during this process to seat the sensor on the PCB surface. During this process, the shim cap pushes down on the epoxy film, encasing the sensor around its perimeter. The chuck of the flip chip bonder ensures that the shim cap and sensor top surface remain at the same height and establish a hydraulically smooth front surface. Once heat cured, silver epoxy is used to fill the drilled holes on the PCB and connect the back side contacts of the sensor to the vias on the endcap.



Figure 4: Assembly steps for a flush-mount sensor package.

In order to facilitate testing, the endcap assembly is then mounted onto a larger fixture (the laboratory test assembly) to provide structural support and electrical connections (Figure 5). The endcap assembly is secured to the top of a brass tube which houses an additional PCB with a buffer amplifier and filter capacitors. The amplifier board is interfaced to the endcap assembly with solder connections. Wire connections from the amplifier board for amplifier power and output emerge from the back of the brass tube and are stress relieved using heat shrink tubing. Finally, an outer nylon sleeve is used for electrical isolation and to ensure mounting flushness in the test fixture.



Figure 5: The fully assembled laboratory test package showing the endcap assembly, brass tube and interface circuitry (left); and a photograph of the assembled device (right).

EXPERIMENTAL SETUP AND RESULTS

This section describes the preliminary experimental characterization of the microphone as well as the package. Measurements of the surface roughness of the endcap assembly, resonant frequency and sensitivity are presented.

Surface Roughness

Once packaged, the surface roughness of selected device is quantified using a Bruker Contour GT-1 scanning white light interferometer (SWLI). Multiple measurements are stitched together to form a complete scan of the entire endcap assembly and are shown in Figure 6. An overall flushness of ~10 μ m is achieved with a sensor to shim cap step height of ~8 μ m.

Resonant Frequency

The voltage chirp excitation response of the microphone is measured using a Polytec scanning laser vibrometer system. The pre-packaged measured resonant frequency of 194 kHz compares favorably with the predicted value of 181 kHz when accounting for fabrication uncertainties. The resonant frequency also provides a qualitative measure of the bandwidth of the device in lieu of an explicit high frequency acoustic characterization method that will be conducted at a later date. The post-packaged measured resonant frequency of 237 kHz indicates a significant amount of thermal stress is imparted to the sensor during the packaging process, which indicates a reduction in achievable sensor sensitivity. An epoxy film specifically designed for substrates with mismatched coefficients of thermal expansion has since been found, and will be tried at a later date to assess its mitigation on the impact of thermal stresses incurred during the packaging process.

Sensitivity

The frequency response of the device under test (DUT) is determined through comparison with a measurement-grade reference microphone in an acoustic plane wave tube (PWT). A PWT is a rigid duct that allows for the propagation of acoustic plane waves, such that microphones mounted at the same lengthwise location along the duct will see the same pressure, provided that the drive frequency is below the cutoff frequency, f_c . This frequency is dependent upon the isentropic speed of sound of the medium and the cross section of the duct.

The experimental setup can be seen in Figure 7, where the DUT and the reference, a Bruel and Kjaer 4138 1/8-in pressure field microphone, are mounted at the end of the PWT. An Agilent 33220A Function Generator in combination with a National Instruments PXI Data Acquisition system is used to generate the test signal and acquire the data. The pseudo-random noise test signal is sent through a Crown XLS 1500 amplifier before reaching

a BMS 4590 compression driver. Measurements are conducted over a 6.4 kHz bandwidth using a 1 Hz bin width and a center frequency of 3.5 kHz. The maximum frequency of 6.7 kHz is the cutoff frequency for the PWT, and the lower limit of 300 Hz is dictated by the poor response of the speaker below that frequency.

The frequency response measurement for the selected microphone is shown in Figure 8 in terms of magnitude and relative phase. The magnitude is flat to within 1.5 dB and the sensitivity taken at 1 kHz is $13.1 \,\mu$ V/Pa (-97.6 dB ref 1 V/Pa).



Figure 6: SWLI measurement of assembled endcap structure; Top view (top), X-profile (middle) and Y-profile (bottom).



Figure 7: Frequency response measurement experimental setup.

Thermal Cycling Analysis

The sensor endcap assembly integrity with regards to thermal cycling is evaluated in order to assess the reliability of the package. The mismatch in coefficients of thermal expansion (CTE) between the PCB and sensor generate high stresses and strains at the attachment point when rigidly connected, such as with a solder

joint [15].

Thermal stability is evaluated by cycling the microphone package from room temperature to 125°C, down to -55°C and back to room temperature multiple times in an Espec ESX-3CA environmental chamber. The frequency response is then measured and compared to pre thermal cycling measurements. The applied thermal cycling profile and resulting measurements are shown in Figure 9. The sensitivity of the device taken at 1 kHz after thermal cycling was 12.6 μ V/Pa, and was within 4% of the pre-cycling measurement.



Figure 8: Measured frequency response of the microphone.



Figure 9: Applied thermal cycling profile (top) and measured sensitivity of the microphone before and after thermal cycling (bottom).

CONCLUSIONS

A flush-mount piezoelectric microphone and associated package for aircraft fuselage array measurements has been developed. TSVs were formulated into the fabrication to eliminate the use of front side wire bonds. The packaged sensor demonstrates an overall flushness to within 10 μ m, showing an order of magnitude improvement from recently reported efforts in similar endeavors [2]. Preliminary characterization shows a comparable sensitivity (13.1 μ V/Pa) and significantly higher resonant frequency (194 kHz) than the most recent piezoelectric aeroacoustic microphone [1]. This piezoelectric microphone and associated package is the first to exhibit a truly flush front surface.

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A NOVEL METHOD FOR MEASURING RING-DOWN IN A HIGH NOISE AND HIGH FREQUENCY SYSTEM USING LASER DOPPLER VIBROMETRY

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ABSTRACT

This paper reports on a novel method for measuring the ring-down of high-frequency MEMS devices in a high-noise environment using laser Doppler vibrometry (LDV). This method was validated using a 220 MHz aluminum nitride (AlN) piezoelectric contourmode resonator, but could be applied to many other high frequency devices where low displacement magnitudes and excessive measurement noise makes traditional ring-down methods impossible. Even when noise dominated the desired signal and with out-of-plane displacements decaying from 120 pm down to only 4 pm, a ring-down time constant was extracted which was within 5.25% of the electrical measurements allowing for precise measurements of quality factor.

INTRODUCTION

A key figure of merit for resonator performance is the quality factor, *Q*. This is an inverse measure of the energy dissipation per cycle of the resonator. High quality factors are necessary in resonators to minimize power consumption and oscillator phase noise. Standard LDV ring-down measurements used in determining the quality factor of a resonator are performed by applying a step function input to the device and measuring the exponential decay in the time domain [1,2]. This is suitable for large displacements (>500 pm) but below this, thermal noise as well as the inherent noise in the LDV results in an unacceptably low signal to noise ratio. Figure 1 shows a ring-down measurement of a 220 MHz resonator in the time domain with no signal averaging applied. The exponential decay characteristic of a ring-down is indiscernible due to the noise level.



Figure 1: A ring-down measurement captured in the time-domain. The drive signal was cut off at $5\mu s$, but because of inherent LDV system noise, the exponential decay is not visible.

To extract a usable signal from this level of measurement noise, repeated measurements must be taken and averaged together. This is not possible at high frequencies due to a 40ns triggering error between the LDV and the function generator. At lower frequencies this small error is negligible, but at 220MHz it is an order of magnitude larger than the period of a single wavelength (4.5ns). Figure 2 shows a simulation of a 220MHz ring-down that has been averaged ten times in the presence of a 40ns trigger error. The result is a severe degradation in the desired signal making ring-down measurements impossible. For time domain measurements it may be necessary to apply as many as 10,000 averages to obtain a clean, low noise signal.

By taking multiple, short time sample measurements and averaging in the frequency domain, substantial averaging can be employed and measurements can be made all the way to the noise floor of the vibrometer (4pm) even when the time signal is dominated by noise.



Figure 2: A simulation of a ring down (in blue) that has been averaged ten times in the presence of a trigger error of 40ns (in red). Averaging causes the magnitude of the transient, exponential decay to decrease by more than an order of magnitude as a result of the variation in its position within each time sample.

THEORY

The LDV takes a short, discrete time sample of a small portion of the ring-down event and performs a fast Fourier transform (FFT) to extract the magnitude and phase of the resonant frequency of the resonator. This is repeated over and over and the results are averaged in order to suppress the noise. There are two methods for averaging that are most commonly used in vibrometry measurements. Magnitude averaging simply averages the displacement and phase values independently, whereas complex averaging combines the two into a phasor and averages the real and imaginary portions of that phasor independently,

$$S = \frac{1}{N} \left(\sum_{n=1}^{N} Re(S_n) + i \cdot \sum_{n=1}^{N} Im(S_n) \right)$$

Where N is the number of averages and S_n represents the phasor. Complex averaging is the preferred method because it is much more

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effective at suppressing Brownian motion and other incoherent noise. In the frequency domain the 40 ns trigger error becomes negligible since it is two orders of magnitude smaller than the 8μ s sample time used in the FFT.

To measure the decaying amplitude of a ring-down profile the timing between when the measurement starts and when the drive signal is cut off is delayed. Figure 3 shows an example of three such delay points. The start of the time sample is progressively delayed with respect to drive signal cutoff. The magnitude of the resonant frequency for each sample is then used to reconstruct the ring-down. For the example shown in FFigure 3 a sample time of 8µs was used and the time step between each sample was 10µs. In practice, the step between each time sample can be considerably smaller than the sample time so that each measurement overlaps the previous one. This maximizes the resolution of the decaying signal. At each of these delay points as much complex averaging can be applied as necessary to suppress unwanted noise.



Figure 3: Short sample times are captured (red area) and an FFT is taken to determine the average magnitude of the sample. The delay between the start of measurement and the cut off of the drive signal is increased so that the entire ring-down event is captured in multiple discrete steps.

EXPERIMENTAL RESULTS

The UHF-120 Ultra High Frequency Vibrometer from Polytec Inc. was used in conjunction with a Rhode&Schwarz SMBV-100A function generator [5,6]. Data from the vibrometer is collected via a LeCroy WavePro 725Zi where it is digitized and then transferred to a PC for post processing. To simultaneously trigger the vibrometer measurement and cut off the drive frequency, an HP33120A function generator produced a square wave at 1kHz. Electrical measurements were taken using a Rhode&Schwarz ZVL network analyzer which were used to determine the devices quality factor and resonant frequency (figure 4).

Six devices were used for validation of our measurement approach. They were 220MHz AlN piezoelectric contour-mode resonators (CMR) similar to the one shown in Figure [3,4]. They are composed of a 1 μ m aluminum nitride device layer sandwiched between a lower 100nm aluminum electrode layer and a 100nm platinum upper electrode layer. The electrode layout and the size of the body of these devices was identical but their designs varied by the length and width of the supporting anchors.



Figure 4: The schematic of the LDV measurement system. A second function generator is used to trigger the vibrometer measurement and the cut off of the drive signal simultaneously. Data is collected on the LeCroy WavePro 725Zi oscilloscope where it is fast-digitized and transferred to a PC for processing.



Figure 5: One of the 220MHz AlN contour-mode resonators used for testing. The body of the resonator is $137\mu m \times 57\mu m$. Depending on drive level, the out-of-plane displacement has a steady-state peak magnitude between 120 and 255pm. At these displacements the drive signal is masked in thermal noise. Measurements were taken at the center of the resonator (marked with an X).

The operating frequency of the resonator is set by the electrode spacing and is governed by,

$$f_0 = \frac{1}{2W} \sqrt{\frac{E_{eq}}{\rho_{eq}}}$$

Where W is the electrode spacing, and E_{eq} and ρ_{eq} are the effective Young's modulus and density respectively of all three layers of the device. The devices were driven at -6dBm and at resonance. The quality factors were extracted through electrical measurements from the VNA and varied between 1161 and 3155. At low power levels the maximum out-of-plane motion of some of the devices was as low as ~120pm at resonance. Figure 4 shows the experimental measurements from a single point at the center of the body of device #1 with a sample time of 8µS and a time step between each sample of 1µS. The sampling rate was 2GHz.

Device Number	<i>f</i> ₀ [MHz]	Anchor Length [µm]	Anchor Length [µm]	Initial Displacement [pm]	Electrical Q	Τ [μS]	LDV Measured Q	% Error
1	220.38	7.5	8	125.20	1610	2.40	1664	3.35
2	220.58	22	15	155.60	1161	1.59	1100	5.25
3	220.53	21	15	125.40	1575	2.38	1650	4.76
4	220.75	11	15	219.40	2207	3.32	2300	4.21
5	220.78	40	15	255.30	3155	4.50	3121	1.08
6	220.81	21	17	225.60	2944	4.18	2900	1.49

Table 1: Six devices were tested using this ring-down method. They had identical electrode configurations but varied in terms of the width and length of their anchors. The resonant frequency and the electrically and LDV measured Quality factors are shown in addition to the error in the LDV measurement as compared with that from the vector network analyzer. There is a maximum of 5.25% error in the LDV measurement.



Figure 5: The resulting experimental profile is due to the fact that each point is a convolution of a portion of the ring-down event. To extract the time constant of the exponential decay, a ring-down profile is simulated using known parameters such as the resonant frequency, steady-state magnitude, drive signal cutoff time and sample time. A measurement process identical to the experimental method is then applied to the simulated data and the time constant is then varied until a good fit is obtained.

At each time delay point, the signal was averaged one hundred times. The experimental data does not follow the expected exponential decay of a traditional ring-down. This is due to the fact that each data point is the average magnitude of the specific frequency over the entire 8µS sample time. If the sample window includes steady-state oscillation as well as a portion of the ringdown event (e.g. the center FFT in Figure 3) the magnitude is a convolution of the steady-state and the ring-down profile. The most efficient way to determine the ring-down time constant is to produce a simulated waveform that includes a steady state and ring-down event and perform a simulated measurement with identical parameters to the experimental measurement. The frequency, steady-state magnitude, drive signal cutoff time and sample time are all known a priori. The time-constant for the exponential decay can be varied until there is a good fit between the experimental and simulated data. Error! Reference source not found. shows the match between the experimental and simulated data from the single spatial point at the center of the

device. For this measurement the extracted time constant τ is 2.4 μ S. To find the quality factor we use:

 $Q = f_0 \pi \tau$

This gave a quality factor of 1664 which matches the electrically measured Q of 1610 to within 3.35%. Note that the simulated data accurately matches the experimental data down to 4pm which is the noise floor of the UHF-120. Furthermore, the ring-down curve is unaffected but the noise floor until it decays to 4pm so the time constant is unaffected by this characteristic of the vibrometer. This measurement method is capable of extracting ring-down measurements from these contour-mode resonators even when there is very little out-of-plane motion. Table 1 above shows the results from the measurements of the six resonators tested. The Q varies by a maximum of 5.25% from the electrically measured Q. Device number two has a ring down time constant of only 1.59 μ S.

CONCLUSION

The ring-down time constant and therefore the quality factor were measured using laser Doppler vibrometry in an environment of high noise and very low displacement values with an accuracy of less than 6%. While contour-mode piezoelectric resonators were used for this paper, this method could be applied to any bulk-acoustic or flexural device that has a measurable out-of-plane motion component. Frequencies up to the 1.2 GHz limit of the UHF-120 could be measured as well, by using smaller time samples and smaller delay steps. In addition to quality factor, this measurement method could be used to determine the presence of non-linearities in the device and it could potentially be used to measure the onset of parametric resonance at previously unachievable frequencies [7].

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ATOMIC LAYER DEPOSITED ETCH STOP LAYERS FOR HYDROFLUORIC ACID

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ABSTRACT

We report on the evaluation of the etch rate in hydrofluoric acid (HF) for various atomic layer deposited (ALD) thin films. Specifically, the intent is to characterize several films as potential etch stop layers as HF is often used as a release etch for various sacrificial films but most commonly SiO₂. Traditionally in MEMS, SiN has been used as an HF etch stop, and we show that plasma enhanced ALD (PEALD) of SiN at 350°C can achieve similar etch rates to SiN from low pressure chemical vapor deposition (LPCVD) at 850°C. Further, the etch rate of PEALD SiN is drastically reduced when the film contains ~10% AlN as a dopant. Additionally, several metal oxide films are evaluated for their etch characteristics as deposited and as a function of post-deposition anneal temperature. Some of the evaluated metal oxide films showed greater HF etch resistance than PEALD SiN after anneals of moderate temperature (~400°C).

INTRODUCTION

Hydrofluoric acid based release etches of sacrificial silicon oxide layers remains one of the most widely utilized processes in MEMS fabrication. Hydrofluoric acid resistant layers providing an etch stop, enable a wide variety of fabrication processes and device design freedom. Recently, the development of HF etch resistant SiN deposited at low temperature (< 400°C) has been driven by the need for improved insulating spacer layers in advanced CMOS fabrication [1]. The films should be conformal, pinhole free at thicknesses of only a few nm, and of comparable etch rate in dilute aqueous HF to low pressure chemical vapor deposition (LPCVD) SiN. These same properties are of interest to MEMS researchers to provide a robust etch stop layer for HF release etch of sacrificial SiO₂. In this paper we present multiple HF etch resistant thin films deposited by plasma enabled atomic layer deposition (PEALD). The demonstrated films include SiN with incorporation of AlN, HfO₂, ZrO₂, TiO₂, and Al₂O₃.

Films deposited through purely thermal atomic layer deposition as well as PEALD are each explored. Additionally, the effect of post-deposition anneals were evaluated by a treating the ALD thin films rapid thermal anneals (RTA) at various temperatures.

EXPERIMENTAL PROCEDURE

Single crystal (100) silicon wafers were utilized as the starting substrate for all depositions. Substrates were cleaned by the RCA process immediately before deposition. The films deposited for this study were performed in a Fiji F202 (Cambridge Nanotech/Ultratech) hot wall reactor equipped with a remote inductively coupled plasma (ICP) generator which was operated at 300 W at 13.56 MHz at 350°C.

Samples were cleaned by the RCA method and then immediately introduced into the ALD reactor for deposition. After deposition, film thickness was measured with spectroscopic ellipsometry (Woollam M2000). If a post deposition anneal was utilized, it was performed on a piece of the deposited wafer loaded on a carrier wafer. Then the film thickness was re-measured to determine any film thickness change from the anneal process.

For the etch, the samples were etched in aqueous HF (different concentrations were used for different films based on

their relative etch rates). The etch was performed at room temperature in a freshly poured HF bath. After a timed length of etch, the samples were removed and immediately rinsed with DI water. Samples were dried in an N_2 gun, and finally, the film thickness was measured once more to determine the change in thickness and etch rate for each sample.

SILICON NITRIDE

Silicon nitride was deposited by PEALD at 350°C utilizing tris[dimethylamino]silane (3DMAS, Sigma Aldrich) as a precursor and N₂ plasma at 300W. A strong correlation between the density of the SiN film and the HF etch rate was discovered [2]. Two techniques for increased film density and thus low etch rate were identified. One is the duration of N2 plasma exposure during each deposition cycle, and the second is a post-deposition H₂ plasma anneal also performed in the Fiji 202 reactor without breaking vacuum after the PEALD process. Utilizing these techniques a pure SiN film from PEALD could achieve an etch rate of 6.1 ± 1.0 Å/min in 100:1 dilute HF. This is not statistically significantly different from the etch results attained from a LPCVD sample deposited at 850°C. This result shows that PEALD is a route to high quality SiN HF etch stop layers at temperatures significantly lower than possible LPCVD and on par with PECVD, while surpassing the etch performance typical of PECVD SiN films. Figure 1 shows the etch rates for LPCVD, PECVD, and PEALD SiN films deposited at the Stanford Nanofabrication Facility for this study.



Figure 1: Wet etch rate in 100:1 dilute HF for silicon nitride films deposited by various methods. Note that PEALD at 350°C can achieve WER similar to LPCVD at 850°C.

We found that the etch rate can be further reduced by the incorporation of AlN into the PEALD SiN. This introduction of controlled levels of AlN into SiN is straightforward to achieve because of the cyclic, layer-by-layer nature of ALD. Cycles of SiN PEALD were intermixed with cycles of AlN from trimethylaluminum (TMA, Sigma Aldrich) and N₂ plasma during the deposition. Figure 2 shows the relationship between incorporated AlN and the etch rate of the film in 100:1 dilute HF. For films with more than 12% Al content as measure by X-Ray photoemission spectroscopy, the etch rate is negligible, verifying

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an optimal etch stop. This level of AlN incorporation relates to roughly 1 cycle of AlN for every 19 cycles of SiN.



Figure 2: Wet etch rate in 100:1 dilute HF for intermixed films of SiN and AlN deposited by PEALD at 350°C as a function of Al concentration.

ALUMINUM OXIDE

Aluminum oxide (Al₂O₃) films were prepared by thermal ALD utilizing alternating precursor pulses of TMA and DI H₂O and by PEALD utilizing alternating precursors pulses of TMA and O₂ plasma at 300W. Deposition was performed at 200°C and the etch was performed in 20:1 BOE for 20 sec. Figure 3 contains the etch rate as a function of post-deposition anneal for both thermal and plasma ALD of Al₂O₃. The rapid thermal anneals were all performed for a duration of 4 minutes with a 20 second ramp time under N₂ atmosphere. While the etch rate of the PEALD sample remains relatively constant under the various anneals, the thermal ALD film has a transition to lower etch rate occurring around 700°C.

From the thickness measurements before and after annealing, we can see change in the thermal films at roughly the same temperature, while the plasma films remain more consistent in thickness change (Figure 4). This provides an indication for film densification within that temperature range which would contribute to the lowered etch rate. This will ultimately need to be established as a definite film densification through a more direct test of film density such as X-Ray reflection refractometry or nanoindentation.



Figure 3: Etch rate of thermal ALD and PEALD Al_2O_3 as a function of post-deposition anneal temperature. The anneals were 4 min in duration and the etch was performed in 20:1 BOE.



Figure 4: Change in film thickness from the post-deposition anneal for thermal ALD and PEALD of Al₂O₃. A decrease in thickness is an indication of film densification.

HAFNIUM OXIDE

Hafnium oxide (HfO₂) films were prepared by thermal ALD precursor pulses alternating utilizing of tris[dimethylamido]hafnium (TDMA-Hf) and DI H2O and by PEALD utilizing alternating precursors pulses of TDMA-Hf and O2 plasma at 300W. Deposition was performed at 200°C and the etch was performed in 20:1 BOE for 600 sec (thermal ALD) and 180 sec (PEALD). Figure 5 contains the etch rate as a function of post-deposition anneal for both thermal and plasma ALD of HfO2. The rapid thermal anneals were all performed for a duration of 4 minutes with a 20 second ramp time under N₂ atmosphere. For HfO₂, the etch rates are significantly lower than Al₂O₃ or SiN and are negligible above 400°C for thermal ALD and above 600°C for PEALD.

The film thickness change under anneal again shows an indication of densification at the approximate temperatures at which the etch rate drops significantly (Figure 6). However, unlike in Al_2O_3 , the thickness change is not monotonic with anneal temperature indicating a more complex film response.



Figure 5: Etch rate of thermal ALD and PEALD HfO_2 as a function of post-deposition anneal temperature. The anneals were 4 min in duration and the etch was performed in 20:1 BOE.



Figure 6: Change in film thickness from the post-deposition anneal for thermal ALD and PEALD of HfO₂.

TITANIUM OXIDE

Titanium oxide (TiO₂) films were prepared by thermal ALD pulses precursor utilizing alternating of tris[dimethylamido]titanium (TDMA-Ti) and DI H2O and by PEALD utilizing alternating precursors pulses of TDMA-Ti and O2 plasma at 300W. Deposition was performed at 200°C and the etch was performed in 20:1 BOE for 180 sec (thermal ALD) and 600 sec (PEALD). Figure 7 contains the etch rate as a function of postdeposition anneal for both thermal and plasma ALD of TiO2. The rapid thermal anneals were all performed for a duration of 4 minutes with a 20 second ramp time under N2 atmosphere. Titanium oxide has comparable etch rates to HfO₂ but achieves a negligible etch rate at even lower anneal temperatures.

The film thickness change under anneal again shows an indication of densification at the approximate temperatures at which the etch rate drops significantly (Figure 8), but again shows a more complex relationship to anneal than Al₂O₃.



Figure 7: Etch rate of thermal ALD and PEALD TiO_2 as a function of post-deposition anneal temperature. The anneals were 4 min in duration and the etch was performed in 20:1 BOE.



Figure 8: Change in film thickness from the post-deposition anneal for thermal ALD and PEALD of TiO₂.

ZIRCONIUM OXIDE

Zirconium oxide (ZrO₂) films were prepared by thermal ALD utilizing alternating precursor pulses of tris[dimethylamido]zirconium (TDMA-Zr) and DI H₂O and by PEALD utilizing alternating precursors pulses of TDMA-Zr and O₂ plasma at 300W. Deposition was performed at 200°C and the etch was performed in 20:1 BOE for 300 sec. Figure 9 contains the etch rate as a function of post-deposition anneal for both thermal and plasma ALD of ZrO2. The rapid thermal anneals were all performed for a duration of 4 minutes with a 20 second ramp time under N2 atmosphere. Zirconium oxide has comparable etch rates to HfO₂ but achieves a negligible etch rate at even lower anneal temperatures.

The film thickness change under anneal again shows an indication of densification at the approximate temperatures at which the etch rate drops significantly (Figure 10), but again shows a more complex relationship to anneal than Al₂O₃.



Figure 9: Etch rate of thermal ALD and PEALD ZrO_2 as a function of post-deposition anneal temperature. The anneals were 4 min in duration and the etch was performed in 20:1 BOE.



Figure 10: Change in film thickness from the post-deposition anneal for thermal ALD and PEALD of ZrO₂.

SILICON OXIDE

As a comparison we measured etch rates of PEALD SiO2 as well this is the most likely sacrificial material which might utilize the etch stops demonstrated. Silicon oxide (SiO₂) films were prepared by PEALD utilizing alternating precursor pulses of tris[dimethylamino]silane (3DMAS) and O₂ plasma at 300W. Deposition was performed at 200°C and the etch was performed in 50:1 Hf for 10 sec. Figure 11 contains the etch rate as a function of post-deposition anneal for PEALD of SiO₂. Note that a lower concentration of HF is utilized than in the other oxide films. The rapid thermal anneals were all performed for a duration of 4 minutes with a 20 second ramp time under N₂ atmosphere. Silicon oxide has etch rates several orders of magnitude over HfO₂, TiO₂, or ZrO₂, and significantly higher than Al₂O₃ and SiN.

The film thickness change for SiO_2 shows a monotonic thickness decrease with increasing anneal temperature matching the monotonic decrease in etch rate (Figure 12).



Figure 11: Etch rate of PEALD SiO_2 as a function of postdeposition anneal temperature. The anneals were 4 min in duration and the etch was performed in 50:1 HF.



Figure 12: Change in film thickness from the post-deposition anneal for PEALD of SiO₂.

VAPOR HF ETCHES

All of the above characterization has been performed with aqueous HF mixtures. However, it is becoming increasing common to perform vapor phase releases of MEMS and NEMS devices. The chemical role of water in the etching mechanisms for these films should not be overlooked. Testing Al₂O₃ and HfO₂ PEALD films in an anhydrous HF etch system (Primaxx μ Etch System), we found negligible etch rates for each film as denosited at 200°C. This extreme selectivity in vapor etches als importance of ALD metal oxide films as HF et

SUMMARY

Evaluation several thermal ALD and PEALD films for HF etch resistance shows several candidates for etch stops or etch barriers. SiN doped with AlN, ZrO₂, TiO₂, and thermal HfO₂ can all achieve negligible etch rates in aqueous HF with a total thermal budget of deposition and anneal of < 400°C. This low temperature process capability allows compatibility with a wide array of materials and post-CMOS fabrication. Further the precise thickness control, highly conformal, and pinhole free nature of ALD means these films integrate well with the unique topologies of MEMS processes.

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ATOMIC LAYER ETCHING OF SUSPENDED ULTRA-THIN STRUCTURES

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ABSTRACT

We performed the first-ever demonstration of atomic layer etching (ALE) on suspended nano-devices, which were fabricated using an atomic layer deposition (ALD) on polyimide process [1]. Two thermal ALE chemistries including $Sn(acac)_2/HF$ and TMA/HF, were used to remove Al_2O_3 from suspended microbridges and microbolometer-type absorption structures [2], [3]. Measurement of the thermal time constants of several microbridges after ALE was used to evaluate finite Al_2O_3 material removal. ALE is currently limited to transistor processing, but will have substantial influence on a wide variety of microdevices. Here, we demonstrate ALE's functionality for the fabrication of ultrathin suspended structures.

KEYWORDS

Atomic layer etching, thin film, atomic layer deposition, nano-fabrication, micropulse calorimetry

INTRODUCTION

ALE has been identified as one of the leading low damage process technologies for future transistor development and will offer unique opportunities for nano- and micro-device fabrication [4]. As device thicknesses continue to shrink to several nanometers, precise etching processes for successful device fabrication will be required. Additionally, the stringent demands of precise tolerances for 3-dimensional finFET devices require extremely precise, low-damage etching processes. Current plasma processing approaches expose devices to large particle fluxes causing damage to surfaces. While there are currently no records of ALE for micro and nano electro-mechanical systems (N/MEMS) it is expected to permeate the field with unique utility for specific fabrication processes.

Similar to ALD, thermal ALE consists of self-limiting, conformal, and atomically precise layer by layer removal of material through thermodynamically favorable vapor phase reaction cycles. However, while in principle, ALE can be thought about as ALD in reverse, it is not a simple reversal of the chemical reactions. Different precursors must be used in the removal of material. The majority of ALE to date (non-thermal) has used ionenhancement or energetic neutral atom beam enhanced surface reactions together with halogenation of the surface to etch the material [5]-[8]. These techniques may provide anisotropic etching for given applications. Recently, thermal ALE has been demonstrated based on spontaneous thermal chemistries. For example, etching of Al₂O₃ may be achieved by an initial precursor fluorinating the surface to form a metal fluoride layer. The second metal precursor can then accept fluorine from the metal fluoride and donate a ligand to the metal in the metal fluoride to form volatile species through a ligand-exchange process. For example, the removal of Al₂O₃ is achieved using tin(II) acetylacetonate $(Sn(acac)_2)$ and HF as the reactants [2]. The overall reaction is:



Figure 1. General schematic of Al_2O_3 ALE surface chemistry using $Sn(acac)_2$ and HF [9]. (Step A) The surface of Al_2O_3 is converted to an AlF₃ surface layer through a fluorination reaction. (Step B) The metal fluoride layer exchanges ligands with the $Sn(acac)_2$ producing volatile species which removes the AlF₃ surface layer.

$$Al_2O_3 + 6 HF + 6 Sn(acac)_2 \rightarrow$$

$$2Al(acac)_3 + 6SnF(acac) + 3 H_2O \qquad (1)$$

A schematic showing the individual $Sn(acac)_2$ and HF reactions is given in Figure 1. The metal fluoride ligand exchange mechanism for etching may be extended to other chemistries as well, including trimethylaluminum (TMA) and HF [3].

ALD technologies have provided an extreme utility by offering conformal coatings for 3-dimensional structures. For example, underside coating of suspended MEMS cantilevers provided superior anti-stiction coatings [10]. The ability to remove material by single atomic layers conformally on 3-dimensional structures will also offer extreme control in many etching processes for N/MEMS. As an example, using ALE for microbolometer fabrication would allow the removal of excess mass, reducing the total heat capacity and improving sensitivity to absorbed radiation. Recently, an ALD microbolometer-type absorption structure was fabricated using an ALD on polyimide technique [1]. The structure consists of a thin ALD W layer surrounded by thin ALD Al2O3 layers, which can be thinned using ALE, if necessary. It should be noted that such a process is not limited to the development of microbolometer-type structures, but may be applied in any case where it is advantageous to remove redundant material post-release or from complex 3-dimensional structures. In the coming years more ALE chemistries will be explored for etching of diverse materials. Thermal ALE will no doubt permeate many micro- and nano-fabrication processes due to its self-limiting, conformal and atomically precise nature.

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Figure 2. Ultra-thin ALD test structures [1], [11]. a) Microbridge with Al contact pads for thermal time constant measurements preand post-ALE. b) Microbolometer-type absorption structure made up of $Al_2O_3/W/Al_2O_3$ as fabricated pre-ALE.

FABRICATION

The fabrication of suspended microbridge test structures (Figure 2a) for ALE demonstration followed a similar process as outlined by Eigenfeld et al. [1]. A sacrificial polyimide layer was spun onto a bare silicon wafer to a thickness of $\sim 3 \ \mu m$ and cured. Next, ALD layers were deposited using a Beneq TFS 200 ALD reactor. The layers were deposited at 300 °C in a trilayer configuration consisting of 6.15 nm Al₂O₃/ 3.7 nm Ru/ 6.15 nm Al₂O₃ The Al₂O₃ was deposited using TMA and H₂O precursors for growth at ~0.13 nm/cycle. The Ru was deposited using thermally activated Ru(EtCp)₂ at 110 °C and O₂ for growth at ~0.04 nm/cycle The ALD layers were then patterned using a positive photoresist mask and a CF₄/O₂ reactive ion etch chemistry. Next, using a negative photoresist lift off process, Al was evaporated to form contact pads at the anchors of the microbridges. Finally, an O₂ plasma ashing process was used to remove the sacrificial polyimide layer and release the microbridges. The microbridges are on the order of $20 - 120 \mu m$ in length and 2 - 4um in width.

Microbolometer-type absorption structures (Figure 2b) were fabricated in a similar process as described above with the absence of Al contact pads. The layers were produced in a custom ALD W reactor and consisted of a nominal $Al_2O_3/W/Al_2O_3$ trilayer, which produces flat suspended absorption structures as previously demonstrated by Eigenfeld *et al.* [1]. The Al_2O_3 deposition was performed at 130 °C for growth at ~ 0.13 nm/cycle. The W deposition was performed at 130 °C for growth at ~ 0.38 nm/cycle.



Figure 3. X-ray reflectivity (XRR) data for thermal ALE of Al_2O_3 using $Sn(acac)_2/HF$ and TMA/HF chemistries. Differences in the periodicity for pre- and post-ALE correspond to varied X-ray interference due to changes in the film thickness. a) The $Sn(acac)_2/HF$ chemistry corresponded to ~ 0.022 nm/cycle etch rate. b) The TMA/HF chemistry at 300 °C corresponded to ~ 0.055 - 0.065 nm/cycle etch rate depending on placement in chamber.

METHODS

Two thermal ALE chemistries have been found to etch Al₂O₃ using a fluorination and ligand exchange mechanism. As demonstrated by Lee et al., Sn(acac)₂/HF and TMA/HF etch Al₂O₃ in a self-limiting process [2], [3]. Figure 3 demonstrates X-ray reflectivity data from Al₂O₃ coated Si test wafers confirming etch rates reported from Lee et al. for each chemistry in a custom built ALD/ALE reactor. Etching using Sn(acac)₂/HF was performed at 200 °C with an etch rate of 0.022 nm/cycle. Etching using TMA/HF was performed at 300 °C with an etch rate of 0.055 – 0.065 nm/cycle depending on wafer placement in the chamber. Special attention was paid to precursor dose pressure and purge times to ensure surface saturation and self-limited behavior. Etch rates are subject to change with varied reactor parameters. For example, the Sn(acac)₂/HF etch rate was increased to 0.046 nm/cycle by varying dose pressures.

Mircopulse calorimetry was used to measure material removal on suspended microbridge test structures. This method was previously used to measure the specific heat capacity of ALD W, as outlined by Eigenfeld *et al.* [12]. The thermal time constants, τ 's, of the microbridge test structures are measured before and after ALE sessions. A pulsed step voltage is applied across the bridge (with a rise time < 1 µs) and a transient current response is monitored, which is several orders of magnitude slower than the voltage rise. This effect is a result of the temperature sensitive resistance responding to joule heating of the Ru and surrounding Al₂O₃ layers. An exponential decay function may be fit to the measured current response to extract τ for a given microbridge length. The experimental τ data can then be fit to extract the thermal diffusivity, α , where τ is given as,

$$\tau = \alpha l^2 / \pi^2, \tag{2}$$

where *l* is the microbridge length and α is given as,

$$\alpha = \kappa / \rho c_p, \tag{3}$$

where κ is the thermal conductivity, ρ is the density and c_p is the specific heat capacity of the microbridge [12]. To compare the measured time constant data to calculations using the expected thickness removal during ALE processing, κ and ρ of the microbridges pre- and post-ALE were measured following the methods outlined by Eigenfeld *et al.* [11]. The microbridges were joule-heated using a steady-state biasing technique and κ extracted using a temperature dependent resistance joule heating model. The



Figure 4. ALE reduces the thermal time constants of microbridges by material removal. After 140 cycles of ALE, about half of the total Al_2O_3 thickness was removed. After 280 cycles of ALE, all of the Al_2O_3 thickness was removed leaving just Ru. Curve fits correspond to the extraction of thermal diffusivity.

thicknesses and densities of the pre-ALE ALD films were also measured using X-ray reflectometry (XRR) on a Bede D1 X–ray diffractometer. The X-ray wavelength for measurements was 1.54 Å, corresponding to the *Ka* transition in the Cu X-ray tube. The Bede REFS software was used to fit the XRR data and extract the thickness and densities of the ALD Al₂O₃ and Ru films. Using the measured κ and ρ and bulk c_p values, the measured thermal diffusivity, α_{exp} , may be compared with the calculated thermal diffusivity, α_{calc} , based on a relative thicknesses of Ru and Al₂O₃ contributions given as,

$$\alpha_{calc} = \frac{\alpha_{Ru} t_{Ru} + \alpha_{Al2O3} t_{Al2O3}}{t_{Ru} + t_{Al2O3}},$$
(4)

where t is the thickness of the Ru or Al_2O_3 layers.

RESULTS

The τ 's of several lengths of the Al₂O₃/Ru/Al₂O₃ test structures were measured pre-ALE and then etched using the Sn(acac)₂/HF chemistry. Figure 4 demonstrates pre and post ALE etching with Sn(acac)₂/HF. After each ALE session, the thermal time constants of the microbridges are reduced as Al₂O₃ is removed in increments of 140 and 280 Sn(acac)₂/HF cycles (Figure 4). Using equation 2, α was extracted (Figure 4 fit lines) for pre- and post-ALE sessions. Using measured κ , ρ , and bulk specific heat capacity, α_{calc} for the first 140 ALE cycles (red data Figure 4) using an expected 3.1 nm top and bottom Al_2O_3 etch falls within 13% of $\alpha_{exp}.$ For 280 ALE cycles, α_{calc} does not agree well with α_{exp} , likely due to complete removal of the Al₂O₃ and oxidation of the bare Ru structure upon transfer from the ALE vacuum system to the electrical probing vacuum system. Sources of error include slight variations in etch rates between the top and underside Al₂O₃ surfaces or the global etch rate due to reactor parameters. It is possible longer precursor exposure would reduce such error to ensure complete underside surface saturation for each ALE cycle. A notable increase in resistance of the Ru microbridges was observed after being exposed to atmosphere for several days.



Figure 5. Thermal ALE trials on microbolometer-type absorption structures with $Sn(acac)_2/HF$ chemistry (b) and TMA/HF chemistry (d). (Figure 2b corresponds to a structure with heating and no ALE.) a) A control for the $Sn(acac)_2/HF$ ALE process. No ALE with 16 hours at 200 °C. b) 67 cycles of $Sn(acac)_2/HF$ at 0.046 nm/cycles. c) A control for the TMA/HF process. No ALE, but 16 hours at 300 °C. d) 51 cycles of TMA/HF. All structures are $16 \times 16 \mu m^2$.

The microbolometer-type absorption structures were subject to both etch chemistries to remove ~3.1 nm of Al_2O_3 on top and bottom of the structure. The $Sn(acac)_2$ /HF chemistry was repeated for 67 cycles at the elevated etch rate of 0.046 nm/cycle and the TMA/HF chemistry repeated for 51 cycles at the typical etch rate. Additionally, the suspended structures were subjected to the etch duration and the given etch temperature with no ALE processing to determine if they would survive at elevated temperature for the etch duration and remain flat (Figure 5). The structures remained flat after being subject to both 200 °C and 300 °C for 16 hours with no ALE processing. After 67 cycles of ALE processing with $Sn(acac)_2/HF$, the structure yielded negligible curling. After 51 cycles of ALE processing with TMA/HF, also yielded negligible curling.

To confirm the exposed metal sidewalls of the microbridge test structures and microbolometer-type suspended structures were not etched during the above experiments, each etching chemistry was tested for selectivity to Al₂O₃ in the presence of Ru and W. The top Al₂O₃ layer in a trilayer of ALD Al₂O₃/Ru/Al₂O₃ on Si was etched using Sn(acac)₂/HF at 200 °C and the top Al₂O₃ layer in a trilayer of ALD Al₂O₃/W/Al₂O₃ on Si was etched using Sn(acac)₂/HF at 200 °C and TMA/HF at 300 °C. For each etch, Al₂O₃ was removed completely, with a 10% overetch based on its thickness and ALE etch rate. The trilayer thicknesses and densities were measured using XRR pre- and post-ALE to determine if the Ru and W films were affected by the ALE chemistries during etch exposure. Figure 6 demonstrates the results of the selectivity etch experiment for Al₂O₃/W/Al₂O₃ using TMA/HF. After etching the top Al₂O₃ layer, an thin native metal oxide layer in the form of WO_3 or RuO_2 was formed after exposure to atmosphere during XRR on the order of a few nanometers. Using thickness and density calculations for oxide growth, the measured metal oxide thicknesses via XRR correspond to the expected oxide growth for an un-etched metal film. Figure 6 demonstrates this result for TMA/HF at 300 °C in the presence of Al₂O₃ and W. Similar results



Figure 6. ALE selectivity results for TMA/HF for Al_2O_3 and W. XRR results (below) show the top layer of Al_2O_3 was completely etched after a 10% overetch and an expected layer of native WO₃ formed.

were achieved for Sn(acac)₂/HF at 200 °C in the presence of Al₂O₃/W and Al₂O₃/Ru. Further experiments for Al₂O₃ etch selectivity are underway for other materials.

CONCLUSION

The first ever demonstration of thermal ALE on suspended NEMS structures was demonstrated using $Sn(acac)_2/HF$ and TMA/HF chemistries. Etching Al_2O_3 conformally on the top and underside of microbridge test structures was confirmed using micropulse calorimetry. Etching of microbolometer-type absorption structures to reduce thermal mass and maintain a flat suspended structure was achieved using TMA/HF. The $Sn(acac)_2/HF$ and TMA/HF chemistries were selective to Al_2O_3 in the presence of ALD W and ALD Ru. The concept of conformal isotropic etching of suspended NEMS structure may be applied to a variety of niche fabrication processes for future devices. Further, ALE of a diverse subset of materials will open unique avenues for micro- and nano-device fabrication.

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HIGH-ASPECT RATIO MICROSTRUCTURES IN BOROSILICATE GLASS BY MOLDING AND SACRIFICIAL SILICON ETCHING: CAPABILITIES AND LIMITS

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ABSTRACT

We have demonstrated a microfabrication technique to create very high aspect-ratio and vertical structures in borosilicate glass. By molding glass in deep silicon trenches, we achieved glass structure aspect-ratios of 42, heights of 212 μ m and vertical wall angles up to 89.7°. We also parametrized the impact of molding parameters (heat treatment time and temperature) and defined the process limitations for defect free structures.

INTRODUCTION

Borosilicate glass is a material of interest in MEMS for optical devices (optical transparency), bioMEMS (chemical stability), power MEMS (thermal isolation), and packaging (electrical isolation). From a fabrication stand-point, borosilicate glass can be anodically bonded to silicon and has a similar thermal expansion coefficient. Unlike for silicon, the state-of-the-art microfabrication etching techniques for glass have limited capabilities to fabricate high aspect-ratio (AR) structures with high verticality. Alternatively, glass molding appears promising. This technique is based on a heat treatment of glass above its softening temperature, allowing it to flow under its own weight or a pressure gradient. Using a mold, we can create different geometries such as trenches, walls or membranes. For example, Kawai et al. have realized up to 50 µm height glass (Pyrex 7740) microgeometries achieving aspect-ratios of 8. They used a silicon mold which is manufactured by deep reactive ion etching (DRIE). Haque et al. also demonstrated the implementation of this technique in specific applications in particular a capacitive pressure sensor and a hermetically sealed resonator. These two studies illustrated the potential of the molding technique to fabricate vertical structures in glass. However, the effect of molding parameters were not studied i.e. the heating time and temperature. Liu et al. have characterized the effect of heat treatment time and temperature on the molding of Pyrex glass, but the study was done on large trenches (width of 1.5 mm and 210 μm depth). Since, they used tetramethylammonium hydroxide (TMAH) wet etching to manufacture the silicon mold, the mold walls were not vertical. In fact, the heated glass bends rather than flows into trenches.

Despite the relevance of molding to create precise glass geometries [1, 2], the limits of the process for high aspect-ratios and the impact of process conditions on the molding capabilities are not well understood. Although Haque *et al.* have reported the challenges to ensure the full filling of the silicon trenches. Additionally, there is no quantitative study of the aspect-ratio dependent molding (ARDM), defined here as the non-uniform velocity of the glass as it flows down trenches of different widths to fill them. So, what is the maximum achievable aspect-ratio by molding borosilicate glass, and what controls this limit?

In this work, we aimed to conduct glass molding into Si DRIE high aspect-ratio trenches and characterize the glass flow behavior as a function of both the micro-trench width and heat treatment temperature to define the molding flow rate and quality. This work aims to obtain a quantitative study of the glass molding to form vertical microstructures in the borosilicate glass as well as the limits of this technique.

MICROFABRICATION PROCESS

The process flow for the fabrication of borosilicate glass microstructures is described in Fig. 1. First, deep trenches are created in a silicon wafer (500 µm thick) by DRIE. The etching time was 70 min to reach up to 230 µm deep. AZ9245 photoresist was used as a mask. The width of trenches is 12, 23, 54, 104, 254 and 506 µm (Fig. 1a). The etched Si wafer and a Borofloat 33 glass wafer (500 µm thick) were cleaned in a Piranha solution (1:2 mixture of H₂O₂:H₂SO₄) for 15 min, and then the two wafers were anodically bonded in an AML-AWB-04-30 bonder (Fig. 1b). The bonding process is done under vacuum (O₂, 150 mTorr) at a voltage of 500 V to form closed low pressure micro-cavities from the array of trenches. The bonding contact force was 200 N, applied uniformly through the surface of 100 mm diameter wafers. Both wafers were heated simultaneously to 370 °C during bonding process. Afterwards, the bonded wafer was diced into square pieces of 15 mm per side. The sample is then heated in an atmospheric furnace and, the pressure difference across the softened glass will push against the viscous forces of the flowing glass (Fig. 1c). The heating temperature and time of treatment were varied. Nitrogen with 100 sccm was used as an environment gas. Since the top glass surface will deform, lapping and polishing of the surface is done to planarize and smooth the surface (Fig.1.d). To create glass structures, the silicon mold is removed by a Potassium hydroxide (KOH) etching (Fig.1.e)



e. KOH silicon etching to create glass structures

Figure 1: Microfabrication process flow of molding glass into silicon trenches and releasing.

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EXPERIMENT AND RESULTS Heat Treatment

A series of 25 samples were tested by varying temperature in the range of 750-950°C and heating time from 10 min to 32 hours. The heating treatment is characterized by five steps. In the first step, the bonded sample is heated rapidly from ambient temperature to 400 °C by an uniform heating rate of 12.6 °C/min. Afterwards, the heating is continued to up the desired treatment temperature at a rate of 3°C/min. After the isothermal heat treatment, the sample is cooled to a temperature of 400 °C with a controlled cooling rate of 0.7 °C/min. Finally, the cooling is continued without controlling the rate by free convection inside the oven down to room temperature for 750 min.

Molding Evolution in Silicon Trenches

Fig. 2 shows an example of glass flow evolution in the trenches at 850 °C and for different time steps (1, 2, 4 and 6h). The SEM cross-section images indicate clearly that there is ARDM for narrow trenches, i.e. the glass flows faster in the wide trenches compared to narrower ones. Since the pressure difference remains substantially constant, the ARDM is due to the viscous effects that are more significant at smaller dimensions.

The variation of the flow length versus heating time is plotted in Fig. 3 for different heating temperatures and trench widths. The error bars were determined by measuring variation of the flow length between different trenches having same dimensions and at the same heat treatment parameters. For a fixed trench width, the flow length increases linearly with the heating time. This linear trend in the data demonstrates that the flow velocity is a constant quantity for a given temperature and trench width. The flow velocity is then the slope of a fitted line. Extrapolation of this data to the heating time equal to zero gives an intercept. This intercept represents the total flow length during the period of heating (before the desired treatment temperature is reached) and cooling. The flow length in the trench widths of 506 μ m is not represented because they were completely filled during the heating ramp, before reaching the desired treatment temperature.



Figure 2: SEM photographs showing the time effect on the molding glass length through silicon trenches for a molding temperature of 850°C.

Glass Flow Velocity

Using the slopes of the plots in Fig. 3, values of glass flow velocity were calculated. Fig. 4 shows the relationship between glass flow velocity and heat treatment temperature for three different trench widths, 12, 23 and 54 μ m. The plots indicate that velocity increases with the heat treatment temperature, following a quadratic trend.



Figure 3: Measured flow length as a function of molding temperature and trench width.



Figure 4: Extracted flow velocity as a function of molding temperature and trench width. The velocity is calculated by linear fitting the data in Fig. 3

This is due to the glass viscosity decreasing as the temperature increases. Additionally, the plots show that the flow velocity is higher in wider trenches. This observation is attributed to the shear friction forces between the flowing glass and trench walls.

Limits of Borosilicate Glass Molding

As the heat treatment time is increased at a given temperature, two effects are observed in the molded glass. The first one is defects on the glass surface. The defects become deeper as the heat treatment time increases. The second one is the appearance of voids at the glass/Si interface when the heat treatment time is much longer. Fig. 5 shows the conditions of heat treatment when these two effects appear. We have identified three zones for the molding of borosilicate glass. In the first zone the molding gave high quality results as shown in Fig. 5a. In the second zone, the molding shows defects on the surface as seen in Fig. 5b. However, these defects in the glass can be removed by a simple mechanical polishing.



Figure 6: Maximum achievable aspect-ratio by molding borosilicate glass into silicon trenches before the appearance of defects. The width of trenches is $23 \ \mu m$.

Finally, in the third zone, the molded glass showed defects on the surface and voids at the Si/glass interface as seen in Fig. 5c and Fig. 5d. These limitations of the heat treatment time suggest that there is a maximum aspect-ratio as molded, AR_m, that can be obtained by molding borosilicate glass. Note that the AR_m is different than the aspect-ratio of the released microstructures. AR_m is defined as the ratio of the glass flow length and the width of trenches. On the other hand, the AR characterizes, in the present work, the final glass geometries after removing the silicon mold. The AR can be greater than the AR_m. By combining the results concerning the flow velocity (Fig. 4) and the heat treatment time limit (Fig. 5) we have calculated the maximum AR_m that can be achieved. Fig. 6 shows the influence of the heat treatment temperature on the maximum AR_m for two limits that are surface and interface limit. The plots indicate that the AR_m has a largest value at around the softening point of the Borofloat33 glass i.e. 821°C. This is therefore the optimal temperature giving the best results for molding borosilicate glass.



Figure 5: Temperature and heating time limits of molding borosilicate glass in silicon trenches. The chart is characterized by three zones. Zone 1: the molding gives high quality results. Zone 2: void formation on the glass surface. Zone 3: void formation on the Si/glass interface and on the surface of glass substrate.

Fabrication of High Aspect-ratio Structures

In order to create glass structures, the molded samples were immersed in KOH solution (45%) heated at a temperature of 95°C to fully remove the Si mold. The etch rate of the Si and the molded borosilicate glass was 2 µm/min and 50 nm/min respectively, for a selectivity of 40:1. Structures with a verticality of up 89.7°, depth of 212 µm and aspect-ratio of 3 were fabricated as showed in Fig. 7a. The glass was molded in 100 µm width and 220 µm depth trenches at 850°C for 180 min. The necessary KOH etching time to remove the silicon mold was 250 min. High aspect-ratios can be achieved by over-etching the glass structures after removing the silicon mold as shown in Fig. 7b. In this case, the glass was molded in 50 µm width and 220 µm depth trenches at 850°C for 330 min. The KOH over-etching has narrowed the structures to a width of 8 µm and 1.9 µm at the bottom and the top of the structures respectively. The over-etching time was 400 min. An aspect-ratio of up 42 was achieved; based on the average width.



Figure 7: SEM of borosilicate glass structures fabricated by molding glass into silicon deep trenches, after, silicon has been removed by KOH etching. (a) $AR\approx3$, height=212µm, wall angle=89.7°. (b) $AR\approx42$, height=207µm, wall angle = 88.6°.

Fig. 8 shows a range of best results of wall angles of borosilicate glass structures obtained using plasma etching and glass molding. Compared to these studies, the molding technique performed in the present work has shown distinguished results in term of wall verticality and structures depth. The wall angle of 89.7° obtained in this work is one of the best results when compared to other studies such as 88° with 120 µm depth obtained by Queste *et al.*, 89° with 15 µm depth by Ichiki *et al.* and 87° with 100 µm depth obtained by Ahamed *et al.*



Figure 8: Comparison of wall angle results from this work and from the literature.

CONCLUSION

This work demonstrates that high aspect-ratios and high wall verticality are achievable in borosilicate glass by molding. With the obtained results, we are able to control precisely the fabrication of glass microstructures. Even though this method showed notable capabilities, it presented limits in term of heating time and temperature.

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DYNAMIC CHARACTERIZATION OF IN-PLANE BULK ACOUSTIC RESONATORS USING HIGH-SENSITIVITY OPTICAL REFLECTION MEASUREMENTS

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ABSTRACT

Two experimental techniques for measuring dynamic displacement and strain for in-plane bulk acoustic resonators are presented. These techniques, optical knife-edge and photoelastic measurements, can characterize in-plane high-frequency vibrations with a degree of precision and simplicity that has not been shown previously with MEMS. The measurements are spatially resolved and can be used to reconstruct the vibrational mode shape of the resonator. Experimental results presented here are acquired using both methods on a single crystal silicon bulk acoustic resonator (SiBAR) with a fundamental resonance frequency of ≈ 13.6 MHz.

INTRODUCTION

Capacitive [1] and piezoelectric [2] bulk acoustic resonators (BARs) have received considerable attention over the last decade due to applications in RF timing, filtering, and sensing. Much of the success of these resonators stems from their ability to vibrate in a specific mode with a high mechanical quality factor and hence a sharp spectral linewidth. Contemporary design of micromechanical resonators is heavily reliant on analytical equations and finite element analysis (FEA) of the device, followed by electrical validation. However, the drawback of purely electrical characterization is that it presents only an aggregate signal at the output transduction port of the system (e.g., capacitively induced current). As a result, all-electrical transduction obscures the internal mechanics of the resonator, making it difficult to determine the causes of spurious modes, nonlinearities, and other phenomena. In contrast, optical techniques can provide more detailed and precise characterization of the resonator through spatially and temporally resolved measurement of the absolute device displacement. Laser Doppler vibrometry has been used successfully to measure BARs but typically only for out-of-plane motion [3]. Recently, three-dimensional laser Doppler vibrometry has been demonstrated but at considerable expense and complexity and with limited bandwidth for BARs [4]. Optical knife-edge [5, 6] and photoelastic [7] measurement techniques provide highsensitivity in-plane motion measurements with simplicity and high bandwidth, as demonstrated by others for surface acoustic waves [7], but have received little attention for BARs, which has motivated the presented research.

DESCRIPTION OF DEVICE & FABRICATION

The devices used in this work are silicon bulk acoustic resonators (SiBAR) that vibrate in their extensional in-plane modes (Fig. 1). The SiBARs are fabricated using standard silicon-oninsulator (SOI) processes. First, metal electrodes are patterned and evaporated using liftoff on an SOI wafer. The device layer of the SOI wafer, which is approximately 10 µm thick, is patterned and etched using deep reactive ion etching (DRIE) to form the transduction gaps and release etch holes. A transduction gap of \approx 480 nm is achieved using 5X reduction stepper lithography. The DRIE is optimized to provide vertical sidewalls with scallop depths of ≈ 80 nm/cycle and scalloping undercut on the order of ≈ 15 nm. The wafer is diced into individual chips and the mechanical structure is released by etching the 2 µm thick buried oxide layer using hydrofluoric acid vapor.



Figure 1: Scanning electron image of a width-extensional single crystal SiBAR with a transduction gap of 480 nm and a measured fundamental resonance frequency of \approx 13.6 MHz. The resonator width is 300 µm, with an expected resonance frequency of 13.8 MHz. These SiBARs are used as a platform for demonstrating precise dynamic measurements of driven motion and energy dissipation mechanisms. Due to the symmetry of the SiBAR, only one quadrant of the device (boxed) is measured and simulated.

CHARACTERIZATION TECHNIQUES

Electrical Measurements

The released SiBAR was first tested electrically using a vector network analyzer (VNA) and ground-signal-ground (GSG) probes. Standard short-open-load-through (SOLT) calibration is performed prior to acquiring the data seen in Fig. 2. It is observed that the device requires a fairly large DC bias to transduce a measurable resonance, and the signal-to-noise ratio (SNR) is low due to the presence of parasitic feedthrough. The effect of parasitics can be reduced by optimized design, a smaller transduction gap, or electronic signal amplification, but as will be demonstrated in further sections, these improvements are not necessary for optical readout and characterization.



Figure 2: Standard electrical testing of the \approx 13.6 MHz SiBAR in air, prior to wire-bonding, using GSG probes and a VNA. Magnitude (a) and phase (b) information are shown. Amplification was not used in these tests. No response is seen below $\approx 20 V DC$ bias, primarily due to the effect of the parasitic feedthrough from the device, bond pads, and handle wafer.

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Figure 3: Optical knife-edge measurements are acquired on the free edge (AA') and photoelastic measurements are acquired on the line of maximum strain: the major axis of the device (BB').

Optical Experiment Setup

The devices are mounted on a printed circuit board (PCB) and wire-bonded. The PCB is mounted on a positioning stage with 10 nm positioning precision along all three linear axes. The SiBAR is actuated by an RF signal from the VNA and a DC bias applied to the body of the resonator. An intensity stabilized helium-neon laser is used as the optical probe ($\lambda = 632.8$ nm). The laser is focused on the resonator surface using a 20X objective mounted on a tube microscope, resulting in a Gaussian spot on the device with a diameter of $\approx 2 \,\mu m$. The reflected optical signal is detected using a Si PIN photodetector with half-power bandwidth of 200 MHz and a gain of 2000 V/W at 633 nm. The photodetector output is fed back to the VNA to provide an output signal relative to the input RF drive signal. The laser output is attenuated using a polarizer to maximize the signal from the photodetector while limiting the local heating due to the photothermal effect. Precise targeting of the laser spot is achieved by viewing the system in real time with a CCD camera on the microscope, and by monitoring the average output power reflected from the resonator using an oscilloscope. The same experimental setup is used for both optical methods and no recalibration or modification is necessary.

Knife-Edge Measurements

Optical knife-edge techniques have been used in the past to measure flexural and bulk acoustic resonators [5, 6]. This technique has the potential to measure displacements on the order of 1 pm / $\sqrt{\text{Hz}}$ [6] and below. The measurements are performed by positioning a focused laser spot on any edge of the resonator surface that has high in-plane displacement. The motion of the edge modulates the reflected optical power, leading to a strong signal at the motion frequency. The leading edge of the SiBAR (AA' from Fig. 3) has minimal strain and maximum displacement along the actuation axis. The laser spot is optimally positioned at the point of maximum displacement sensitivity by scanning the spot across the gap and monitoring the photodetector output. As the leading edge of the resonator is driven to move in plane, the change in reflected power is determined by the displacement of the edge. Fig. 4 shows the magnitude and phase of the optical knifeedge signal from the resonator. The inset illustrates the knife-edge scheme and the positioning of the laser spot. Unlike electrical characterization, this method can be used to extract the absolute displacement of every point on the leading edge of the resonator by scanning the laser over the entire edge. In general, this technique can be extended to measure planar displacement on any edge with optical contrast, such as etch holes and electrodes on a piezoelectric resonator.



Figure 4: Reflected knife-edge signal when the laser is focused on the leading edge of the resonator (i.e., point of highest displacement and zero strain). A clear signal is acquired (both magnitude and phase), with an SNR better than 30 dB. The resonator was actuated with 10 mW RF power and 10 V DC bias in vacuum at 1.33 mPa (10 μ Torr). Inset: Diagram showing knifeedge measurement with position of the laser spot (not to scale).

Measurements can be acquired on edges of any orientation, allowing displacement measurements along any planar vector. Thus the vector displacements of many points on the resonator can be measured to reconstruct the device dynamics. The measurement SNR is a function of the optical contrast between the resonator and the free space gap. For this SiBAR, the contrast is between the resonator body and the transduction trench (480 nm wide, 12 μ m deep). For the current measurement, an SNR of \approx 15 dB is achieved at DC bias levels as low as 1 V, and up to 40 dB has been measured at higher voltages. The optical contrast can be improved by removing the substrate under the trench, or by using materials with dissimilar reflectivities for the resonator and the substrate.

Photoelastic Strain Measurements

Another optical method for characterizing the dynamics of high frequency resonators is to utilize the photoelastic effect, in which the index of refraction for the resonator material is modulated by strain-inducing acoustic waves. This technique has been used in the past to measure the propagation of surface acoustic waves [7]. As the resonator undergoes periodic in-plane strain, the refractive index of the material changes at the same rate, which in turn modulates the reflected intensity of the laser spot. The change in refractive index as a function of the dynamic strain is given by $\Delta n = -(0.5n^3p_{ij})\Delta\varepsilon_{ij}$, where *n* is the nominal refractive index, p_{ij} is the set of photoelastic coefficients for the material, and $\Delta \varepsilon_{ii}$ is the change in strain [7]. Most significantly, this technique does not require an edge with optical contrast and can be used on unpatterned surfaces. A single material with uniform optical properties and a clean flat surface is the ideal platform for performing photoelastic measurements. The advantage of using a single crystal silicon device such as the SiBAR is that the photoelastic coefficients of the material are well known, thus enabling the extraction of absolute planar strain at any point on the surface. The largest surface strain in the resonator is located along the central line (BB' from Fig. 3). Representative amplitude and phase data are shown in Fig. 5. As with knife-edge measurements, the photoelastic measurements can be taken at multiple points on the surface in order to generate a composite vector plot of the surface strain dynamics of the resonator.



Figure 5: Photoelastic measurement at a point along the major axis of the resonator. It is clear that there is strong intensity modulation at the resonance frequency. Both magnitude and phase information are shown, with an SNR of ≈ 25 dB for the magnitude. Same test conditions as in Fig. 4.

Mode Shape Identification

A major advantage of using these two optical techniques is that they provide spatially-resolved measurements across the entire resonator, thereby enabling reconstruction of the vibration mode shapes. The fundamental mode shape along the leading edge of the SiBAR was measured with the knife-edge technique and the strain profile along the center axis of the SiBAR was measured with the photoelastic technique. These results are compared with those from FEA simulations in Fig. 6. While there is some qualitative agreement between the expected and measured mode shape and strain profile, the differences are large enough to indicate that either the measurement, the simulation, or both are inaccurate. Due to the simplicity and repeatability of the optical measurements, we believe that the FEA model is the less accurate of the two due to unmodeled fabrication imperfections and the meshing complexity of the etch holes. This assertion is supported by our observations of mode suppression and a significant loss of transduction efficiency that were not predicted by the FEA simulations.



Figure 6: Fundamental mode shape and dynamic strain for the SiBAR. (a) modal displacement from FEA, (b) planar displacement along AA' (measured and simulated), (c) strain profile from FEA, and (d) photoelastic strain measurement along BB' (measured and simulated). Same test conditions as in Fig. 4. Simulation deformations and color scales are exaggerated for visual effect. Data in (b) and (d) are normalized to show the qualitative fit.



Figure 7: Knife-edge measurements as a function of DC bias. Unlike the electrical data, optical measurements have high SNR even at very low DC bias. Furthermore, optical readout is limited only by the noise in the optical path and not by device and wafer parasitics. Same test conditions as in Fig. 4.

Actuation at low drive levels

The optical techniques discussed above have far higher sensitivity to displacement and strain than the electrical measurements for the tested resonator. Within the range of 10 MHz to 100 MHz, motion can be detected at a DC bias as low as 1 V. An SNR of at least 15 dB was achieved throughout our measurements (Fig.7). The low drive voltage reduces the likelihood of inducing nonlinearities and heating, and does not require signal amplification electronics. The optical methods also make it possible to investigate thermomechanical noise in resonators and RF self-actuation. Lower RF power does not change the response magnitude, but does make the measurement noisier.

Higher Modes and Frequency Limits

The analysis above is presented for a 13.6 MHz SiBAR, which is well within the measurement limits of the presented system. The current configuration is bandwidth limited by the photodetector (200 MHz bandwidth). SiBARs with fundamental resonance frequencies up to 120 MHz have been measured with SNR \approx 30 dB and fundamental frequencies up to 220 MHz have been detected. Higher harmonic resonance modes can also be measured with ease. Figure 8 shows the first four odd-numbered in-plane vibration modes for a SiBAR with a fundamental frequency of 28 MHz. The data shown here was measured using the knife-edge technique. We expect that the true bandwidth limits of the measurement system will be set by a combination of the decreasing displacement and strain magnitudes and increasing detector noise as the operating frequency increases. Ongoing and future experiments involve detailed system and device characterization using photodetectors operating up to 9 GHz, and will be presented elsewhere.

Signal Magnitude and Quality Factor

The magnitude of the reflected signal is a direct indicator of the displacement or strain. As such it varies over the surface of the resonator, enabling mapping of these quantities and a reconstruction of the dynamics of the resonators. For the presented SiBAR, which is fairly large, the displacement signal is stronger and less noisy than the strain signal. For smaller SiBARs, with higher frequencies and lower absolute displacements, we expect that the strain signal will be stronger than the displacement signal.



Figure 8: Fundamental width-extensional resonance and the first three odd-numbered higher modes for a SiBAR with a width of 150 µm. All modes are measured using the knife-edge technique. The resonator was actuated with 10 mW RF power and 10 V DC bias in air. The bandwidth of the photodetector prevents measurement of modes higher than 200 MHz.

Both measurements provide the same quality factor (Q)values, at any point on the resonator, within the experimental error. This Q is the direct mechanical Q of the SiBAR, and is an indicator of the intrinsic and design dependent mechanical dissipation processes (i.e., phonon loss, electron loss, thermoelastic damping, viscous damping, and tether loss). As such, these methods provide a more accurate way to measure and isolate the various dissipation mechanisms than using the 'loaded' O values found in electrical measurement techniques. For the current SiBAR, viscous damping (due to etch holes and the transduction gaps) limits the Q to \approx 10,000 in air, and thermoelastic damping, which is exacerbated due to the etch holes, limits the Q to \approx 66,000 in vacuum [8]. It is expected that higher frequency designs, without etch holes and operated in vacuum, will allow us to mitigate these dissipation mechanisms. The Q of resonators with higher frequencies is expected to be limited by tether loss and phonon loss. The presented optical techniques will be used to study these loss mechanisms in future work.

Convolved Strain and Displacement Measurements

It is important to note that knife-edge and photoelastic measurements can be convolved when there is high strain along an edge, such as the edges of an etch hole. The strain at the leading edges of the SiBAR approaches zero so the presented knife-edge measurements were not influenced by the photoelastic effect. This was verified by measured data at points just inwards of the free edge that have near-zero photoelastic response. Conversely, at any 'solid' surface on the resonator there is no knife-edge signal, and any response is purely photoelastic. The deconvolution of the two signals based on analytical models for optical reflection will be addressed in future work.

CONCLUSION

Two complementary optical reflection measurement techniques for motion metrology of high frequency in-plane MEMS resonators were presented. The knife-edge technique can be used to measure planar displacement on any device edge with optical contrast, while the photoelastic measurement technique can be used on any clean solid material that has reasonable photoelastic coupling. Both techniques provide high sensitivity and resolution as compared to contemporary electrical methods, and are simpler to set up and use than other solutions such as 3D laser vibrometry. These techniques can be used to reconstruct the vibration mode shapes of resonators, allowing MEMS designers to better understand and optimize their designs for improved performance. These techniques also provide a more sensitive way to measure the mechanical dissipation in high frequency resonators, and can provide a path to a better experimental understanding of dissipation, even making it possible to separate and isolate different underlying dissipation mechanisms. The initial set of experiments with SiBARs indicates that there are differences between the measured and simulated mode shapes that cannot be easily explained. The possible causes for these differences and their effect on the performance of the resonator are topics of ongoing and future investigation.

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ELECTROHYDRODYNAMIC JET PRINTING: A 3D PRINTING TECHNIQUE FOR SENSOR FABRICATION

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ABSTRACT

Electrohydrodynamic jet (e-jet, EHD) printing is a candidate technique for maskless MEMS fabrication. It achieves improved spatial resolution compared to inkjet printing, and has a wider range of acceptable inks. This paper presents new results for high viscosity ink, sub 2 μ m polymer features, and printed electrodes. We demonstrate submicron precision in droplet-to-droplet positioning with *in situ* monitoring. Rapid, sub-optical measurement of printed patterns provides a unique method for characterizing e-jet printing performance with specific interest in the applications of liquid resist patterning for nanoimprint lithography and the direct printing of thermally-sinterable conductive lines for sensor and actuator fabrication. Directions for the development of e-jet printing are discussed.

INTRODUCTION

Current manufacturing processes for micro and nanotechnology are inherently expensive due to required cleanliness, precision motion control, custom photomasks, and instrumentation. Large volume production is required to recover capital expenses. The recent extension of the maskless additive manufacturing (AM) paradigm to the microscale relaxes substrate planarity restrictions, eliminates harsh chemical and high temperature processes, and reduces material consumption [1]. Hence micro-additive manufacturing (μ -AM) enables prototype or low volume production at the microscale like mesoscale AM does at the mesoscale.

Electrohydrodynamic jet printing (e-jet, EHD) is one such μ -AM approach in controlled ink droplet formation and deposition through the generation of an electric field between a charged micro-capillary pipette and a grounded substrate as shown in Fig. 1 [1]. In recent years, e-jet printing has demonstrated superior resolution to inkjet printing at a lower cost than lithographic fabrication techniques [2]. Because of the ability to cost-effectively print high-resolution droplets and lines with a range of inks, e-jet printing has shown tremendous promise for applications such as printing metallic (Ag) interconnects for printed electronics [3], [4], [5] and bio-sensors [6], [7], [8].

This paper describes the e-jet printing process, its capabilities and limits, and then compares e-jet to inkjet printing for μ -AM, and presents new high-resolution printed patterns on an integrated e-jet printer and scanner.

BACKGROUND: E-JET PRINTING Applications

High-resolution liquid deposition is needed for microfabrication, photoresist patterning, and functionalizing microsystems. E-jet printing has emerged as a cost effective and versatile, high-resolution AM technique for meeting this highresolution liquid deposition need. E-jet applications include: 1) prototyping microdevice designs, 2) depositing resist droplets such as for nanoimprint lithography, 3) direct fabrication of customizable microsystems, 4) multi-material and heterogeneous fabrication utilizing parallel deposition capabilities, and 5) selective deposition by drop-on-demand that enables targeted functionality.



Figure 1: Schematic of the e-jet printing setup. Control variables include offset height and applied voltage. Increased voltage leads to a Taylor cone formation and droplet ejection from the cone tip to print droplets or continuous lines.

E-jet printed sensor applications are reviewed in [1] including gas, temperature, chemical analyte sensing. A route to producing MEMS ceramic sensors is proposed to use e-jet printed viscous polymer precursors to ceramics [9]. E-jet printed biomolecules on photonic crystals have been shown to increase sensitivity to biomarkers [10]. Recent e-jet printed sensors include the electrodes and photoconductive material for a photoresistor [11], and semiconducting SnO₂ lines (5 μ m width) to sense NO gas [12].

One potential application to utilize the high-resolution and precision printing capabilities of e-jet printing is depositing resist before UV nanoimprint lithography (UV-NIL). Standard UV-NIL approaches use a spin-coated layer or inkjet printed pattern of resist on a substrate. An imprint mold with ~10 nm scale features is pressed onto the resist before UV exposure. Printed resist patterns offer the advantage over films for faster filling of the mold and air bubble dissolution [13]. E-jet-printing smaller resist droplets could further improve UV-NIL performance.

Process

An e-jet printer consists of a hydrophobic, conductive microcapillary nozzle that undergoes an applied voltage relative to a counter electrode at or below the substrate. Under this applied voltage, the ink meniscus forms a cone that extends into a narrow jet to deliver liquid to the substrate. The jetting behavior is affected by nozzle geometry, standoff height between the nozzle and the substrate, and material surface tension. Droplet base diameter (d_{base}) scales with nozzle inner diameter (d_n), and inversely with applied voltage (V) as given in Eq. (1) [14], [15].

$$d_{base} \propto d_n^m V^{-p}, \quad 0 < m < 1, \quad 0 < p < 1$$
 (1)

For a nozzle diameter of $d_n = 2 \ \mu m$, applied voltages of V = 200 to 800 V are typical to achieve base diameter values smaller than the nozzle diameter, $d_{base} < 2 \ \mu m$. Continuous and pulsed modes of applied voltage for e-jet printing are shown in Fig. 2. Pulsed printing enables controlled jetting at a specified time or

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Figure 2: Schematic of continuous and pulsed printing. Nonrectangular pulse waveforms including sinusoids are also used.

position (for a nozzle voltage signal that is coordinated with substrate motion) with a specific volume determined by the pulse width [16].

Capabilities

Diverse materials have been e-jet printed, as reviewed in [1]. Conductive traces have been deposited by printing and then sintering metal nanoparticle suspensions. Biological materials and UV-curable polymers have also been patterned by e-jet printing. E-jet printed droplets and lines carry an electrical charge from the deposition process, thus e-jet printed charges can be patterned on a substrate for self-assembly [5].

Limitations

The standoff height affects the electric field strength at the meniscus and thus the jetting behavior. A typical standoff height for sub-10 μ m printed features is <50 μ m. A substrate height variation of ±10 μ m can inhibit ink ejection or allow undesirable jetting behavior such as satellite droplets or scattering. The sensitivity to substrate height variations has inspired research into e-jet sensing and control as well as printhead design [17], [18], [19]. In addition to standoff height sensitivity, EHD hysteresis of the meniscus during jetting can cause the first droplets of a pattern to be larger or smaller than subsequent droplets. Additionally, nozzle clogging is a concern for e-jet printing, especially with small nozzle openings of 0.1-10 μ m. Ink suspensions must be filtered to prevent clogging and volatile solvents are avoided.

Comparison with Inkjet Printing

Drop-on-demand inkjet printing has been used to print electrical, optical and biological materials and 3D structures [20]. Either thermal bubble or piezoelectric actuation may be used, but thermal actuation restricts ink formulation to a limited boiling point range. Piezo inkjet (PIJ) ink choice is restricted by the requirement of an ink viscosity in the range of about 2 to 50 mPa-s. The temperature dependence of liquid viscosity allows some viscous inks to be PIJ printed at elevated temperatures [20]. The complex ink ejection mechanism requires MEMS-fabricated multithousand nozzle printheads with engineered features to address clogging, satellite droplet, and nozzle life issues [20].

E-jet printing provides several orders of magnitude advantage (< 100 nm droplets) over the smallest achievable inkjet droplet size (>20 μ m), which drives feature resolution. Assuming droplets form spherical caps with fixed contact angle, the base diameter is related to contact angle and droplet volume as shown in Fig. 3. E-jet printing has achieved sub 100 nm feature sizes while printing onto unstructured substrates with a variety of materials [1], [6]. The ability of nearfield e-jet to produce orders of magnitude smaller droplet volumes gives it nearly three orders of magnitude advantage in smallest droplet base diameter.



Figure 3: Droplet volumes and base diameters are related in the spherical cap droplet model for various equilibrium contact angles, θ . The 1° contact angle shows that highly wetting systems have a base diameter 5 to 10 times the in-flight diameter. Typical PIJ droplet volumes are 0.5-500 pL [20].

METHODS

E-jet printing was performed on a custom printing and scanning testbed at the University of Michigan, shown in Fig. 4. This printer has linear motor X, Y and Z stages with bidirectional repeatability ~0.2 μ m. The substrate plane is aligned with the staging plane using a manual tip/tilt stage. A UV light source is used to cure printed patterns. The setup also contains an atomic force microscope (Nanite AFM) for *in situ* sub-optical measurement of printed, cured patterns. The experimental setup is automated for printing, curing, and scanning of arrays of patterns. The vacuum substrate mount can accommodate up to 100 mm wafer substrates. For the droplet printing examples shown, pulsed e-jet printing was performed with time delays before and after substrate motion.



Figure 4: The printer components are labeled with stations for the nozzle, AFM scanner and UV curing light.



Figure 5: AFM scan and 3D view of boxed droplet from a 3x3 field of e-jet printed high-viscosity droplets (17,500 mPa-s): mean diameter 11µm, mean volume 47 fL. Printed features are UVcured Loctite 3526 Light Cure Acrylic. Raw AFM scan base flattened.

RESULTS

A high viscosity (17,500 mPa-s) UV-curable acrylic adhesive, Loctite 3526, was successfully e-jet printed at room temperature. Fig. 5 shows one such pattern with a droplet diameter of 11 μ m. This viscosity is nearly three orders of magnitude higher than the viscosity of inks that can be inkjet printed. The e-jet printing of this high viscosity ink exhibited an electrospinning behavior within the short (45 μ m) distance of ink travel. Pulsed printing was used to limit jet whipping to achieve individual droplets.

A UV-curable mercapto ester mixture, Norland Optical Adhesive 81 (NOA81), of viscosity 300 mPa-s, was e-jet printed with decreasing pitch to find the minimum pitch for a given set of printing parameters in Fig. 6. The onset of merger at a pitch of 12 μ m indicates d_{base} + scatter < 13 μ m for these printing parameters.

A high resolution (< 10 μ m) pattern was e-jet printed in Fig. 7 with two passes of equal density, with the second pass printed in the interstices of the first pass. Droplet positioning in the second pass showed scattering from its intended print site, possibly due to forces from the first printed and cured pass droplets.

A high resolution pattern with 2 μ m pitch was e-jet printed in one pass in Fig. 8 to leave an uncovered area with the text "EJET." This pattern highlights the ability to print high-resolution images with microscale precision and accuracy in droplet placement.

15 umH				
13 um H				
12 um H			111	
11 um H			围	

Figure 6: A micrograph of an e-jet printed test pattern in which the printed pitch decreases by 1 μ m in each row until merger occurs. Ink is NOA81 printed on a gold-coated glass slide. In this test, merger begins at a pitch of 12 μ m.



Figure 7: AFM scan of an e-jet printed field: mean diameter 3.4 μ m, mean volume 1.2 fL. Pattern printed in two passes: one pass of 8x8=64 droplets and a second pass of 7x7=49 droplets. Droplets were printed at X and Y pitches of 5^* sqrt(2)=7.1 μ m, giving a designed diagonal pitch of 5 μ m between droplets of different passes. Printed features are UV-cured NOA81. Raw AFM scan base flattened and processed for step line removal.



Figure 8: Atomic force microscope (AFM) scan of a field of 15x46 e-jet printed droplets: mean diameter 1.6 μ m, mean volume 0.3 fL. Material is UV-cured NOA81. Raw AFM scan base flattened.

To cover an area of 1 mm^2 with a droplet array of similar quality to Fig. 8 using the single-nozzle e-jet printer in Fig. 4 requires 7 minutes with 10,000 droplets in pulsed printing (~15 pL/min time-averaged flow rate). In continuous e-jet printing, the same 1 mm^2 area may be covered in < 1 minute with parallel lines of 10 µm pitch. These reported e-jet speeds are not yet optimized for commercial applications. E-jet printing throughput can be improved at the cost of reduced spatial resolution by using a larger nozzle at higher voltage (farfield e-jet printing). This might be a useful method of depositing, at inkjet resolution, highly viscous inks that cannot be inkjet printed.



Figure 9: A micrograph of a silver interdigitated electrode with 500 μ m length and 30 μ m line widths, printed in 10 seconds.

Figure 9 shows an e-jet printed interdigitated electrode pattern. The ink is a 25 wt.% silver nanoparticle solution in an organic solvent (UT Dots, Inc.). The highly wetting ink spreads to 30 μ m widths and exhibits the coffee ring effect. The deposition of continuous lines was performed at speeds up to 3.5 mm/s. Repeated patterns of the e-jet prints shown in Fig. 5-9 are candidates for implementing the advanced monitoring and control strategies recently presented [21].

CONCLUSION

The presented capabilities and new prints of insulating resists and silver nanoparticle solutions has shown that e-jet printing is an alternative micro-fabrication technique for: 1) prototyping microdevice designs, 2) depositing resist droplets for nanoimprint lithography, 3) direct fabrication of customizable microsystems, 4) multi-material and heterogeneous fabrication utilizing parallel deposition capabilities, and 5) selective deposition through dropon-demand that enables targeted functionality. Despite these advantages, there remain unexplored challenges in ink and surface chemistry characterization, printhead design, and integrated control to further enhance capabilities for printing functional devices. Future work will explore novel approaches for addressing these challenges.

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FABRICATION OF DOUBLE-SIDED MICROFLUIDIC STRUCTURES VIA 3D PRINTED TRANSFER MOLDING

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ABSTRACT

In this work, we demonstrate the use of 3D printed molds for rapidly fabricating multi-layer PDMS-based microfluidic devices. Because 3D printing allows for versatile and cost-effective mold construction, it can produce significantly more varied features than those generated by soft lithography. We first discuss adaptations to single-layer 3D molding, including a glass bonding technique to compensate for the limitations of surface roughness and 0.55mm built-in inlet and outlet ports to eliminate fabrication steps. Next we introduce two-sided fabrication methods, facilitated by novel built-in alignment marks. These techniques allow the construction of formerly difficult to achieve features such as non-planar 350 µm membranes, used to fabricate a single-layer membrane valve which actuates at 200kPa, and single-layer microfluidic vias, used to generate 3D flow patterns. Lastly, we demonstrate an intra-layer bonding technique where a custom 3D printed stamp selectively applies liquid PDMS adhesive, compensating for surface roughness while preventing channel clogging. Together, these techniques enable the rapid assembly of multi-layer PDMS-based microfluidic devices, combining the versatility and speed of emerging 3D printing technology with the known mechanical and biological properties of PDMS favored by microfluidic researchers.

INTRODUCTION

Microfluidics has rapidly advanced in the fields of chemical and biological research, commonly known as Lab-on-a-Chip (LoC), since 1980s due to its unique ability to make low-cost, highthroughput platforms [1-3]. The most far-reaching breakthrough in microfluidics has been the development of soft-lithography - using rigid micromachined molds to shape elastomeric polymers. Among the polymeric materials, Poly(dimethylsiloxane) (PDMS) is commonly used due to its numerous ideal properties, including its low cost, strength, transparency, and especially biocompatibility [4].

Traditional methods for fabricating microfluidic devices involve photolithography to construct micro-molds with very fine features; however, the process can be lengthy and costly. Additionally, soft lithography is limited to rectilinear features constructed through additive micromachining processes. [5] For example, while circular channels are common in large fluidic systems and are beneficial for microfluidic devices with internal movable components, [6] to date few groups have developed techniques for circular channels in microfluidics. [7]. The increasing demand for microfluidic devices is particularly high for double and multi-layered devices to allow for the implementation of more sophisticated structures and components (*e.g.* valves, pumps, and other active control mechanisms). Although multi-layer PDMS manufacturing techniques have been demonstrated, they are often time consuming, labor-intensive, and inaccurate.

3D printing has presented a unique route to build multi-layer microfluidic devices directly or indirectly with the additional molding process. For example, various groups have used 3D printers previously to make simple microfluidic devices with complex and truly 3D geometries, including microfluidic devices without moving elements, such as resistors, [8] mixers, modular components, [9] and microfluidic devices with movable components, such as capacitors, diodes and transistors. [10] Although 3D printed microfluidic devices are currently limited by: (1) the available resolution of the printer; (2) surface roughness; [11] and (3) material types, [12] the rapid developments of the 3D printing technologies are expected to advance and address these matters in the coming years. Additionally, while direct 3D printing is a rapid process for prototyping, for making multiple copies of microfluidic devices, it is slower, more expensive, and less reliable than transfer molding performed by pouring polymer into a mold. Thus, we present a hybrid approach to combine 3D printing with molding.

This work advances the 3D printed transfer molding technique from single-sided microfluidics [13,14] to multi-layer microfluidic

manufacturing, utilizing the ease of 3D printing to create multiple molds with alignment structures to shape multiple layers of PDMS structures and quickly assemble them at the end. Using this molding method, we create complex geometries in PDMS, including vias, thin membranes, and rounded channels and demonstrate rapid assembly of multi-layer microfluidic devices using built-in alignment marks which allow precise positioning of each layer without the need for a microscope.

EXPERIMENTAL TECHNIQUES 3D printed molds

Microfluidic devices were designed and converted from a positive to a negative mold shape using the computer-aided design program SolidWorks. 3D printing of molds was achieved using a Projet 3000 3D printer. During printing, the Projet 3000 deposited layers of structural epoxy (VisiJet® EX200 plastic material) and sacrificial wax (VisiJet® S100 support material); the wax was used as a temporary support for hollow spaces as well as to provide a foundational layer for the mold, and was removed during post-processing. [15]

Mold post-processing

Following printing, the molds were cleaned to remove the sacrificial wax. First, the molds were baked in a VWR 1330 FM oven 75°C for 45 min to melt the sacrificial wax. The molds were then washed in a sequence of three cleaning baths for 10 min in each bath to remove leftover wax: warm Bayes mineral oil, Ajax dish detergent in water, and potable water. The baths were heated to 75°C to ensure the wax did not re-solidify, and were placed on a hotplate with a magnetic stir bar to enhance removal of wax, oil, and soap, respectively. The molds were then dried and residual water was removed by baking the molds at 80°C for 60 min. After cleaning and drying, the 3D printed molds were treated with an anti-adhesive agent (Trichloro(1H,1H,2H,2H-perfluorooctyl)silane (PFOTS), Sigma Aldrich) gas, making the surface hydrophobic to facilitate the rapid removal of PDMS. [16] The molds were placed in vacuum desiccator with 10 drops of PFOTS agent for 30 min. Shorter times resulted in PDMS bonding to the mold and longer times caused a build-up of PFOTS which inhibited complete curing of the PDMS near the surface. [11,17]

PDMS molding

The 3D printed molds were placed onto a foil-wrapped 3D printed molding tray, which reduces PDMS waste. PDMS (Sylgard

184 Elastomer Kit) was prepared using the standard 10:1 as the base:curing agent ratio. The PDMS mixture was degassed in a vacuum chamber for 10 minutes and then poured into the 3D printed molds. The filled molds were then placed in the vacuum chamber for 45 min to degas and increase PDMS conformity. Following the degassing, the molds were baked in an 80 °C oven for 50 min. The PDMS microfluidic devices were removed from the molds by first cutting away excess PDMS and then by manually peeling the PDMS loose from the mold. The finer the printed features, the more carefully this removal must be performed. Provided no features have broken during the de-molding process and PDMS did not permanently bond to the mold, the molds are reusable without an additional cleaning process. If during de-molding PDMS adheres excessively to the 3D printed mold, the hydrophobicity of the molds must be 'recharged' by repeating the PFOTS treatment (approximately every 10-20 moldings).

RESULTS AND DISCUSSION

Figure 1 illustrates the process flow for fabricating PDMS microfluidic devices using 3D printed molds. In this example, three microchannels with elliptical-shape reservoirs of dimensions $1.5x5.9 \text{ mm}^2$, $1.1x5.9 \text{ mm}^2$ and $0.7x5.9 \text{ mm}^2$ were designed as shown in **Fig. 1a**. The overall dimension of the mold was 20x20x2 mm³ with quarter-circle (R = 2.5mm) pillars at the four corners facilitating removal of the PDMS after curing. Each channel was constructed with built-in fluid inlets and outlets molded from pillars 0.55mm in diameter and 5mm in height. The inlet pillars were strengthened by widening them halfway down to 1mm in diameter.



Figure 1. Illustration of the 3D printed transfer molding technique and process. The microfluidic device mold is designed using CAD and the PDMS structure can be bonded onto a glass substrate to make the enclosed channels and reservoirs system. The complex multi-level microfluidic device is fabricated using the 3D printed mold shown, and includes two layers of overlapping fluid flow, an elliptical membrane, a "Quake" membrane value, multiple microfluidic vias, built-in fluid inlets, and alignment marks to create a stacked, multi-layer device.

The device mold was fabricated *via* the 3D printing process and PDMS was applied, cured and released from the mold by means of the PDMS molding steps described in the Experimental section. Built-in fluid inlets can be easily incorporated to the device through the mold design, further simplifying fabrication by eliminating the need for a hole-punching step. These built-in inlets were reliable to pressures of 4 ATM. Narrower inlets were shown to hold against higher opposing pressures as the channel inlet gripped the couple more strongly. However, narrower inlets faced trade-offs with the fragility of the 3D printed mold, breaking during post-processing.

The PDMS device was then bonded to a glass slide to create closed microfluidic channels. Clearly, this 3D printed transfer molding and bonding technique can be used to fabricate conventional microfluidic circuit devices commonly produced by the soft lithography methods with the following advantages: faster and less complex process steps; easy to create complex 3D geometries; ability to fabricate circular channel cross-sections; and built-in microfluidic connectors. For multi-layer devices, a PDMS-PDMS bonding technique has been developed with a 3D printed stamp as well as the use of built-in alignment marks for rapid assembly of more than one layer of prototype PDMS structures in this work without a microscope. These techniques make possible the design, fabrication and assembly of a complex microfluidic system as shown in **Fig. 1**. For these more complex devices, the basic procedure of generating a CAD model, 3D printed mold, and PDMS replica of the mold remains the same, for a complex double-sided device incorporating a variety of microfluidic device features.

Single-sided molding

Using 3D printed molds, semi-circular and fully circular channel geometries are easily fabricated (**Fig. 2**). Surface texture in the channels arises from the interface between sequential rows of epoxy deposited during the 3D printing step. **Figure 2d** shows the PDMS device after release from the 3D printed molds, with a close-up view of the device surface. The average surface asperity of the PDMS microfluidic devices in this work, as measured by surface profilometry is approximately 20 μ m (**Fig. 2d**). This value is comparable to microfluidic devices fabricated directly by 3D printing, although transfer molded PDMS devices can exhibit significantly smaller channel diameters. Additionally, surface roughness in 3D printed devices is higher than in devices fabricated by conventional soft lithography, although this feature resolution problem is expected to be overcome as 3D printers improve. [18,19]

Due to the surface roughness of the 3D printed molds, it is difficult to achieve a tight seal when bonding PDMS and glass using standard techniques such as oxygen plasma and ozone surface treatments. For this reason, specialized bonding techniques are needed to create close-channel microfluidic devices with the 3D printed transfer molding process. Some surface roughness can be reduced by performing a standard surface treatment such as oxygen plasma and then tightly clamping the two components together, mechanically compressing surface profile. However, this technique is unreliable and often causes the glass to break during PDMS-glass bonding. A more reliable technique uses uncured PDMS as both bonding agent and as filler which overcomes drawbacks associated with surface roughness from 3D printing. The method, involves spin coating a layer of PDMS onto a glass slide to achieve a solid bond between the PDMS device and glass slide (Fig. 2f). First, a thin layer of PDMS was spin-coated onto a 20x20 mm² glass slide (1800RPM). Next, the slide was placed onto a 95°C hot plate for one minute to allow the PDMS to partially cure, and then the cured PDMS component was pressed firmly into the still-curing PDMS and held for one minute. Finally, the pressure was released and the device cured for an additional five minutes. The PDMS was held steady during this bonding to prevent slippage.

Figure 2 shows images of fully rounded microfluidic channels fabricated using the 3D printed transfer molding process. The channels are built by bonding two semi-circular channel PDMS devices. Using built-in alignment marks, the top and bottom PDMS layers can be easily assembled without a microscope (**Fig. 2b**). Fully rounded fluidic channels may have a specific use in optofluidic lithography. [18] During device manufacture and curing, features contract, allowing components to freely move. However, when used in conjunction with channels with uniform height, this lubricating fluid gap also prevents a tight seal, causing leakage within fluidic diodes or transistor-like components. Due to the physics of thin film fluid flow, there is a direct trade-off between speed of optofluidic feature movement under pressure-driven flow and the rate of fluid leakage, which may be alleviated by 3D molding.



Figure 2. Fabrication capabilities of PDMS microfluidic devices achieved using single-sided molding with a 3D printed template. (a) Photograph of a device assembly process with circular channel geometries. (b) Conceptual illustration of the use of built-in alignment marks for circular channel assembly. (c) Photograph of a device with a circular channel cross-section. (d) Photograph of surface of molded PDMS with zoomed inset, and (e) Profilometer measurement of the mold surface. (f) Process for PDMS-Glass bonding of 3D transfer molded PDMS.

MULTI-LAYER DEVICES

The increasing demand for microfluidic devices is particularly high for multi-layered devices. [13,14,20,21] Devices with more than one layer allow for the implementation of far more sophisticated and useful structures/components, such as valves or pumps, within the device as well as eliminating geometrical constraints by enabling fluidic detours and *vias*. Creating conventional multilayer microfluidic devices requires at least two lithography steps and one PDMS-PDMS bonding step (*e.g.* for "Quake" membrane valves) [22] and up to four lithography steps and three PDMS-PDMS bonding steps (*e.g.* for PDMS-based fluidic transistors).

Multi-layer construction is used to make a multiple layer device primarily to overcome the limits of 2D traditional microfluidic systems and give a traveling fluid within a device an extra dimension to travel in. In addition, with 3D printed molding, rapid assembly of multilayer microfluidic devices is easily achievable through the use of built-in alignment marks. Alignment marks can be used on the molds, PDMS microdevices, or combinations of the two, making several fabrication techniques possible (see Supporting Information for more detailed descriptions of mold-mold, PDMS-mold, and PDMS-PDMS alignment marks). Alignment marks enable precise positioning of each layer without the need for a microscope.

Thin Membranes and Vias

With the 3D printed molding approach, complex microfluidic structures including thin membranes and *vias* can be easily achieved using two-sided molding (**Fig. 3a**). By creating mold features which nest between top and bottom molds, an empty region can be made which is filled by PDMS during vacuum degassing. This work was able to consistently

demonstrate membranes down to 200 µm thick. Below that, surface interaction effects (possibly lack of oxygen) [11] interfered with PDMS curing.

Additionally, two-sided molding techniques may be adapted by using with columns that run from the bottom mold and fit into the upper mold, thereby constructing intra-layer fluidic channels. These fluidic *vias* allow fluid to flow between the top and bottom face of a single layer of PDMS (**Fig. 3d**). *Vias* reduce the complexity of multilayer manufacture by allowing three dimensional channel detours, meaning that channels may only require a limited fluidic path to overcome obstacles, such as other microfluidic channels.



Figure 3. Two-sided molding techniques: vias and thin-film membranes. (a) Fabrication flow for two-sided microfluidic device, including via detour and arbitrary thin membrane, formed by molding on both sides of PDMS. (b) 350 μ m domed membrane and (c) 350 μ m sinusoidal membrane. (d) Abbreviated fabrication process for Celtic knot-inspired device with sixteen fluidic vias.

Membrane Valve

Another common use for multilayer microfluidic devices is in membrane valves, which use pneumatic or hydraulic pressure in one fluid laver to moderate fluid flow in a secondary laver. [6] Commonly, these membrane (or "Quake") valves are multi-layer constructions which use a number of pneumatic inputs to control complex arrays of microfluidic reactors. Specifically, these membrane valves require two lithographic manufacture steps (lower, and control layers), a spin-coated thin layer of PDMS (the membrane layer) and one alignment step between the upper lower layers. In this work, we fabricated a Membrane Valve using a single-stage double-sided microfluidic molding technique, by linking a detour via with a thin membrane in an upper layer (Fig. 4). The membrane was 350 μ m thick, and the lower channel was 500 µm thick (Fig. 4b). Standard membrane valves need a photoresist reflow step during manufacture to allow the bottom layer to close fully, we were able to implement a rounded lower channel directly from the CAD file. To characterize the closing behavior of membrane valve, we ran a Gate pressure parametric sweep (Fluigent MAESFLO®) and measured the resulting sourcedrain flow rate (Fluigent Flow Unit L). We found that the valve began closing at 160KPa and fully closed by 220KPa, and had a mostly linear response during the transition (Fig. 4d)



Figure 4. "Quake" membrane valves generated by single-step double-sided molding procedure. (a) Conceptual and (b) crosssectional view of membrane valve. (c) Microscope image illustrating the active valve region. (d) Valve characteristic curves under parametric gate pressure sweep.

CONCLUSION

Rapid assembly methods for polymer microfluidic devices fabricated using 3D printed technology were presented, where 3D printed molds were used to manufacture components made out of PDMS. With these design features, the number of fabrication techniques possible to manufacture was expanded. In addition to alignment marks, which were used to optimize the bonding procedures during the fabrication of the devices, features such as thin membranes can easily be made. Because the size of the membrane can vary with the expanded resolution design limits of the 3D printer, the list of purposes a microfluidic device can perform with the feature are expanded. Bonding procedures were also investigated with the 3D printed molds and procedures such as PDMS-Glass and PDMS-PDMS bonding were evaluated. Bonding applications such as the double-sided channels and multilayer construction were explored in order to measure the effectiveness of the bonding procedures. With 3D printing, molding and bonding of devices can be completed in under a couple hours, and the possibilities of different features and purposes for a microfluidic device are expanded as resolution of features are limited to sizes around 50 µm.

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LOCALIZED BIONANORECEPTOR 3D-ASSEMBLY VIA ELECTROWETTING: AN INTEGRATED MICRO/NANO/BIO FABRICATION TECHNOLOGY

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ABSTRACT

This work presents localized functionalization of bionanoreceptors, genetically modified *Tobacco mosaic viruses*, on high-aspect-ratio micropillar arrays using electrowetting principles. This approach allows wetting of the structurally hydrophobic component with TMV solution resulting in a near-complete coverage (~95%) of TMVs over the dense microstructures with clear functionalization boundaries. The functionalization morphology and biochemical activity of the electrowetted TMVs are confirmed using SEM and fluorescent labeling, respectively. A 5-fold increase in fluorescence intensity is achieved with μ PAs over planar substrate. The TMV-based hierarchical functional structure enabled by electrowetting can serve as a high-surface-area component for next generation micro-devices.

INTRODUCTION

Recently, integration of biological molecules into microfabrication processes has been extensively explored for the development of a wide-range of micro-/nano-devices. Their natureinherited structural and functional versatility combined with advances in biology and bioengineering has great advantages for creating tailored nanoscale functionalities on devices for a wide range of applications including energy storage and sensing devices [1].

Tobacco mosaic virus (TMV) is one of the most extensively studied biological macromolecules which has been successfully integrated with various microfabrication processes. It is a plant virus featuring a high-aspect-ratio (HAR) structure (300 nm in length and 18 nm in diameter) with an ultra-high density of identical functional groups expressed on their surfaces [1]. Previously, the advancement of genetic/chemical engineering technologies allowed modification of the TMVs to be used as either biological or chemical sensing probes on microfabricated transducers [2-5]. Also, cysteine-modification of wild-type TMVs (TMV-1cys) enabled integration of the nanoscaffolds into microfabrication processes via self-assembly and electroless metallization to create high-surfacearea energy storage devices [6]. Hierarchical electrodes, where nanoscale TMVs are combined with microscale structures, amplified electrode surface areas even more so [7]. The high surface area functional architecture is very attractive not only for energy storage devices but also for on-demand miniaturized sensing platforms where would benefit from the very dense bio/chemical recognition events for higher sensitivities. However, access of TMV solution into HAR microcavities has been limited and hindered further increase of device surface area, a phenomenon attributed to the hydrophobic nature of the dense μ PAs [8].

Here, we address the aforementioned challenges through the use of electrowetting principles for localized functionalization of biological receptors (TMV-1cys) from solution on high surface area microstructure. Electric potential applied across the interface between TMV droplets and HAR-µPAs results in a breakdown of structural hydrophobicity, driving down the TMV solution into the vertical microcavities. The access of TMV solution in the lateral direction is self-limited due to the surface tension created at the wetting boundaries allowing localized bionanoreceptor

functionalization. The resulting high-surface-area structures with the electrically or chemically functional surfaces bring great advantages for the development of TMV-based energy storage devices or bio/chemical sensing platforms.

MATERIALS AND METHODS Structural hydrophobicity of Au-coated Si-µPAs



Figure 1: (a) Schematic of HAR- μ PAs fabrication process, and optical microscopy of 10 μ l TMV droplet on (b) planar Au and (c) HAR- μ PAs surfaces.

The fabrication process for the HAR- μ PAs is described in Fig. 1a. A Si wafer is etched down via DRIE using a patterned negative photoresist (NR9-1500PY, Futurrex) as the etch mask. The resulting Si micropillar arrays are passivated with 300 nm PECVD Si₃N₄ followed by sputtering of Cr (30 nm)/Au (150 nm). The footprint of the final μ PA electrode is 1x1 cm² with 10:1 (70 μ m in height, 7 μ m in diameter) and 7:1 (70 μ m in height, 10 μ m spacing between nearest pillars) aspect ratios for pillars and spacings, respectively. Such geometry of the μ PAs is selected for this work based on our previous studies which showed limited TMV assembly on μ PA densities higher than the selected geometry [8]. Also, the HAR- μ PAs with spacing aspect ratio higher than 7:1 showed unstable wettability transition during the electrowetting step (data not shown). This is expected to be due to a poor coating of sputtered Cr/Au inside the deep micro cavities limiting electric potential to be

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applied at the droplet- μ PAs interfaces. Considering the importance of high spacing aspect ratio geometry for increasing both the surface area and bionanoreceptor densities, a method to make good electrical connections between the dense micro-network is critical for the success of this technology.

Fig. 1b and 1c demonstrates the wettability of HAR- μ PAs by comparing the contact angle measurement of 10 μ l TMV solution droplet (0.2 mg/ml in phosphate buffer (pH 7)) on planar Au (Fig. 1b) and the HAR- μ PAs (Fig. 1c) surfaces. While the planar Au surface exhibits hydrophilic nature with 60 ° contact angle, the HAR- μ PAs results in hydrophobic characteristic measuring 145 ° contact angle. This is close to the theoretically expected value calculated based on the Cassie equation (1), where θ^* is the apparent contact angle on the HAR- μ PAs, ϕ_s is the fraction of the solid in contact with the TMV solution, and θ_0 is the contact angle on the planar Au substrates [9-10]. The TMV droplet on HAR- μ PAs are readily movable with no traceable liquids remaining on the electrode surface.

$$\cos\theta^* = -1 + \phi_s(1 + \cos\theta_0) \tag{1}$$



Figure 2: Schematics of the TMV droplet on μPAs (top) with optical microscopy from the top of the μPAs interfacing droplet edges (bottom). The (a) Casie-Baxter state and (b) the Wenzel state of the droplet is clearly observed in the microscopy images (focusing at edge-on pillar tips) taken before and after electrowetting, respectively (scale bars: $10\mu m$).

Structural wettability transition via electrowetting

The initial wetting state of the TMV droplet on HAR-µPAs can be described as Casie-Baxter state where liquid-air boundaries form on the very top of the uPAs as shown in Fig. 2a. The top end of each micropillars function as an arrayed surface defect where the pinning of the liquid phase limits the access of the solution into the vertical cavities. It is well-known that an electric potential can induce transition in structural wettability (from Casie-Baxter to Wenzel state) by reducing the surface tension at the solid-liquid boundaries [10]. As described in Fig. 2b, an electric potential of 0.6 V (DC) is applied for 1 minute by sandwiching a 10 µl droplet between the bottom µPAs and a planar Au electrode placed on top of the droplet using a 3D-printed 1 mm-thick spacer. The electrowetted HAR-µPAs are incubated overnight (18 hours) at room temperature for the self-assembly of TMV-1cys onto Au surfaces. The electrodes were placed in a plastic boxes sealed with parafilm to prohibit sample drving during the self-assembly.

After the overnight self-assembly step the electrodes are rinsed in phosphate buffer solution in pH 7 (PB), followed by either a standard metallization process, which involves palladium surface activation and electroless nickel coating [6], or a fluorescent labeling step to be evaluated in SEM and fluorescent microscopy, respectively. For the fluorescence analysis, each electrode is rinsed in PB after the overnight self-assembly process, and a drop of 50 µl Fluoresceine-5-Maleimide reagent (excitation: 494 nm, emission: 518 nm, Thermo Scientific) solution (0.3 mg/ml in PB) is added to each of the electrodes for 2-hour labeling reaction at room temperature. After the labeling reaction, the electrodes are rinsed in PB, and further immersed in 3 ml PB for 30 minutes to get rid of any excess/unbound labeling reagents before imaging. Α fluorescent microscopy set up (MVX10, Olympus), equipped with a broadband excitation light source and a designated imaging software (cellSens), is used to collect the data. All images are collected for 2 seconds exposure, and the images are analyzed using ImageJ to compare the differences between the samples.

RESULTS AND DISCUSSIONS

Morphology of electrowetted TMVs on HAR-µPAs

The side-view optical micrographs shown at the bottom of Fig. 2a and 2b confirm the wettability transition from *Casie-Baxter* to *Wenzel* state; while the clear liquid-air boundaries between the liquid-solid pinning points are observed before the electrowetting, no liquid-air boundary is observed on top of the μ PAs after the electrowetting step. We expected the successful wettability transition by also observing apparently diminished contact angle after the electrowetting step. Droplets sandwiched without electrowetting results in removal from the HAR- μ PAs surface by the hydrophilic top electrode (planar Au) while disassembling the experimental set up.

Comparisons of the cross-sectional SEM images shown in Fig. 3 confirm the successful functionalization of TMVs along the deep microcavities of the HAR-µPAs enabled by the electrowetting process. Fig. 3a shows the cross-sectional SEM of TMV-coated micropillars located at the center of the wetted area. Compared to our previous studies, which showed limited TMV coating at the bottom of the pillars (the electrodes were immersed in 1 ml TMV solution for the self-assembly step) [8], near-complete (~95%) TMV coverage of the pillar side-walls is achieved confirming the excellent wetting of the dense microstructure with the TMV solution. Fig. 3b shows pillars that are exposed to a TMV droplet without electrowetting. Only isolated TMVs are observed with most of the micropillar surfaces exposed with DRIE traces from its two-step etch process. This indicates that the *Casie-Baxter* state of the droplet is stable throughout the overnight self-assembly process, and

the present TMVs are likely due to the rinsing or the post metallization processes which could introduce unbound TMV particles into the cavities.



Figure 3. Cross-sectional SEM images of the nanostructured TMVs on the μ PAs. (a) Electrowetting allowed near-complete TMV coverage along the deep μ PAs sidewalls compared to the (b) non-electrowetted μ PAs. (c) The clear functionalization boundary is observed at the wetting edge demonstrating the discrete nature of the method.

Fig. 3c shows the electrowetted-TMVs at the wetting edge on HAR- μ PAs. A distinct boundary is achieved, with a representative single pillar side-wall exhibiting both TMV nanotextures and exposed DRIE etch steps on the wetted and un-wetted sides, respectively. This is attributed to limited lateral access of the TMV solution controlled by surface tension. The initial limitation of the structural hydrophobicity here plays an advantageous role to locally confine the bionanoreceptor functionalization on high surface area

microstructures. The localization capability of the electrowetting offer great opportunities for developing TMV-based arrayed sensing platforms for multi-analyte detection/analysis system.

Biochemical activity of the electrowetted TMV-1cys

Biochemical activity of TMV-1cys assembled by electrowetting on HAR- μ PAs is evaluated using a sulfhydryl (-SH on cysteine) specific fluorescent labeling reagent, Fluorescein-5-Maleimide. Three different conditions are investigated – 1) HAR- μ PAs with electrowetting-assisted assembly of TMV-1cys, 2) HAR- μ PAs without electrowetting, and 3) planar Au substrate self-assembled with TMV-1cys – to evaluate the wetting process and increased nanoreceptor density by the microstructures.



Figure 4. Fluorescence microscopy from (a) electrowetted TMVs on μPAs , (b) non-electrowetted TMVs on μPAs , and (c) non-electrowetted TMVs on planar substrate. (d) Comparison of normalized average green fluorescence intensity between (a) and (c).

Fig. 4a-c compares the fluorescence intensities between the three samples. Fig. 4a and 4b shows the green fluorescence collected from the HAR- μ PAs with and without electrowetting, respectively. The electrowetted electrodes results in localized TMV functionalization within the wetting boundary while the unwetted electrode barely emits any fluorescence even from the top of the pillars which had been in contact with the TMV droplet during the overnight incubation. This result confirms that the electrowetted TMVs are still chemically functional/selective to maleimides, and the reactivity only remains within the functionalization boundary. The fluorescence intensity from a planar Au electrode is shown in

Fig. 4c where a droplet of TMV solution was placed on the surface overnight for the surface functionalization. Comparing the results with Fig. 4a, the fluorescence intensity is much lower for the planar electrodes with less uniform functionalization of the nanoreceptors over the wetted area. The higher intensity at the wetting edge on planar substrate is attributed to local concentration difference within the droplet due to the surface tension and some liquid drying during the overnight process. Compared to the circular ring on the planar substrates, we note that the edge of the functionalization area of the HAR- μ PAs exhibits angled feature. This is due to the reduced surface continuity for the micropillar arrays compared to the flat surface.

Fig. 4d quantitatively compares the average fluorescence intensities measured from the planar and HAR- μ PAs electrodes. The intensities collected from the wetted areas are averaged, and normalized by the saturation intensity value (256 bit quantization using ImageJ). A 5-fold increase in fluorescence intensity is achieved for the same exposure time of 2 seconds, which is attributed to an increase in nanoreceptor density within the functionalization area. The enhancement factor from the HAR- μ PAs is slightly less than the theoretically expected value of 6.3. Potential reasons for the results are 1) wider focal depth for the 3D structure compared to the planar substrate, and 2) possible emission losses within the volume of the μ PAs. Despite the potential limiting factors the technology is still powerful to be implemented for optical sensing applications with the significant increase in the fluorescence intensity.

Localized TMV-1cys functionalization on a single HAR-µPAs

Two droplets of TMV solution with 10 μ l volume are placed on top of a single μ PA electrode (1x1 cm²), and simultaneously electrowetted to demonstrate the advantages of the localized functionalization technique. Only two droplets were able to be located on the same chip due to the limited device footprint and manual control for droplet positioning. A precise control of droplet sizing/positioning on the hydrophobic dense microstructure surface is necessary for reliable implementation of the technology for potential applications.

Fig. 5 shows a fluorescent microscopy taken from the interface of the two functionalization area on a μ PA electrode. The very discrete nature of the technique is demonstrated with clear separation between the local fluorescent intensities. The overlaid grayscale analysis further confirms the discrete localization with uniform fluorescence intensity levels confined by sharp edge gradients for both functionalized regions.

The result strongly supports the idea of creating bionanoreceptor functionalized arrayed platform on a single μ PA electrode. As discussed above, methods for precise control of droplet positions and sizes along with the advances in chemical/genetic modification technology of bionanoreceptors will deploy this technology for the development of TMV-based multiplexed sensing platform.

CONCLUSIONS

This work reports a breakthrough in TMV functionalization on HAR- μ PAs by utilizing electrowetting principles. The electrowetting converts the original limiting principle – structural hydrophobicity of HAR- μ PAs – to an enabling technology which allows near-complete coating of bioreceptors on high surface area microstructures with localization capabilities.

The 5-fold increase in fluorescence intensity from the hierarchical architecture confirms the biochemical reactivity of the electrowetted TMV-1cys. The demonstrated results are critical evidence that the technology can contribute for the fabrication of high performance (energy/power densities, sensitivities, etc.) microdevices. This opens up new possibilities in micro/nano/bio integrated fabrication technologies and platform for developing miniaturized-systems including micro energy storage devices and biological/chemical sensors.



Figure 5. Fluorescence microscopy of two localized TMV functionalizations on a single μPA substrate. A line scan measuring grayscale intensity is overlaid.

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MILLED MICROMOLDS FOR LARGE AREA ALL-ELASTOMER "ROBOT SKIN" WITH 3-AXIS TACTILE SENSING VIA CONTACT RESISTANCE

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ABSTRACT

This work demonstrates the first large area, all-elastomer "robot skin" capable of 3-axis tactile sensing, and features a rapid fabrication process and novel taxel geometry. The milling-based fabrication process avoids clean room time while producing features over multiple length scales, from 10's of microns to 10's of centimeters, and molds all-elastomer materials to create a mechanically flexible skin. In the presented design, normal force range and resolution were found to be 5 N and 1 N, respectively, and shear force range and resolution were found to be 430 mN and 100 mN, respectively. These values can be tuned via taxel geometry. Cyclic shear force testing was also conducted and no hysteresis was observed up to 100 cycles.

INTRODUCTION

As the field of robotics progresses towards autonomy, advanced tactile sensors are pivotal in enabling safe and dexterous interaction between a robot and its environment [1]. Robotic tasks that generally rely on vision alone, such as grasping, are greatly enhanced with the addition of tactile sensing [2]. Shear force sensing in addition to normal force sensing is especially important in detecting incipient slip of a grasped object [3]. Other wearable systems such as exoskeletons [4], shoes [5, 6], and gloves [7, 8] also stand to benefit from sensor rich "robot skins" that provide real-time force vectors over a large area.

Over the past three decades, notable progress has been made in the field of tactile sensing. Camera-based tactile sensors, in which a soft material is pressed and the deformation is processed visually, have been able to achieve microscale spatial resolution but they're typically limited to a small sensing area and have large, specialized hardware [9]. More compact and versatile sheets of tactile sensor arrays have also been developed [10], and leverage MEMS manufacturing to create microscale sensor geometries essential to multiaxis sensing. However, this method typically results in laborious and complicated multilaver assembly with sub newton force ranges [11]. MEMS manufacturing also limits the sensing area to less than that of a silicon wafer [12]. Other tactile sensors which have large sensing areas have been limited to normal force sensing only [13, 14], or have had limited flexibility [15]. Therefore, there is a need for a flexible, large area tactile sensor array capable of shear force sensing in addition to normal force sensing.

SENSOR ARCHITECTURE

Flexible tactile sensor arrays typically utilize parallel-plate style capacitors [16], or piezoresistive serpentines or strips to detect applied loads [17]. Prior work demonstrated an all-elastomer capacitive tactile sensor with high dynamic range in three axes [10], but required femtofarrad resolution which was difficult to reliably achieve even with proper electrical shielding. Meanwhile, elastomer-based piezoresistive sensors tend to suffer from mechanical hysteresis [18]. A new sensor modality was needed to be more applicable in the field, while avoiding sensor hysteresis.

A contact resistance sensing technique was developed in which two conductive features, referred to as the "pillar" and "pad", come into physical contact as loads are applied, Fig. 1. As a



Figure 1: 3-axis force sensing architecture. An applied load causes adjacent features to come into contact with one another, causing predictable changes in contact resistance in both normal and shear directions.

normal force is applied, the pillar and pads flatten and expand through Poisson's effect, and come into contact causing a uniform decrease in contact resistance on each side. Meanwhile, a shear force results in a differential contact resistance; contact resistance decreases in the direction of shear and increases on the opposite side. Elastomers such as polydimethylsiloxane (PDMS) would be especially favorable for this architecture since they are incompressible (Poisson's ratio near 0.5), which maximizes lateral expansion under normal deformation.

FABRICATION

Computerized numerical control (CNC) milling and micromachining have been widely used to fabricate lab-on-a-chip devices [19, 20], PDMS microstructures and adhesives [21, 22], and even pneumatic logic circuits [23]. In this work, a milling process to cast a conductive elastomer was developed to achieve microscale features over a large area, Fig. 2. This was preferred to clean room fabrication from prior work [10] due to being larger in area and significantly less resource intensive with respect to both time and money. For example, clean room work requires the outsourcing of masks for photolithography, expensive machines and chemicals, and many hours of processing time by a highly trained individual all while being limited to the working area/volume of a silicon wafer. Meanwhile, with the developed manufacturing process it takes less than a day to go from concept to in-hand and ready for testing without sacrificing microscale features.

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Figure 2: [Left] Fabrication flow chart. Acrylic is milled, refilled with CNT/PDMS, coated with PDMS, and peeled from mold. [Top] Milled acrylic mold. Scale bar is 1 cm. [Center] Robot skin being peeled from the mold. [Right] Isometric view of the robot skin. [Bottom] Close-up of a single taxel in the acrylic mold. [Bottom Right] Close-up of a single taxel after peeling from the mold.

A stock of Acrylic (McMaster-Carr, 8560K355) was milled in a Roland Modela ProII MDX-540 using a 406 µm diameter endmill (Microcut USA, 82016), which had a workspace of approximately 12 in by 16 in. NC instructions were coded in Tool Path Language, and generated using CAMotics 1.0.0. No rough cutting for planarizing purposes was necessary as seen in other work [23]; the stock was sufficiently planar as received. Instead, it was mounted in the CNC machine and leveled by a manual procedure: trenches 0 µm, 100 µm, 200 µm, and 300 µm deep were milled in the four corners of the stock followed by minor adjustments until each corner exhibited three trenches. The stock was cut at 10 mm/min at 7000 rpm in taxel areas, and 80 mm/min at 10,000 rpm elsewhere, and finished in approximately 2-3 hours. In the presented design, this method produced features that were 400 µm and 600 µm deep, had a minimum size of 30 µm, and created an array of 6 by 6 taxels spaced every 1 cm. The total area of the mold was 7 in by 4 in.

The mold was refilled with a conductive elastomer. Carbon nanotubes (Cheap Tubes, 030203) and 10:1 PDMS (Dow Corning, Sylgard 184) were mixed at a total weight percent of 7 wt.% carbon nanotubes in a centrifugal mixer (Thinky, ARE-310) at 2000 rpm for 90 sec. CNT's were found to be favorable over other particles, such as carbon black and silver nanopowder, due to exhibiting excellent mechanical and electrical properties in PDMS with high repeatability. The resulting tar-like CNT/PDMS mixture was spread over the mold and planarized using a screen printing squeegee (Ryonet). 10:1 PDMS was then poured over the mold, placed in vacuum for 20 min to remove air bubbles, and cured on a



Figure 3: Test setup to characterize the robot skin in 3 axes. A Thorlabs stage equipped with a square probe was used to apply displacements to a taxel, and the resultant forces were read with an ATI force sensor. Voltages were collected with an Arduino Uno (not pictured).



Figure 4: Normal and shear force testing of a single taxel. Each curve corresponds to a single pad of the taxel. In shear force tests, a light normal force of 1 N was applied prior to shearing to improve contact between the test probe and taxel.



Figure 5: Cyclic shear force testing of a single taxel up to 100 cycles. A normal force of 5 N was applied before cyclic loading.

hotplate at 160°C for 60 min. Lastly, the entire robot skin was peeled from the mold, which can be reused further saving time and money.

Each taxel consisted of one pillar and three adjacent pads, with nominal gaps of 30 μ m between the pillar and pads. Four pads could not be accommodated due to the space requirements of the electrical routing to the pillar. The total robot skin thickness was 980 μ m, with a PDMS layer thickness of approximately 380 μ m. The completed mold and robot skin can be seen in Fig. 2.

ROBOT SKIN CHARACTERIZATION

Sensor characterization was done using a Thorlabs PT3-Z8 3-axis stage equipped with a 3 by 3 mm Delrin probe, and an ATI Nano17 force sensor with 3-axis sensing, Fig. 3. A voltage divider was constructed to measure taxel voltages (i.e.: contact resistances). 3-axis force testing was conducted on a single taxel, as well as cyclic testing up to 100 cycles.

As a normal force was applied, a decrease in voltage was observed across all three pads as intended, Fig. 4. The taxel was unresponsive below 3 N, saturated above 8 N, and had a resolution of approximately 1 N. This was because below 3 N the pillar and pads are not yet in contact, while above 8 N the sensor can compress no further. The range can be tuned by adjusting the pillar height, pad height, and gap between pillar and pads. This force range is still useful for robotic manipulation applications [24].

Shear forces were applied in the direction of each pad, Fig. 4. A small normal force of 1 N was applied before shearing to improve contact between the test probe and the taxel, while minimizing the influence of normal force on the results. No change in voltage was observed after applying this normal force. In each shear case, a decrease in voltage was observed across the intended pad, while the voltage of the other pads remained relatively unchanged. Shear force range and resolution were approximately 430 mN and 100 mN, respectively. Above 430 mN of shear force slip was observed so the sensor couldn't be tested at higher forces.

Cyclic shear force testing was conducted by applying a normal force of 5 N followed by loading and unloading of approximately 400 mN of shear force towards V_3 , Fig. 5. The pad in the direction of loading decreased in voltage while the opposite increased in voltage, as intended. The magnitude of the increase in voltage was higher than the decrease due to the pad coming out of contact with pillar. No hysteresis was observed after 100 cycles. However, the out of plane pad, V_2 , experienced a slight increase in voltage. This may have been due to a slight decrease in the measured normal force, from 5 N to 4.8 N, as the experiment carried on and the taxel underwent stress relaxation.

A 3D-printed rectangular block (Veroblue, RGD840), weighted with a 1 kg mass, was applied to the robot skin and voltages were collected across the entire 6 by 6 array, Fig. 6. Change in voltage was calculated after averaging the voltages of each pad, and the largest changes in voltage were localized in the expected area. A modest amount of variance in voltage was observed in the area of the block, and may be due to the variance in taxel quality across the robot skin. Improving the overall yield and quality of individual taxels across the entire robot skin is a subject of current work.



Figure 6: Spatial data of a 1 kg block resting on the robot skin. Large variations in data may be caused by the quality variation between individual taxels.

LIMITATIONS

Although affordable manufacturing of large area robot skins with 3-axis sensing and simple electronics has been demonstrated, the most significant drawback was dynamic range. This can be partly mitigated by tuning the taxel geometry, but still lacks the dynamic range of some previous 3-axis sensors [10, 25]. To improve dynamic range, the sensing area could be encapsulated with PDMS and capacitive sensing could be used similar to previous work, but at the cost of simple electronics [10]. The current dynamic range is still useful for applications such as grasping, and demonstrating this is a subject of current work. It was also found that at high normal forces (above 8 N), the taxels became relatively insensitive to shear forces as the compressed sensor could not deform further; this is an inherent limitation of the design.

During fabrication, a high amount of force is applied to the acrylic mold as the CNT/PDMS is planarized by hand. During this step, it was found that small gaps tend to break. With a gap of 30 μ m, yield was estimated at 80-90%. In the future, larger gaps could be fabricated to improve yield while also increasing the normal force range, or a more delicate planarization process could be developed.

The sensor architecture left the sensing elements exposed to the environment, which could potentially lead to damage from repeated use. Also, conductive objects such as metals were not compatible with this architecture since they created an electrical short between the pillar and pads. A patterned insulating layer could be placed on top of the robot skin to mitigate this.

Electrical routing was fabricated in the same plane as the sensors, limiting the taxel areal density. However, taxel density can increase if a smaller array (ex: 3 by 3 with a spacing of 3 mm, ideal for fingertips) is desired because the amount of routing is significantly less. A smaller diameter endmill for the routing could also be used. An alternate approach would be to implement a multilayer design that bonds two fabricated layers: a sensing layer and an electrical routing layer. This could also potentially enable each pillar to be surrounded by four pads rather than three.

CONCLUSION

This work presented a rapid and affordable manufacturing process based on CNC milling, and featured a novel 3-axis tactile sensor architecture using contact resistance. A large area elastomeric robot skin was fabricated with a 6 by 6 array of taxels spaced 1 cm apart. The fabrication process produced features as small as 30 μ m without the need of a clean room. Dynamic range was approximately 5:1 in the shear and normal directions, but still had a force range and resolution relevant to robotic manipulation applications. This work may help accelerate the pace of elastomer MEMS research, and result in new conductive elastomeric sensors.

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NON-PLANAR ELECTRODES MADE FROM BOND WIRES FOR IMPEDANCE SPECTROSCOPY OF 3D-MICROTISSUES

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ABSTRACT

We present a new versatile method for integration of out-ofplane microelectrode configurations on planar microchips. The technology relies on the precise formation of user-defined gold bond-wire loops and has been used to develop a multi-electrode electrical microimpedance device for interrogating 3-dimensional microtissue structures. 3D microtissue spheroids are receiving growing attention, as they closely reproduce *in vivo* conditions in drug screening applications.

BACKGROUND / STATE OF THE ART

Microtissue spheroids constitute a popular three-dimensional tissue-model system in pharmaceutical compound development and are widely used as organ models in basic research [1]. Owing to their increased use over the past years, new readout methods are required that are compatible with these three-dimensional microtissue structures. Most analysis methods are applied to cell-based assays and are optimized for 2D cell layers. This holds also true for electrical impedance spectroscopy (EIS), a label-free analysis method, which holds great potential for studying cell and tissue structures [2], but which mostly relies on planar microelectrode configurations. Devising 3D microelectrode configurations that are amenable to investigate spheroid microtissue structures is challenging, as most microfabrication processes rely on planar photolithographic patterning methods.

DESCRIPTION OF THE NEW METHOD OR SYSTEM

In this paper we introduce a fabrication concept for vertical microelectrode configurations through a versatile process and demonstrate their application in electrical impedance measurements. The three-dimensional electrode configurations were formed from gold wires by using a ball-wedge automatic wire-bonding process. By varying the loop shaping parameters, not only the height of the electrodes, but also the inclination of the wire loops can be controlled.

The micro-impedance configuration was fabricated by arranging eight of these microelectrodes in a circular pattern around a micro-well. A 100 mm / four inch glass wafer (Borofloat33) was used as a substrate. First a two-layer photo resist was spun on and processed to form the lift-off masking layer. Subsequently, a metal stack consisting of a 20 nm WTi10 alloy adhesion layer and a 200 nm platinum layer was deposited by ion beam deposition. With the final lift-off solvent soak and cleaning steps the metal patterns were unveiled, which form the bond pads for the out-of-plane electrodes, a set of coplanar electrodes for comparison and the interconnect contact pads. Thereafter, the wafer comprising four chips with dimensions of 32 mm x 32 mm each was diced. On single chip level, the wirebonding was carried out, using an automated gold ball-wedge wirebonder (3088iP, ESEC / BESI, Switzerland) to form the vertical electrodes. By modifying the loop shaping parameters, the wire loop can be configured to form either symmetrical 'hair-pin' shaped loops or to get an inclined loop as depicted in Figure 1B. A final multipurpose SU-8 epoxy resin layer was applied and patterned using standard photolithography techniques. The SU-8 acts as a passivation layer to cover the platinum tracks and the bond pads, and is used to form

the micro-well. Only a short section of the wire loop that protrudes sideways into the culturing well is exposed and forms the vertical electrode. As the wire loop shape can be controlled with the bonding parameters, precise control over the vertical position of the electrodes is possible.







Figure 1: A: SEM picture showing the planar and wire electrodes in a circular arrangement around the central measurement site; B: close-up of a planar and wire electrode showing the tilted wire arrangement.

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Figure 2: A: Overview of the functional device; B: close-up of the SU-8 well and the electrode configuration

EXPERIMENTAL RESULTS

Figures 1 and 2 show scanning electron micrographs of the wire electrodes and photographs of the fabricated chip (32 mm x 32 mm). The close-up view shows the arrangement of the eight wire electrodes and the eight planar electrodes around the culturing well of 1 mm in diameter that has been defined by a 550- μ m-thick SU-8 layer. Figure 3B schematically shows, how the wire loops protrude into the well at a height of 300 μ m. The loops are inclined by up to 300 μ m. Shapes and heights in the range of 250-600 μ m could be reproducibly and reliably varied by using an automated

wire-bonder.



Figure 3: A: Arrangement of the electrode device on a glass substrate in a culture well made of an epoxy-sealed glass ring; B: schematic overview of the measurement well showing the planar and wire electrodes and the corresponding electrical field lines.

As schematically illustrated in Figure 3, wire electrodes feature larger sensitivity for determining size (and growth) of spheroids as compared to planar electrodes as a consequence of the better positioning of the spheroid in the electric field (see Figures 3 and 4).

We further tested the chip in a "tomographic" mode with two differently sized spheroids at different positions in the well (Figure 5). The inter-electrode impedance was measured between all possible 56 combinations of planar and wire electrodes. The impedance changes with respect to the impedance values of an empty well without a tissue – revealed significant changes at the electrode, which was closest to the tissue. A localization of the tissue can then be used for targeting automated microscopy or for tomographic reconstruction of the tissue shape inside the well.



Figure 4: Comparison of wire and planar electrode configurations. The differential signal was obtained by subtracting the signal magnitudes of measurements without tissues from those with tissues. The wire electrodes provide larger differential signal sensitivity than the planar electrodes.



Figure 5: Normalized impedance changes (normalization has been done by division through values of an empty well without tissue), overlaid to the corresponding micrograph. The location of the tissue can be clearly identified according to the largest impedance changes (thick red lines). Small changes in impedance are depicted as thin blue lines.

CONCLUSION

In this paper we presented a versatile method to fabricate devices with out-of-plane electrodes. By combining a wirebonding process with standard lithography processes for the metal and SU-8 patterning, devices with arbitrary electrode arrangements can be conveniently fabricated in short time, as compared to using more complex techniques like laser patterning, 3D lithography etc.

A higher sensitivity of the wire electrodes compared to the planar electrodes has been shown and proof-of-principle experiments for "tomographic" applications revealed the position of a microtissue spheroid.

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ORIGAMI FABRICATION OF THREE-DIMENSIONAL BIOFUEL CELLS WITH NOVEL ANODE MATERIALS

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ABSTRACT

We demonstrated a novel origami technique for fabricating three-dimensionally structured microbial fuel cells (MFCs) by using paper as a substrate. A simple 3-D MFC was created from a 2-D sheet of paper having four functional compartments (i.e. anode, reservoir, proton exchange membrane (PEM), and aircathode) through high degrees of folding along pre-defined creases. The device rapidly generated power with a small amount of bacteria-containing liquid through rapid adsorption and attachment of the bacterial cells to the anode. Graphite-polymer composite and graphite ink with activated carbon were readily applicable as novel anodic materials on paper and generated enhanced performance than the conventional graphite ink or gold anode by a factor of 12fold or 4-fold, respectively.

INTRODUCTION

Biomass can be an excellent energy harvesting source that is readily available even in resource-limited environments [1]. Typically, microbial fuel cells (MFCs) have been used as energy transducers that convert the biological energy in biomass directly into electricity via microbial metabolism, and, as such, MFCs are gaining acceptance as a future alternative green energy technology and energy-efficient wastewater treatment method [2-4]. MFCs are comprised of anodic and cathodic compartments separated by a proton exchange membrane (PEM) so that only protons or other cations can travel from the anode to the cathode. A load connects the two electrodes to complete the external circuit. Microorganisms oxidize organic matter in biomass, completing respiration by transferring electrons to the anode. During this reaction, chemical energy is captured in the formation of adenosine triphosphate (ATP) throughout the electron transport chain. With the successful validation of the conceptual macro-sized MFCs as a low-cost renewable energy technology, recent research has focused on miniaturizing MFCs for potentially powering point-of-care (POC) diagnostic tools due to low-cost, environmentally friendly features and easy accessibility in those resource-limited regions [5-7]. However, the promise of this technology has not yet been translated into practical POC applications, because (i) even smallscale MFCs require a relatively high start-up time to accumulate and acclimate microorganisms on the anodic surface (several hours to days), (ii) their device configuration is complicated with necessary multifunctional parts (anode, cathode, and ion exchange membrane) along with microfluidic tubing for liquid inlets/outlets, and (iii) their operation requires additional power/equipment to continuously inject organic fuels.

In this work, we reported a novel origami technique for fabricating three-dimensionally structured MFCs by using flexible paper substrate (Fig 1). A 3-D MFC was directly created from a 2-D sheet of paper through high degrees of folding along pre-defined creases (Fig 1a~1d). The device included four functional layers; (1) anode, (2) reservoir, (3) proton exchange membrane (PEM), and (4) air-cathode. This fabrication/material breakthrough allowed the creation of a novel MFC platform made only of paper



Figure 1: (a) ~ (d) Origami fabrication of the paper-based MFC and (e) schematic diagram of a cross section of the device and its test setup. (1) anode, (2) reservoir, (3) PEM, and (4) air-cathode).

substrates unlike our previous carbon cloth-based paper devices [8-10]. The MFC configuration/operation became simplified by using paper because the microfluidic reservoir/PEM were easily patterned with hydrophobic wax and the device had the ability to flow the fluid through capillary action without external pump/tubing. In addition, the device rapidly generated power with a small amount of bacteria-containing liquid through rapid adsorption and attachment of the bacterial cells to the anode (Fig 1e). The device platform developed in this work also provided a simple technique to explore anodic materials on a paper substrate. Graphite-polymer composite and graphite ink with activated carbon were readily applicable as a novel anodic material on paper and generated enhanced performances than the conventional graphite ink or gold anode. This work will represent the fusion of the art of origami and paper-based MFC technology, which could provide a paradigm shift in the architecture, material, and design of integrable and stackable paper-based batteries.

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Figure 2: (a) \sim (d) Fabrication processes of the paper-based MFC and (e) the device size (mm). Six devices were prepared with different anodic materials.

EXPERIMENTAL SET-UP Device Fabrication and operating principle

The 2-D sheet of paper (Whatman #1 filter paper) was designed and patterned to have folding tabs on which the anode, reservoir, PEM, and air-cathode were functionalized, respectively (Fig 2). The hydrophilic zones were first prepared with hydrophobic wax boundaries (Fig 2a). The right tab patterned with melted wax was used as the PEM, providing hydrophobic properties and proton conductance (Fig 2b). An anodic material was applied on the middle tab with an inlet hole for bacterial injection (Fig 2c). The left tab was used as a paper reservoir to contain bacterial cells and their organic fuels (Fig 2d). An activate carbon (AC) based air-cathode was constructed on the top tab of the paper. Anode/reservoir/PEM/air-cathode sandwiched construct was well aligned by folding the tabs along pre-defined creases (Fig. 1). The bacterial cells oxidized the organic media in the reservoir layer, completing respiration by transferring electrons to the anode. The protons produced during the microbial metabolism diffused across the hydrophobic wax-based PEM and reduced to water in the presence of oxygen at the air-cathode.

Anode preparation on paper

We prepared five different anode materials for the paper-based MFC. Four different anode materials were screen-printed on the

hydrophilically-patterned paper zone with the surface area of 7.34 cm² (Fig 2e); (i) composite graphite particle/PAA (Polyacrylic composite graphite particle/40% PTFE acid). (ii) (Polytetrafluoroethylene) (iii) graphite ink with AC, and (iv) graphite ink alone. The graphite ink (Ercon Inc.) has been widely used as a common electrode material for paper-based devices [11]. The additive polymers such as PAA and PTFE can be very useful as an anode material due to their facile processibility, hydrophilicity, biocompatibility and stability, all of which lead to a high bacteria density with high biodiversity while the carbon materials increase the mechanical properties and electrocatalytic activities [12, 13]. Graphite particle (particle size distribution: $0.2\mu m \sim 20\mu m$) and AC can create activation centers on anodic surfaces to improve the bacterial adhesion/biofilm formation and the electron transfer efficiencies [14]. (v) The fifth anode was prepared by depositing 100nm of gold (Au) on the substrate with chrome as the adhesion layer. Since gold is one of the most standard biocompatible materials for MEMS structures, many micro-sized MFCs use gold as an electrode material [15].

Air-cathode preparation on paper

AC-based air-cathode was first constructed on one side of the paper with carbon spray to provide structural support and to function as a current collector [16]. Then, the AC catalysts (CABOT Corporation) (15 mg/cm²) with a blinder solution was applied on top of the carbon-sprayed paper zone, which was subsequently air-dried for 24h. The blinder solution was prepared by adding a mixture of (i) 1200 μ L of 5 wt% Nafion solution, (ii) 150 μ L of DI water, and (iii) 600 μ L of isopropanol into a beaker, followed by ultrasonication for 1 min. Due to the porous structure of the paper, the other side of the paper allowed the oxygen to reach carbon to complete the redox reaction for the MFC operation [17].

Inoculum and measurement setup

Shewanella oneidensis MR-1 were grown from -80°C glycerol stock cultures by inoculating 20mL of L-broth medium with gentle shaking in air for 24h at 35°C. The L-broth media consisted of 10.0g tryptone, 5.0g yeast extract and 5.0g NaCl per liter. Both cultures were then centrifuged at 5,000rpm for 5min to remove the supernatant. The bacterial cells were re-suspended in a new medium and used as an anolyte for the device. We measured the potentials between the anodes and the cathodes with a data acquisition system (National Instrument, USB-6212), and recorded the readings every 1 min via a customized LabView interface. An external resistor connected between the anode and the cathode closed the circuit. The current through this resistor was calculated using Ohm's law.

Bacterial Fixation and SEM Imaging

The paper-based MFCs were disassembled, rinsed, and adherent bacteria on each anode were immediately fixed in 2% glutaraldehyde solution overnight at 4°C. Samples were then dehydrated by serial 5 min transfers through 50, 70, 80, 90, 95, and 100% ethanol. Fixed samples were examined using an FESEM (Field Emission SEM) (Supra 55 VP, Zeiss).

RESULTS AND DISCUSSION

After the inoculum was dropped through the inlet of the device, the voltage curves with and without the load were measured (Fig 3a). Before closing the MFC circuits with resistors, the open circuit voltages were recorded for 8 minutes. Then $100k\Omega$ and subsequently $10k\Omega$ was connected to enable current generation.



Figure 3: (a) Voltage profiles measured from the devices with six different anodic materials. (b) Open circuit voltages were measured for the first 8 minutes and then all devices were connected to (c) 100 k Ω and (d) 10 k Ω external loads and the currents were calculated. (Poly(amic) acid: PAA, Polytetrafluoroethylene: PTFE)

Open circuit voltages (OCVs)

Fig 3b shows the OCVs of the MFCs with different anodic materials; graphite particle/PAA, graphite particle/PTFE, graphite ink with AC, graphite ink and gold. The OCVs increased and reached saturated values, which were relatively less than that of conventional MFCs (Fig 3a). Oxygen penetration into the anode abiotically and biotically reacted with the anode and reduced the OCVs [18]. Measured OCVs varied between the different MFCs, clearly indicating performance variations according to the anodic materials. The composite anodic materials such as graphite particle/PAA, graphite particle/PTFE, and graphite ink with AC generated the lower OCVs than the graphite ink and gold.

Current generation

After reaching an approximately stable OCV, we connected a 100K Ω resistor between the anode and cathode electrodes (Fig 3c). After 5 minutes of operation (Fig 3a), the resistor was replaced with the 10k Ω to harvest more current generation. The highest current was obtained by the graphite ink with AC as an anode, which was twelve times higher magnitude than that of the graphite ink only. This result indicates that AC particles provide a larger bio-accessible surface area and higher electron transfer efficiency than on the graphite ink-based anode. The graphite particles with PTFE also showed a good performance in current generation. The PTFE has long been used as a binder material for fabricating gas diffusion electrodes in fuel cells because of its chemical stability and hydrophobic nature [19]. Furthermore, the use of PTFE leads to a more porous structure which can improve the surface area and the affinity of the anode electrode for the bacterial cells' attachment [20]. The hydrophilic property of the material also contributes to the higher performance of the MFC. However, the higher hydrophilic features, the thicker and more condensed bacterial biofilm may be formed, clogging the pores and consequently hindering the transport of nutrients/waste products. Moreover, incorporation of PTFE can decrease the electric conductivity of the electrode [20]. Therefore, the concentration of PTFE must be optimized to maximize the performance. In this work, 40% PTFE with the graphite particles generated the best performance.

Although the MFC with the graphite particle/PAA showed lower current production than the other composite materials, the current generation gradually increased with time under both resistors (Fig 3a). This is mainly due to the fact that the introduction of PAA can increase the hydrophilicity of the anode and provides better bacterial attachment with time. The best performance was obtained by the 60% PAA with the graphite particles. The gold material showed relatively higher current under the 100k Ω resistor, but their performance significantly degraded with the 10k Ω . Although the gold is one of the most standard biocompatible materials for bio-devices, it is not appropriate for the disposable, inexpensive paper-based device applications. Overall, the maximum current generation (3 μ A) was obtained from the graphite ink with AC followed by graphite particle/PTFE



Figure 4: SEM images of the (a) reservoir and (b) anode (graphite ink with AC) used in the paper-based MFC. Scale bar is $2\mu m$.

composite, which was much higher than the conventional graphite ink-based anode on paper. Very densely packed bacterial cells were observed on the paper reservoir and on the graphite ink/AC anode after current generation (Fig. 4). The strong capillary force of the paper reservoir contributed to rapid adsorption of the inoculum and promoted aggregates of bacterial cells in the paper matrix, allowing a number of cells to attach to the anode.

CONCLUSION

This work provided a novel technique for manufacturing 3-D MFCs by using paper as a substrate and applying the art of origami. Moreover, we investigated the several composite materials to optimize surface characteristics for bacterial attachment and electron transfer efficiencies. The carbon-PTFE composite and the graphite ink/AC showed the potential as a novel anode material for paper-based MFCs to increase surface area, porosity, biocompatibility, conductivity and biofilm formation. In particular, the graphite ink/AC material outperformed conventional graphite ink by a factor of 12-fold and even biocompatible gold electrode by 4-fold.

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PHOTOLITHOGRAPHY-FREE LASER-PATTERNED HF ACID-RESISTANT CHROMIUM-POLYIMIDE MASK FOR RAPID FABRICATION OF MICROFLUIDIC SYSTEMS IN GLASS

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ABSTRACT

A photolithography-free laser-patterned hydrofluoric acidresistant chromium-polyimide tape masking method for rapid prototyping of microfluidic systems in glass is presented. The patterns are defined with a diode-pumped solid-state laser. Minimum feature size is limited to 30 μ m; minimum spacing between features is limited to 40 μ m. The patterned glass substrates are etched in 49% hydrofluoric acid at ambient temperature with soft agitation (up to 18 min). This method demonstrates comparable results to current more sophisticated masking methods in terms of the etched depth (up to 95 μ m in borosilicate glass), feature undercut in isotropic etch (about 1.47), mask hole density (negligible, practically zero) as well as high yield and reliability.

INTRODUCTION

The physical and chemical properties of glass as well as its compatibility with common microfabrication processes make it an advantageous material for numerous MEMS and bio-chip applications: microfluidic devices used for bioanalysis, mass spectrometry [1, 2], microflow cells for single molecule handling of DNA [3], micro polymerase chain reaction (PCR) devices for DNA amplification [4].

Wet etching of glass in hydrofluoric (HF) acid solution is often the preferred microfabrication method due to the high etch rate, smoothness of the etched surface, and compatibility with batch processes. Currently, there are three major masking methods used for wet etching of glass in HF acid: metal or silicon-based hard masks, photoresist masks, and using a composition of a hard mask layer with photoresist layer on top of it.

Although metal-based or silicon-based hard masks show excellent adhesion with the glass substrate and high resistance to HF acid solution [5-8], high concentration of pinholes in the field and notch defects on the edge of the etched geometries are the primary reasons for failure [9]. Increasing the thickness of the hard mask helps to reduce the number of pinhole defects but introduces the problem of residual stress in the mask [8, 10].

The use of a hard mask with a hard-baked photoresist layer on top of it has been demonstrated to be a more successful masking method for deep wet etching of glass and fused silica substrates [11-13]; reported to withstand a 20 h etch with resulting depth of $525 \,\mu$ m in fused silica [14].

The application of photoresist mask alone on glass or fused silica surface was reported to survive \sim 30 min HF acid etch with resulting depths of 35 µm in fused silica [15, 16]. Photoresist mask preparation is sophisticated and prone to failures [5, 6]. Thus, a combinational mask consisting of a hard-mask layer and a thick photoresist layer is the preferred masking method due to difficulties of the single-layer masks.

We report a new glass masking method with a chromium metal layer and adhesive polyimide tape (Kapton® tape) for rapid prototyping of microfluidic systems in glass using wet etching. Several examples of fabricated microsystems are demonstrated (Fig. 1). A variety of shapes and sizes are available for liquid and gas phase chemical detection applications. Successful bonding of the fabricated chips with either anodic or thermal fusion bonding demonstrates that no material was etched in the mask protected regions. The ink-filled microchannels (Fig. 1C) show no leakage or channel clogging defects. The proposed method can be applied to individual microchip-sized glass substrate or to wafer-level microfabrication processes. A 4 inch wafer containing 6 microfluidic chips is laser-patterned in 22 min, etched for 15 min in 49% HF acid, and anodically bonded to silicon wafer in 3 h. The bonded wafers containing microfluidic systems are cut into individual chips with a dicing saw (Disco DAD 321, Japan).

METHODS

Glass substrates and mask preparation

The experiments are performed with 50x50x0.8 mm borosilicate (Borofloat® 33 by Schott Co.) glass slides. The slides are cleaned in 4:1 Piranha solution (H₂SO₄/H₂O₂) and sputter-coated with a 180±28 nm thick chromium layer. A 50 µm thick polyimide tape with adhesive side (Kapton[®]) is applied on the chrome coated surface of the slide. The tape is gradually applied



Figure 1. Microdevices with initial channel width of 40 μ m are etched in 49% HF for 15 min with chromium-polyimide tape mask. Etched microchannels are 75 μ m deep and 225 μ m wide. (A,B) Etched glass wafer is anodically bonded to silicon surface. (C) Surface with etched microchannels is bonded to a plain glass surface in thermal fusion. The wavy microchannels are filled with red ink. (D) SEM image of the wiggly channel 70 μ m deep.

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Solid-State Sensors, Actuators and Microsystems Workshop Hilton Head Island, South Carolina, June 5-9, 2016 from one side of the slide with soft plastic spatula (12.5 mm wide) to avoid air bubbles and ensure good contact of adhesive. In order to see the effect of each masking material individually, three groups of substrates are prepared: 8 glass slides with chromium metallized layer only - no adhesive polyimide tape; 6 glass slides with adhesive polyimide tape only – no chromium metal layer; 18 glass slides with chromium metal layer and adhesive polyimide tape.

Defining micropatterns with laser

The geometries are patterned with a commercial diodepumped solid-state laser (Samurai UV Marking/Micromachining System, model 3530-30; equipped with telecentric lens, F=103 mm; DPSS Lasers, Santa Clara USA). The laser parameters are set at 1.5 Watt, 70 kHz frequency, and 100 mm/sec scan speed with path repetition of 100. With these power parameters and installed optical lens, the minimum feature size is limited to the diameter of the laser beam (30 μ m). The minimum spacing between features is limited by the thermal shrinkage and adhesive contact area of the Kapton® tape to 40 μ m. The inner area of the geometries with dimensions larger than the laser beam diameter is filled with multiple laser passes with a 10 μ m spacing increment from passto-pass. The minimum power input to define geometries is preferred to avoid distortion of fine features and glass cracking.

Mask for etch optimization

The mask for etch optimization consists of 12 regions containing straight lines and some chess-style squares. Each region contains 19 lines of the same width but distributed spacing. The width of the lines increases from 35 μ m, in region one, to 200 μ m, in region twelve, which allows to investigate the dependence of the etch rate on geometry opening width. In each region, the spacing width between lines increases from 40 μ m to 373 μ m. The variable spacing allows us to determine the minimum features spacing (highest density) as a function of the masking material and etched depth. The mask pattern is composed in CAD and imported into WinLase® commercial laser marking software (Lanmark Controls Inc., USA). The actual mask dimensions are confirmed for each masking method with a profilometer (DektakXTTM, Bruker Corp.).

Glass etching in 49% HF acid

The laser-patterned substrates are cleaned from debris with a simple DI water rinse and 1 min DI water sonication bath. The prepared substrates are etched in 49% HF acid with soft agitation. A preliminary experiment was done before the main study to determine the maximum etch time period for each masking method. The number of prepared glass substrates in each group corresponds to the maximum number of minutes that each mask survives.

To determine the etch rate and confirm the maximum time period for each of the three masking methods, the glass substrates of one group are placed into the acid bath and taken out at one min increments until noticeable defects occur in the mask. All etching is done in a fume hood equipped for acid wet etching with an operator wearing acid resistant personal chemical protection.

Characterization of the etched microstructures

The quality of the etched features is characterized with a profilometer (DektakXTTM, Bruker Corp.). For each masking method, three scans are made at different positions across each region for every etch period (min). The measured depths of the etched lines are used to evaluate the etch rate and its dependence on mask opening width. The measured widths of the etched lines

are used to evaluate the undercut ratio and measured widths of the spacing regions are used to evaluate the minimum features spacing. All profilometer data files are saved as a .CSV file for the subsequent data processing steps. The widths and depths of the etched features are analyzed with MATLAB[®]. In each region, three profilometer scans, over 19 etched features, yield 57 profiles for evaluating depth and etch rate, width and undercut ratio per each time period (min). The only exception is the adhesive polyimide mask which shows strong dependence of undercut ratio on features spacing; only three feature profiles are available for evaluating etch parameters for each line width and spacing width.

The computer processed results are confirmed with scanning electron microscope images and measurements.

RESULTS AND DISCUSSION

In order to study the individual effect of each masking material, the three groups of substrates were compared in terms of etch duration (maximum depth) and undercut ratio.

Figure 2 shows an SEM image of the cross section of the etched profile and compares the development of the undercut distance for the masking methods. Chromium metal hard mask demonstrates minimum undercut for depths up to 45 μ m but fails a longer acid etch. Addition of adhesive polyimide mask over the chromium layer allows for a deeper etch with no compromise in undercut ratio. Although adhesive polyimide tape mask on glass is suitable for shallow etches, the wide undercut makes it unsuitable for applications with high density features.

Figure 3A demonstrates the dependence of the etch rate on the initial mask opening width of the feature. Wider geometries are etched at faster rates. The chromium metal hard mask demonstrates little dependence of the etch rate on the mask opening width while an adhesive polyimide tape mask shows



Figure 2. (A) Cross sectional profile of 75 μ m deep lines etched in 49% HF using Chromium-Polyimide mask. (B) Mask undercut for the three masking methods. Initial mask opening width is 98 μ m. The feature width increases with the etched depth. The best mask demonstrates minimal isotropic undercut and largest depth.



Figure 3. All values and error bars are average estimates based on 57 profilometer measurements of the etched features. Error bars show one +/- STD error. (A) Etch rate as a function of mask opening (geometry width). (B) Under etch (undercut) ratio as a function of mask spacing (width between etched regions).

strong dependence for microgeometries with mask opening width <60 μ m. The strong dependence of the etch rate for adhesive polyimide mask may be due to the presence of residual adhesive layer (glue) between glass and polyimide. The laser ablated parts of the glue (burned flake particles) may fill the narrow gaps and prevent acid access to the etched cavity. Longer DI water sonication bath or more optimized laser settings may reduce this dependence and allow narrow geometries to be etched to the same depth as wider openings.

Although the composite chromium-polyimide mask has the lowest etch rate, it survives the longest HF acid etch, up to 18 min in comparison to 6 min for chromium and 4 min for the polyimide mask. The longer etch allows us to make microstructures with twice the depth. The overall decrease in the etch rate for the chromium-polyimide tape mask in comparison to the chromium mask may be due to the increased thickness of the mask (50 μ m) and not fully optimized laser settings. The laser settings are kept constant for all 3 masking methods for direct comparison of mask stability in etch (eliminate dependence on laser patterning) otherwise the laser settings should be optimized for a specific masking material.

Figure 3B compares the three masking methods in terms of the geometry undercut ratio and helps to evaluate how close one can place microstructures that are to be etched to a certain depth. The isotropic undercut ratio is evaluated with measurements of width and depth of the etched features. The ratio is defined as the half difference between final width of the etched feature and initial width of the mask opening divided by the depth of the etched feature. Chromium and chromium-polyimide masking methods demonstrate a low undercut ratio (~ 1.47) and allow for a relatively high density of etched features. The adhesive polyimide tape mask demonstrates a high dependence of undercut ratio on the feature spacing. Any features that are placed closer to each other than 180 μm fail to survive a 4 min (28 μm deep) etch; the mask is lifted up and peels off. The undercut ratio decreases as the spacing between features increases. The error bars in figure 3 correspond to one standard deviation above and one standard deviation below the average value.

Figure 4 compares the quality of the etched features for the 3 masking methods. The SEM images show not only undercut of the etched features but pinhole and edge defects of the mask. The chromium hard mask (Fig. 4A and B) shows a high number of pinhole defects. The defects are especially harmful when located at the edges of the etched features. The chromium mask is also very susceptible to scratches introduced while handling the substrate. The adhesive polyimide mask is simple to apply but shows severely washed away feature edges that lack sharp corners (Fig. 4C and D). The polyimide tape mask applied on a clean glass surface can withstand only a shallow etch, up to 30 µm versus 45 um for the chromium mask, and 80 um for the chromiumpolyimide mask. The chromium-polyimide mask (Fig. 4E and F) demonstrates not only the deepest etch but also the highest quality against pinhole and scratch defects. Comparing pinhole density of the 3 masks, we find that polyimide covered substrates have no pinhole defects.

The quality of the chromium layer for all chromium layer masked and chromium-polyimide masked substrates should be the same because they all were coated in a single batch. Thus better optimization for quality of the chromium layer will improve both masking methods.

The presented results characterize masking methods for the deepest (aggressive) etch in 49% HF acid. Shallow and more controlled etches can be done with various concentrations of Buffered Oxide Etchant (BOE) solutions. The proposed mask can withstand an hours-long BOE etch but the etched depth is very shallow due to lower etch rate of glass. The mask eventually fails mainly due to duration of the etch period.

CONCLUSION

We describe a new glass masking method with: mask stability in terms of maximum etch rate and maximum etched depth; feature density in terms of undercut ratio and minimum feature spacing. Due to its resolution and etch depth limitations, the proposed method is not suggested as a full substitute to currently-used methods involving a metal hard mask with thick photoresist. But we find the proposed method to be very useful for rapid microfabrication of gas and liquid microfluidic systems because of its simplicity and reliability. The demonstrated 6 microfluidic chips, on a single 4 inch wafer, were fabricated in 1 h and anodically bonded in 3 h while preparation of a reliable metalphotoresist mask requires hours of cleanroom time. Air bubblefree application of polyimide tape mask on chromium coated glass surface demonstrated repeatable results with multiple wafers. The use of a laser to define the microfluidic features in the chromiumpolyimide tape mask side-steps the costly and time consuming



Figure 4. Compare the quality of the etched features for the three masking methods. (A, B) Chromium mask; 30 μ m deep etch. Initial square size 90 μ m and line width 45 μ m. (C, D) Polyimide tape mask; 17 μ m deep etch. Initial square size 250 μ m and line width 57 μ m. (E, F) Chromium and polyimide tape mask; 65 μ m deep etch. Initial square size 200 μ m and line width 57 μ m.

photolithography step. Possible rapid change of microfeatures layout and size is very beneficial for faster microfluidic chip design development and performance optimization.

The proposed method of fabrication can potentially be implemented in research institutions with no access to a standard clean-room facility. Chromium coated glass slides can be acquired from commercial supplier. The laser patterning step does not require a dust-free environment. Glass HF etch can be done in a regular fume hood equipped for wet etching. The bonding step is the only step that requires a particle free environment.

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3-D PRINTED MICROFLUIDIC DEVICES FOR ELECTROHYDRODYNAMIC GENERATION OF CORE-SHELL MICROPARTICLES

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ABSTRACT

We report the first 3-D printed coaxial electrospray devices for uniform generation of core-shell microparticles; the microencapsulation technology is of great importance in applications such as controlled drug release and self-healing composites. The devices, fabricated with stereolithography, produce core-shell microparticles from coaxial spouts with 150 µm-thick walls fed by 700 µm-diameter microchannels. Experimental characterization of single- and double-emitter devices using deionized water as inner flow and sesame oil as outer flow demonstrate uniform emitter operation; the per-emitter current is proportional to the square root of the flow rate of the driving liquid (water) and is independent of the flow rate of the driven liquid (oil), as predicted by the theory. The average outer diameter of the core-shell microparticles can be modulated by varying the flow rates fed to the spouts.

INTRODUCTION

Coaxial electrospray is an electrohydrodynamic process that creates core-shell microparticles by atomizing a coaxial electrified jet composed of two immiscible liquids [1]. Coaxial electrospray has several advantages over other microencapsulation technologies including higher encapsulation efficiency and more uniform size In the electrospray phenomenon, a high distribution [2]. electrostatic field acting on the free surface of a liquid triggers an instability that transforms the meniscus into a conical shape, called a Taylor cone, that balances the capillary forces and the electrostatic pressure [3]. In the electrospray cone-jet mode, a jet of liquid is ejected from the apex of the cone due to the high electric fields present there; the jet breaks up into droplets. In coaxial electrospray, the liquid fed to the Taylor cone has a coaxial structure composed by an inner liquid and an outer liquid that are immiscible. The same coaxial structure is present in the emitted jet, which generates core-shell droplets when it breaks up: the core of the particle is made of the inner liquid, while the shell of the particle is made of the outer liquid.

The stability of a coaxial Taylor cone and its electrified compound jet strongly depend on the physical properties of the working fluids as well as the supplied flow rates and applied bias voltage. Scaling laws of the dependence of the emitted current and droplet size on the flow rates and the physical properties of the liquid while the emitter is operating in cone-jet mode have been reported [4]. Also, the shell thickness and outer diameter of the core-shell particles can be estimated as a function of the flow rates and the expressions for the inner and outer diameters of the jet [5].

In coaxial electrospray the liquid with the smaller electrical relaxation time is the driving liquid, and the liquid with the larger electrical relaxation time is the driven liquid. A very stable coaxial electrospray configuration entails having the core of the coaxial flow made of the driving liquid, while having the outer layer of the coaxial flow made of the driven liquid; in this case, the electrical stresses acting on the charged inner liquid-outer liquid interface are efficiently transmitted by viscosity to the bulk of the outer liquid, setting it into motion.

Additive manufacturing is a set of free-form fabrication

technologies that create solid objects using a computer-aided design (CAD) file as template. Additive manufacturing started as a visualization tool of mesoscaled objects, but recent developments in the resolution and capabilities of 3-D printing suggest that these manufacturing processes could address the complexity, three-dimensionality, and material processing capabilities of many microsystems [6]. Stereolithography (SLA) is an additive manufacturing process that creates solid objects from the photopolymerization of a resin using ultraviolet light. High-resolution SLA can be used to manufacture freeform microfluidics at a small fraction of the cost per device, infrastructure cost, and fabrication time of a typical silicon-based microfluidic system [7].

Herein, we report the first 3-D printed microfluidic devices that generate core-shell particles via electrohydrodynamic jetting. Unlike traditional, i.e., uniaxial, electrospray that has been investigated for over 100 years [8] and of which many MEMS implementations exist [9], coaxial electrospray was discovered in 2002 [1] and no microfabricated coaxial electrospray source had been reported due to the inherent three-dimensionality and complexity of its hydraulic system.

DESIGN

The 3-D printed microfluidic devices, made of an ABS-like material, have one or two 12 mm-tall internally-fed coaxial electrospray emitters that are fed by two 700 μ m-diameter, 37 mm long helical channels (Figure 1). The channels act as high hydraulic impedances that regulate and uniformize the inner and outer flows fed to the emitter. Each emitter spout is designed to produce a coaxial flow and to enhance the electric field on the liquid meniscus. The emitter tip has a diameter of 2.5 mm and both the inner and outer outlets of the spout have a hydraulic diameter of 700 μ m. Each emitter occupies a 9.6 mm² footprint.

Each device has a 7 mm by 24.25 mm by 24.25 mm frame with two liquid reservoirs with a capacity of 250 μ L each that feed the emitter(s). The frame includes four through-holes that can be used to clamp the device to a chuck with liquid feedthroughs via M4 screws. Between the emitters and the through-holes there are curved spill guards that protect the screws of any spilled liquid.



Figure 1: Left: schematic of a single-emitter coaxial electrospray source showing the helical feed channels, liquid reservoirs, and spill guards. Right: photograph of a single-emitter device next to a 0.3 mm-diameter mechanical pencil; the different colors of the liquids supplied to the device evidence the helical channels that feed the emitter nozzle.

Solid-State Sensors, Actuators and Microsystems Workshop Hilton Head Island, South Carolina, June 5-9, 2016

CHARACTERIZATION

The apparatus shown in Figure 2 was used to characterize the 3-D printed coaxial electrospray sources and to explore how the flow rates and bias voltage influence the stability of the microencapsulation process. Deionized water and sesame oil are loaded into 1 ml syringes controlled by a Dual Syringe 33 Harvard Pump. The chip is clamped to the aluminum chuck using screws and the liquid feedthroughs of the chuck are connected to the syringe pump. The screws are also used to integrate an extractor electrode, i.e., a laser-cut 250 µm-thick 304 stainless steel plate with apertures that line up with the axes of the coaxial electrospray emitters of the chip. The collector electrode is a 250 µm-thick 304 stainless steel plate. The aluminum chuck is grounded, a 4.2 kV to 6.4 kV negative bias voltage is applied to both extractor and collector electrodes, using a dual output Gamma High Voltage, 20W, 1 mA Power Supply. The emitted current was measured with a 485 Keithley picoammeter and recorded by a DS6000 Rigol oscilloscope, the picoammer was connected in series with a large resistor.



Figure 2: Schematic of experimental apparatus.



Figure 3: (a): compound droplet of deionized water covered by sesame oil at zero electrical field; (b): uniaxial electrospray of deionized water mixed with Rhodamine B; (c): coaxial electrospray of deionized water mixed with Rhodamine B (inner flow) and sesame oil (outer flow). In Figure 3(c) the emitted jet is visible.

Figure 3(a) shows a compound meniscus of deionized water (internal liquid) and sesame oil (external liquid) at the spout of a coaxial electrospray emitter that is not energized. The internal layer of the compound meniscus was illuminated with an ultraviolet lamp shining at an appropriate direction. If the negative bias voltage on the extractor electrode is set to about 5kV, the liquid meniscus triggers electrospray emission in cone-jet mode. In Figure 3(b) only the internal liquid was fed to the chip, resulting in uniaxial electrospray emission, while in Figure 3(c) both the internal liquid and the external liquid were supplied, resulting in coaxial electrospray emission. In both cases, the deionized water was dyed with Rhodamine B to more easily distinguish it from the sesame oil.

Coaxial electrospray modes

In electrospray different spraying modes can take place according to the magnitude of the electrostatic field and flow rate fed to the emitter. For a given flow rate, increasing the magnitude of the electrostatic field results in operating the emitter in the dripping mode, the cone-jet mode, or the multi-jet mode [10]. In the dripping mode, the bias voltage causes an electrostatic force on the meniscus that is smaller than the hydrodynamic forces, the surface tension forces, and gravity. Therefore, the generation of droplets in this regime is set by the balance between gravity and surface tension (i.e., the droplets fall down if the gravity is large enough to overcome the surface tension), although the droplets still transport charge when a non-zero bias voltage is applied; however, in the dripping mode no core-shell droplets can be generated. In the cone-jet mode the strength of the electric field is large enough to trigger the formation of the Taylor cone, which produces coreshell particles via the break up of the jet emitted by its apex. The cone-jet mode is very stable. Further increasing the bias voltage leads to emission instability; the multi-jet mode appears when more than one jet is emitted from the surface of meniscus. The surface of the meniscus could also become close to the outlet of the emitter, leading to pulsating and unstable jets.

The emitted current was measured to identify the coaxial electrospray modes. To validate this approach, the leakage current through the dielectric was first characterized using a dry chip while the negative bias voltage between the chip chuck and the extractor electrode was set at 5kV. From these measurements, an average leakage current equal to 6 nA was obtained, which is at least twenty times smaller than the current measurements obtained with chips fed by the working liquids.



Figure 4: Per-emitter current versus time for deionized water flow rates between 0.06 ml/hr and 0.21 ml/hr and a fixed sesame oil flow rate of 0.06 ml/hr. Steady cone-jet emission occurs when the deionized water flow rate is equal or larger than 0.15 ml/hr; below that value, dripping emission occurs.

In the wet experiments, the extractor bias voltage was set at around -5kV and the inner and outer flow rates were varied between 0.06 ml/hr and 0.30 ml/hr; the results of these experiments are shown in Figure 4. The dripping mode was observed for several flow rate combinations (red and black lines); for instance, when the flow rate of the sesame oil is 0.06 ml/hr, the dripping mode occurs when the water flow rate falls below 0.15 ml/hr. The dripping mode is identified by a seemingly random fluctuation in the emitted current between zero and about 150 nA, which is in striking contrast to the steadiness of the emitted current in the cone-jet mode (green and yellow curves). The emitted current in the cone-jet mode is very constant, even after many minutes of continued emission.

When the deionized water and sesame oil flow rates are set at 0.30 ml/hr and 0.10 ml/hr, respectively, and the extractor electrode is positioned 6.4 mm from the emitter spout(s), steady cone-jet mode is achieved for extractor bias voltages between -5.4 and -6.3 kV. Similarly, if the plate is positioned 4.5 mm from the emitter spout(s), a stable Taylor cone can be generated for the wider bias voltage range between -4.2 kV and -5.9 kV. However, if the extractor electrode is positioned 2.6 mm from the emitter spout(s), the range of bias voltages that generate a stable Taylor cone is -4.5 to -4.8 kV. It is important to mention that in these experiments the per-emitter current was always very steady and the same (100 ± 4 nA). Therefore, the extractor voltage and position of the extractor electrode seems to have no influence on the per-emitter current; however, the electrical field acting on the Taylor cone increases as the separation distance between the emitter spout and the extractor electrode decreases, which has a significant influence in the formation of the Taylor cone. In addition, for a fixed extractor electrode-to-emitter nozzle separation, increasing the oil flow rate requires to also increase the magnitude of the bias voltage to maintain a stable Taylor cone because the oil affects the electrical fields felt by the driving liquid.



Figure 5: Per-emitter current I versus deionized water flow rate Q when sesame oil's flow rate is 0.10 ml/hr.



Figure 6: Per-emitter current I versus sesame oil flow rate Q when deionized water's flow rate is 0.30 ml/hr.

Per-emitter current versus flow rate characteristics were also collected to investigate the influence of both flow rates in the formation of a stable coaxial cone-jet. The experiments show that the scaling law that governs standard, i.e., uniaxial, electrospray also applies to the coaxial electrospray. However, in the case of the coaxial electrospray emitter, the flow rate that influences the emitted current is only that of the driving liquid. Steady peremitter currents, which are associated with stable cone-jet formation, are proportional to the square root of the per-emitter flow rate of the driving liquid, -in this case deionized water (Figure 5); this is in agreement with the scaling laws reported in However, the per-emitter current is not the literature [4]. significantly affected by changes in the driven liquid flow rate -in this case sesame oil (Figure 6). The close similarity between the electrical behavior of the uniaxial electrospray and the coaxial electrospray is expected because the oil is very inefficient at transporting charge and for practical purposes it only supplies dielectric material that surrounds the conductive material, similar to operating a standard electrospray emitter in air.

Core-shell particle observation and measurement

Compound microdroplets were collected on a cover slide placed on top of the collector electrode. The separation distance between the collector electrode and the extractor electrode was varied between 6 mm and 20 mm, and the collector electrode was biased with voltages between -1 kV and -6 kV. In order to collect microdroplets, a cover glass was positioned carefully above the collector plate once that electrospray process was running steadily. Then, the collector voltage was turned on for 10 seconds, after which the syringe pump and both power supplies were switched off. After that, the cover glass was observed in an optical microscope. In these experiments the deionized water was dyed with Rhodamine B. After droplet deposition, the cover slide was flooded in water mixed with fluorescein, which greatly helped identify the structure of the compound droplets.

Collection of microdroplets on the slide was observed when the collector electrode-to-extractor electrode separation was at 6 mm and the collector bias voltage was set at -5.6 kV, i.e., until the magnitude of the bias voltage on the collector electrode is greater or equal to the bias voltage on the extractor plate; this is because the electrical field between the collector and extractor electrodes is close to zero (see Figure 7). In other words, when the collector electrode is biased at a less negative voltage than the extractor electrode, the microdroplets decelerate, which leads to collecting the microdroplets on other surfaces.



Figure 7: Axisymmetric finite element simulation of the electrical field (magnitude is shown) and electrical potential (contour lines) for a coaxial electrospray emitter. The chuck was grounded, both extractor and collector bias voltage were set at -5.6 kV. These conditions correspond to those of the experiment used for droplet collection.



Figure 8: (a): fluorescent image of core-shell microdroplets (red) flooded in water with fluorescein (green); (b): close-up of microdroplets with core made of deionized water with Rhodamine B coated by a sesame oil shell.



Figure 9: Statistical distribution of the outer diameter of the coreshell particles for different deionized water mixed with Rhodamine B – olive oil flow rates [ml/hr].



Figure 10: Per-emitter current from single- (green) and doubleemitter (blue) 3-D printed coaxial electrospray sources for various flow rates. Experiments 1, 2, 3, 4 and 5: sesame oil flow rate = 0.50 ml/hr, deionized water flow rate = 2.0, 1.75, 1.50, 1.25 and 1.00 ml/hr, respectively. Experiments 6, 7, and 8: deionized water flow rate = 0.40 ml/hr, sesame oil flow rate = 0.20, 0.15, and 0.10ml/hr, respectively. The double-emitter devices are assumed to operate uniformly, i.e., the per-emitter current and flow rate are equal to diving the total current and total flow rate by two.

The core-shell structure of the droplets collected on the slide is visible using fluorescent microscopy, as shown in Figure 8. In Figure 8(a), red microdroplets can be seen in a green background because the red-dyed cores of the particles are encapsulated in sesame oil, which refrains their mixing with the water dyed with fluorescein. In Figure 8(b), the red core of the compound droplets, as well as the translucent oil shell, are visible.

The diameter distribution of compound microdroplets produced with different combinations of driving liquid and driven liquid flow rates was characterized using metrology of images from an optical microscope (Figure 9). In each case, 500 droplets were measured to estimate the average and standard deviation of the outer droplet diameter. The experimental data demonstrate that the droplets' average outer diameter and standard deviation can be modulated by controlling the flow rates. In particular, if the flow rate of the driving liquid is significantly larger than that of the driven fluid, the distribution has associated a smaller standard deviation. In addition, smaller flow rates result in smaller compound droplets.

Figure 10 shows the per-emitter current from single- and double-emitter sources under various flow rate conditions; the results suggest that the emitters are operating uniformly.

CONCLUSION

We reported the first 3-D printed coaxial electrospray devices for generation of core-shell microparticles. Characterization of single- and double-emitter devices using deionized water as inner flow and sesame oil as outer flow demonstrate uniform emitter operation and generation of core-shell microparticles with narrow diameter size distribution. Current research efforts focus on achieving high-throughput core-shell microparticle generation through emitter multiplexing [7] combined with integration of electrodes via MEMS assembly technologies [11].

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SOLID STATE ELECTROCHEMICAL ALKALI SOURCES FOR COLD ATOM SENSING

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ABSTRACT

Alkali sources are used as getters, to coat thermionic emission electrodes and to provide alkali atoms for precision quantum sensors such as clocks, magnetometers, accelerometers and gyroscopes. Commercial alkali sources are electrically heated to 400 - 700°C, driving a chemical reaction which produces the alkali. These sources use high current, high power, operate at high temperatures, turn on and off slowly, and evolve impurities. We report here on solid state electrolytic sources for the production of cesium and rubidium at temperatures of 80°C to 170°C. The advantages of electrolytic sources over thermally driven sources are reduced current and power consumption, faster turn-on and turn-off, the ability to monitor the quantity of generated alkali by current integration, and reduced temperature of operation. Resistivity and activation energy of the solid ionic conductors was measured vs. temperature. Alkali vapor pressure was detected by optical absorption in a custom vacuum system. Strong alkali signals (comparable to commercial sources) were measured when current was passed through the samples. Reversing the polarity of the applied voltage results in ambient alkali absorption. These Solid State Ambipolar Alkali Sources (SSAAS) show potential as high purity, precision alkali sources for cold atom sensors where size, weight and power must be reduced.

INTRODUCTION

Alkali sources have been used for decades to coat thermionic emission electrodes and as vacuum getters. Recently, laserinterrogated atomic sensors such as highly accurate clocks [1], magnetometers [2] and inertial sensors [3] have been developed. The most accurate of these sensors operate in a high vacuum, using alkali atoms trapped by magneto-optical traps (MOT's) prior to performing an optical or microwave based measurement of time, magnetic field or acceleration. For these sensors, clouds of $\sim 10^7$ atoms are needed. The alkali partial-pressure must be maintained at an acceptable level (~ 10^{-8} to $< 10^{-9}$ Torr) over a potentially wide environmental temperature range. The vapor pressure of the pure metals are too high for a solid Cs or Rb source to be used. Maintaining the correct background pressure of alkali is further complicated by the kinetics of absorption, adsorption and desorption from the vacuum chamber walls, particularly with glass which can absorb substantial quantities of alkali [4]. An ideal source would be low-power, fast, well-controlled and could also act as a sink to regulate alkali vapor pressure.

Commercial alkali sources from SAES [5] and Alvatec [6] use thermally driven chemical reactions to liberate alkali, and may produce contaminant gasses as well. Typically 4-8 amps is used to resistively heat the device to its operating temperature of 400-700°C [7], producing unwanted light, heat and magnetic fields which can interfere with sensor operation.

It has long been known that alkali can be produced by solid state electrolysis of glass [8, 9]. Gong et al. [10] demonstrated the

formation of Cs vapor cells by building a CsBO₃ glass piece into a vapor cell wall, and driving Cs into the cell using a molten salt anode at 325°C. Later, Cs generation was demonstrated with CsBO₃ glass as an independent source (not part of a vapor cell wall) and using a solid metal anode at 170°C [11].

Chalcogenide glasses based on S, Se or Te doped with alkalihalides have been extensively developed for infra-red optics, and are known to have higher ionic mobility than most oxide glasses [12]. Chalcogenide glass is also less hygroscopic than oxide glasses with high alkali content. Na- β "-alumina is a ceramic phase of Al₂O₃ and Na with high ionic mobility, which can be substituted for other alkali through a high temperature diffusion process [13].

We report here for the first time chalcogenide (sulfide and selenide) glasses doped with Cs and Rb and Rb- β -alumina which produce high purity alkali by electrolysis when small currents (μ A-mA) are passed through them.

Figure 1 shows a cartoon of an electrochemical source, with Rb ions drifting from anode to cathode under a bias voltage. We have also demonstrated for the first time absorption of alkali by reversing the bias voltage of a Rb β -alumina source, allowing both emission and absorption of alkali with the same device. A silver anode provides charge compensating ions for the migrating Rb ions.



Figure 1. Cross-sectional diagram showing Rb ion migration through source from anode to cathode.

GLASS FABRICATION

Selenide glass (GeSe₂ + Ga₂Se₃) samples containing 30% or 40% CsI, and sulfide glass containing CsCl were produced at Iowa State University (ISU). Selenide glass with 25% or 30% RbI was produced at the University of Rennes. Rb-substituted β -alumina was obtained from Ionotec.

The glass samples from ISU were prepared from appropriate amounts of 99.99%+ purity constituent elements (Ge, Ga, Se, CsCl or CsI) then sealed in evacuated silica ampules. The silica glass ampules were placed in a rocking furnace and heated at 1°C/min to 850-950°C and then held for 12 hours. Different soaking temperatures were used to determine the optimum temperature. It was found that 950°C provided a high enough temperature to ensure complete reaction among the elements and yet not so high that vapor pressure would break the sealed glass tube. After soaking for 12 hours, the ampule was immediately transferred to a furnace that was

978-1-940470-02-3/HH2016/\$25©2016TRF DOI 10.31438/trf.hh2016.50 Solid-State Sensors, Actuators and Microsystems Workshop Hilton Head Island, South Carolina, June 5-9, 2016 preheated to the glass annealing temperature of 250 to 270°C for four hours.

ELECTRODING

Anode: The back anode needs to inject mobile ions into the glass to avoid building up space charge as the alkali ions migrate up towards the cathode. This has proved a problem in previous work. In [10] a molten NaNO₃ salt anode was used as a source of compensating ions. Developers of high temperature alkali ion guns [14] using ion-exchanged Cs-mordenite electrolyte found that current flow was limited by anodic processes causing oxidation of the host lattice. Space charge formation led to a rise in voltage necessary to drive current through their device.

It is well known that metals such as Ag and Cu form nonblocking electrodes which inject ions into glass and have high ionic mobility [15]. For this reason continuous Ag electrodes were chosen for our anodes. The use of solid ion-injecting electrodes prevents development of space charge regions or oxidation of the host lattice anions.



Cathode: Cathodes were applied by evaporation of Al through an etched Invar shadow mask, held in place by magnets. The role of the cathode is to supply electrons to neutralize the alkali ions migrating to the surface. To allow the alkali atoms to evaporate without being blocked by the metal fingers, it is desirable to make these fingers narrow and to cover a small fraction of the front surface. Figures 2, 3 and 5 shows samples after application of the cathode finger array. These samples are 10-14 mm diameter and 1-3 mm thick. The shadow mask has finger widths resulting in 160 μ m wide metal lines with 400 um spacing, or about 60% open area.



EXPERIMENTAL METHODS

A vacuum chamber was constructed from a 6 port stainless steel cube for the measurement of alkali generation. A mechanically backed turbo-pump was used for evacuation to the low 10^{-7} Torr range after bake-out. This pump could be valved off and an ion pump used to maintain vacuum.

A multi-pin electrical feedthrough to the chamber has connections for a small ceramic heater with built-in thermocouple, high voltage and ground to drive the solid state source, and driving a commercial source from SAES or Alvatec, used for demonstrating alkali absorption.



Figure 6. 25 mm X 25 mm heater, CsCl-sulfide glass sample and Be-Cu spring clip.

Alkali Measurement: A diode laser tuned to an optical transition of the alkali species of interest is passed through the chamber just above the sample to measure the concentration of each alkali [Cs] or [Rb]. To detect small absorption signals we used a lock-in detection method, the block diagram of which is shown in Figure 7. Phase modulation is applied using an electro-optic modulator (EOM) to put 2 GHz sidebands on laser light locked to an atomic transition. The EOM is driven almost to carrier suppression such that most of the optical power is in the sidebands. These sidebands are sufficiently detuned from the Doppler broadened atomic transition. An RF switch driven with a 32 kHz square wave gates between light on-resonance and (modulated) light off-resonance.



Figure 7. Electro-optical detection block diagram.

A lock-in amplifier referenced to the 10 kHz modulation provides the change in amplitude due to the absorption. The sensitivity of the measurement is currently limited by residual amplitude modulation of the EOM to Cs vapor densities of \sim 1 \times 10⁸/cm³. The detected alkali density is calibrated using standard alkali vapor cells (e.g. Thorlabs GC25075-CS).

CONDUCTIVITY MEASUREMENTS

The alkali ion conductivity vs. temperature was measured at ISU by small signal AC impedance techniques extrapolated to DC, after applying and baking silver paint electrodes on both sides. Results for 6 samples (5 material compositions) are shown in Figure 8. The β '' alumina has the highest conductivity. The Rb and Cs doped selenide glasses have a conductivity which depends strongly on the concentration of alkali [12], although activation energy for the various glass samples is similar at ~ 0.77 eV. High conductivity is an advantage as it allows operation at lower voltages and temperatures.

DC current-voltage vs. temperature measurements were made using evaporated Al finger electrodes on the front, and continuous silver electrodes on the back. Due to various effects including current crowding, space charge and electrode interfacial reactions, the ionic conductivity measured this way is typically 100X lower than the small signal AC measurements. A plot of current-voltage vs. temperature for a CsCI-sulfide glass is shown in Figure 9.



Figure 8. Log₁₀ conductivity (AC impedance technique) vs. 1000/*T(K) for 5 materials.*

ALKALI EMISSION

Samples were placed on the ceramic heater in the vacuum system, held in place by a Be-Cu spring which also connects electrically to the top finger array. The resonant laser was directed over the top surface of the glass sample and its absorption was measured to determine the alkali vapor concentration. Alkali density was calibrated by comparison with the absorption of standard Cs or Rb vapor cells maintained at a known temperature.

CsCl-Sulfide Glass: A sample of CsCl-GeSe₂-Ga₂Se₃ glass from ISU was metallized with Ag on the back and Al fingers on the front. Current-voltage curves with temperature as a parameter were measured, shown in Figure 9. The current roughly doubles for every 10°C increase in temperature.

Cs vapor emission from this sulfide glass as measured by laser absorption is shown in Figure 10, held at 130°C with a bias voltage of 1200 V and current of 5 μ A, cycled on and off twice. Cs losses are primarily to the chamber walls and (much less) to the pumps.



Figure 9. DC current-voltage-temperature plot for CsCl- Ge-Gasulfide glass shown in Figure 4.



Figure 10. Optical absorption signal for Cs emission from sulfide glass sample at 130 °C. Current through sample is 5 μ 4.

CsI-GeSe₂-Ga₂Se₃ Glass: A 40% CsI doped selenide glass supplied by ISU was electroded with Al fingers (cathode) and continuous Ag or Cu (anode). Strong Cs generation signals were measured after biasing to 1 kV at 170°C and 44-50 μ A (Figure 11).



Figure 11. Cs generation from CsI-GeSe₂-Ga₂Se₃ glass.

Kinetics of alkali absorption and desorption from the chamber walls largely determine the concentrations and rate of change of the measured alkali in vacuum. Curve fitting the decay time of the Cs concentration shown in (e.g. Figure 11) gave a value of 1.25 s regardless of whether the chamber is sealed or actively pumped, because at low pressure the chamber walls dominate the alkali partial pressure kinetics. Due to chamber wall effects, we do not yet know how fast the solid state sources can be turned on and off, but it is likely much faster than the present generation of thermally driven sources [16].

RbI GeSe₂-Ga₂Se₃ Glass: A RbI doped glass supplied by the University of Rennes was electroded in a similar manner to the previous samples (Al cathode fingers and continuous Ag back anode). Strong Rb absorption signals were seen when the glass was forward biased at 170° C and 1 kV, (Figure 12). Current through the sample increased from 80 μ A to 250 μ A at constant voltage, which is most likely due to sample self-heating. A constant current drive would provide improved stability compared to constant voltage.



Figure 12. Rb generation signal from 30% RbI doped selenide glass.

β" Alumina Ceramic: Figure 13 shows Rb emission from a Rb β" alumina sample biased at 50 volts and a current of 0.7 mA at 120°C. Calibration of the atom density was performed by comparison with a standard Rb filled ampule. The strength of the Rb signal from this source was comparable to that from a SAES Rb source driven at 5 amps. The higher mobility of Rb in the β" alumina allows this source to operate at reduced temperature and voltage.



Figure 13. Rb generation from Rb doped β '' alumina.

Alkali Absorption: By reverse biasing the device, a surface region depleted of alkali ions is created. This ion-depletion region

is capable of absorbing incident alkali atoms, ionizing them and driving ions into the bulk. To demonstrate absorption, a Rb SAES source was mounted in the chamber and run at 4.88 amps for several minutes to establish a constant background Rb pressure. A Rb doped β '' alumina source was then reverse biased at -130 V (-0.21 mA) and 130°C, creating a Rb depleted surface while the SAES source was left on. The measured Rb concentration was reduced by 30-50%, as shown in Figure 14. Considering the small size of the SSAAS device (~1.1cm²) compared to the chamber walls (> 280 cm²), this demonstrates remarkably efficient absorption.

The measured Rb concentration is an integral over the path of the laser through the vacuum system. At each point in the path, the vapor pressure is proportional to an average over the entire 4π steradians solid angle of the incident flux of alkali atoms emitted from the surrounding surfaces. The laser passes close to the surface of the SSAAS device, and for that part of the integral, the SSAAS device is almost half the solid angle.



Figure 14. SSAAS device reverse biased as absorber. Background level set by SAES source at 4.88 A. SSAAS absorber turned on and off several times is able to absorb 50% of the Rb produced by the SAES source.

CONCLUSIONS

We have demonstrated solid state electrochemical alkali sources which, when compared to commercial thermally driven chemical sources, require greatly reduced current and power, operate at lower temperatures (70-170°C, compared to 400-700°C), exhibit faster turn-on and turn-off, and can both source and sink alkali using direct current control. The output of alkali was demonstrated to be comparable in magnitude to commercial SAES alkali sources consuming 5 amps.

Cold atom clouds formed in Magneto-Optical Traps (MOT's) used in high precision atomic clocks, magnetometers and inertial sensors require only about 10^6 to 10^8 atoms in a typical measurement. Solid state electrochemical sources will reduce the power, current and temperature required to produce alkali atoms by orders of magnitude. As cold-atom sensors enter the marketplace they will benefit from the high purity, small size and low power offered by these novel solid state atom sources.

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STABILITY OF UV/OZONE TREATMENT OF PLASTICS FOR CAPILLARY-DRIVEN MICROFLUIDICS

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ABSTRACT

In this paper, we report on the long-term stability of UV/Ozone (UVO) treatment of plastics which is a simple method to generate hydrophilic surfaces for capillary-driven flows in microfluidic devices. Previous reports on the stability of UVO-treated plastics were limited to 4 weeks. Here, we present new findings on the long-term stability of UVO-treated plastics for up to 16 weeks and also demonstrate for the first time that the storage condition has a significant impact on the surface stability. In particular, we show that the hydrophobic recovery of some UVO-treated plastics can be inhibited by storing them in dehumidified or vacuum conditions, making them useful for applications requiring long-term storage.

INTRODUCTION

Microfluidics is a promising technology that has been applied to many biomedical applications including cell biology, pharmacology and medical diagnosis [1, 2]. These applications generally require precise control of liquids in a timely manner which involves the use of bulky or costly equipment. Alternatively, accurate displacement of microfluidic flows can be achieved using actuated on-chip micropumps [3, 4]. While micropumps can provide precise, configurable flow rates, they increase the complexity and costs associated with microfluidic devices. The realization of microfluidic platforms that are simple, portable and inexpensive requires alternative means for fluid transport without externally-actuated components. Towards this end, passive pumping is a promising approach that doesn't require any moving components and can greatly simplify device fabrication and operation.

Among passive pumping methods, one of the most common is capillary pumping which employs surface tension forces to 'pull' liquids through capillaries and microchannels. This approach also offers simplicity in controlling the flow rate which mainly depends on the liquid viscosity and geometry and surface properties of the channels [5, 6]. In particular, the most important parameter for generating capillary flow is the surface hydrophilicity. Therefore, by controlling the hydrophilicity of the channel surfaces, it is possible to design microfluidic systems capable of precise, autonomous flow rates.

Previous reports have demonstrated capillary flows in silica and glass devices [7-9], however, these materials require complicated micromachining methods and are difficult to dispose. Capillary flows in polydimethylsiloxane (PDMS) microchannels have also been demonstrated using chemical surface treatments [10], however, PDMS is gas permeable and incompatible with some chemicals. Thermoplastic polymers have been extensively investigated over the past decade as a suitable material for microfluidic devices due to their low cost, disposability, optical transparency and biocompatibility [11]. One major drawback of plastics is that they are mildly hydrophilic in their native form, limiting their use for capillary flows. The surface of plastics can be made more hydrophilic by various surface treatments. For example, hydrophilic chemicals have been grafted onto plastic surfaces [12]. However, these methods can involve long and complicated fabrication processes and/or require the use of expensive equipment. An alternative approach is to oxidize the surface via exposure to an oxygen (O₂) plasma [13]. However, the stability of oxidized surfaces is poor, especially for PDMS where the surface undergoes hydrophobic recovery within a few hours [14].

Recently, a UVO treatment was demonstrated for generating hydrophilic surfaces on cyclic olefin copolymer (COC) which remained stable for up to 4 weeks [15]. In addition to enhanced stability, surface oxidization can also achieve thermal bonding at low process temperatures by preventing bulking and deformation of microfluidic structures [16]. While UVO treatment is a promising approach to enhance the surface hydrophilicity of plastic, further work is needed to evaluate its stability for applications requiring long-term storage (> 4 weeks).

In this work, we studied the stability of UVO-treated plastics under different storage conditions for up to 16 weeks. We focused our efforts on poly(methyl methacrylate) (PMMA), polycarbonate (PC), and COC because they are the most attractive plastics for biomedical applications due to their excellent optical transparency, biocompatibility and manufacturability [17]. Surface characterization of UVO-treated and untreated plastic samples was performed using x-ray photoelectron spectroscopy (XPS) and contact angle measurements. PMMA microchannels were fabricated and tested to demonstrate the effectiveness of UVO treatment for generating capillary-driven flows.

MATERIALS AND METHODS Sample Preparation

PC and PMMA were purchased from McMaster-Carr (Elmhurst, IL) and COC was purchased from Zeon Chemicals (Louisville, KY). For stability studies, COC and PMMA were cut into 1 cm x 2 cm pieces using a CO_2 laser cutter (Universal Laser Systems, Scottsdale, AZ) and PC was cut using a band saw. UVO treatment was performed using a UVO cleaner (Novascan Technologies, Ames, IA). For stability studies, samples were treated for 20 min, 40 min, 60 min or 80 min at room temperature and stored in air, a desiccator or vacuum-sealed bags (Weston vacuum sealer, Strongsville, OH).

Contact Angle Measurements

Static contact angle measurements were carried out using a VCA-2000 video contact angle analysis system (AST Products Inc., Billerica, MA). 0.5 μ L droplets of distilled water were dispensed onto the samples using a pipette. Contact angle data was acquired at ambient conditions from three different samples at a minimum of three locations per sample. The data is plotted as the mean value for eight separate measurements with outliers removed. Fresh samples were used for each set of measurements.

XPS Analysis

XPS spectra were obtained using a Perkin Elmer Phi 5400 ESCA system (Physical Electronics, Chanhassen, MN) at pressures between 10^{-9} and 10^{-8} torr, pass energy of 29.35 eV, and a 45° take-off angle. Elemental composition were calculated from the relative intensities of the C 1s and O 1s peak areas obtained from the survey spectral after subtraction of a linear background.

Microchannel Fabrication

Microfluidic devices were fabricated by cutting microfluidic features in PMMA using a laser cutter, applying UVO treatment and bonding the pieces together using double-sided adhesive film (Adhesive Research, Glen Rock, PA). The devices consist of three layers, as shown in Fig. 1. The width, height and length of the channel are 150 μ m, 1.87 mm and 30 mm, respectively, and the diameter of the inlet and outlet is 2 mm.



Figure 1: Fabrication process flow for PMMA microchannels.

RESULTS AND DISCUSSION

Influence of UVO Treatment on Surface Hydrophilicity

Contact angle measurements were performed on PMMA, PC and COC samples to investigate the influence of UVO treatment time on surface hydrophilicity. The data is plotted as the relative change in the contact angle (CA) following UVO treatment:

Relative change in
$$CA = \frac{Treated CA - Non treated CA}{Non treated CA} \times 100\% (1)$$

As shown in Fig. 2, UVO-treated samples exhibit significant reductions in contact angle compared with untreated samples for all three plastics. The contact angle of COC and PC drop significantly by $60.6\% \pm 2.5\%$ and $70.1\% \pm 3.2\%$, respectively, after 20 min of treatment, whereas PMMA exhibits a less significant reduction $(31.0\% \pm 2.9\%)$. All three plastics experience further reductions in contact angle with longer treatment times. Among the three plastics, PC exhibits the largest reduction in contact angle compared with PMMA and COC: after 80 min of UVO treatment, the contact angle of PC is reduced by $87.8\% \pm 0.8\%$. In contrast, the contact angle of COC does not significantly change with longer treatment times. Our results for COC match well with those reported by Bhattacharyya et al [18], who also showed a negligible change in contact angle with treatment times > 20 min. For PMMA, a correlation between the treatment time and surface hydrophilicity was observed where longer times resulted in higher relative changes in contact angle. These results are consistent with those reported by Tsao et al [19] who also showed that the contact angle of PMMA is proportional to UVO treatment time. Based on this approach, the surface hydrophilicity of plastics can be tuned by simply adjusting the UVO treatment time.

UVO-induced surface oxidation is facilitated by 185 nm and 254 nm ultraviolet light which results in the removal of organic contaminants and non-destructive atomic layer etching. In the presence of oxygen, 185 nm light generates ozone while 254 nm light simultaneously excites organic molecules on the surface. The 254 nm light also generates free radicals on the surface that react with oxygen, thereby generating more oxygen-containing species [19]. Therefore, the degree of surface oxidation can be determined by investigating the surface chemistry. XPS measurements were performed on untreated and UVO-treated plastic samples to examine



Figure 2: Relative change in contact angle of PMMA, COC and PC after UVO treatment. Error bars represent the mean \pm standard deviation (SD) of eight measurements.

the changes in surface chemistry resulting from the treatment process (Table 1). XPS measurements immediately following 80 min of UVO treatment are shown in Table 1 (a), which reveal that UVOtreated samples exhibit significantly higher oxygen/carbon (O/C) ratios compared with untreated samples. This is due the introduction of additional oxygen species on the polymer surface, indicative of the formation of the hydrophilic oxide layer. This data supports our contact angle measurements (Fig. 2) which show that UVO treatment enhances surface hydrophilicity. XPS measurements of UVO-treated samples after 16 weeks under different storage conditions are shown in Table 1(b). The O/C ratios for all stored samples are lower than those of freshly UVO-treated samples. Samples stored in dehumidified conditions exhibit the largest O/C ratios compared with those stored in vacuum or air for all plastics. Interestingly, the O/C ratios for plastic samples stored in vacuum are only slightly higher than those for samples stored in air. These results suggest that under proper storage conditions, UVO-treated plastics can retain a robust oxide layer and maintain surface hydrophilicity.

Table 1: XPS analysis of the O/C ratio on the surface of UVOtreated and untreated plastics immediately following treatment (a) and after 16 weeks under different storage conditions (b).

(a)	PMMA	COC	РС
Non-treated	0.32	0.20	0.19
Treated	0.48	0.72	0.44
(b)	PMMA	COC	РС
Air	0.27	0.31	0.34
Dehumidified	0.41	0.38	0.38
Vacuum	0.34	0.34	0.36

Stability of UVO-Treated Surfaces

In addition to analyzing the surface chemistry, we investigated the stability of UVO-treated samples by performing static contact angle measurements for 16 weeks. Measurements were performed on samples that were treated for 80 min and stored under different conditions (vacuum, dehumidified and air). As shown in Fig. 3, the contact angle of all three plastics gradually increase over time as a result of hydrophobic recovery. Among the three storage conditions, samples stored in air exhibited the most substantial hydrophobic



Figure 3: Long-term contact angle measurements of UVO-treated PMMA, COC and PC under various storage conditions. Each data point represents the mean of eight measurements.

recovery. In particular, the contact angle of COC increased significantly after only 3 weeks whereas PC and PMMA were stable for up to 12 weeks and 16 week, respectively. Interestingly, the storage condition had a negligible impact on the stability of PMMA, which remained stable for the entire duration of this study (16 weeks). In contrast, the degree of hydrophobic recovery for COC and PC was greatly influenced by the storage condition. Specifically, the contact angle of PC stored in air increased by ~180% compared with dehumidified samples whereas vacuumstored samples increased by only ~40%. For COC, the contact angle of samples stored in air and vacuum increased by $\sim 160\%$ and $\sim 75\%$. respectively compared with dehumidified samples. These results are consistent with the XPS measurements presented in Table 1 which showed that the sample stored in air exhibited the lowest O/C ratio while the dehumidified sample exhibited the highest O/C ratio. Based on these collective results, we show that storing UVO-treated PC and COC samples in dehumidified and vacuum conditions can substantially reduce their hydrophobic recovery by preserving surface oxidation and minimizing surface contamination, thus improving overall surface stability.



Figure 4: Capillary flow in untreated (left) and 80 min UVO-treated (right) PMMA microchannels. Colored liquid is dispensed at the inlet of the chips using a pipette.

Effectiveness of UVO Treatment for Capillary Flow

demonstrate the effectiveness of UVO-treated То microchannels for capillary-driven flow, we performed flow experiments using PMMA microchannels with varying UVO treatment times. Experiments were also carried out using untreated PMMA microchannels for comparison. 40 µL of colored liquid was manually dispensed into the inlet of the microchannels using a pipette. As shown in Fig. 4, liquids quickly filled the 80 min-UVOtreated microchannel in < 4 sec while negligible flow was observed in the untreated microchannel. We also measured the volume flow rate for microchannels with various UVO treatment times (Fig. 5). From this plot, we observe that there is a clear correlation between the treatment time and the flow rate due to the enhanced surface hydrophilicity generated from longer UVO exposure. This method can be applied to more complex microfluidic networks to develop capillary-driven systems that offer enhanced automation for more simplified operation. These results also suggest that UVO-treated microchannels can remain effective in pumping liquids via capillary flow after prolonged periods of storage when properly stored in dehumidified or vacuum conditions.



Figure 5: Volume flow rate as a function of UVO treatment time for PMMA microchannels. Each data point represents the mean \pm SD of three measurements.

CONCLUSION

UVO treatment is a simple method for generating hydrophilic surfaces on plastic, making it particularly useful for capillary flowbased microfluidic applications. In this work, we showed that UVO treatment time has a significant impact on the surface hydrophilicity of PC, COC and PMMA where longer times resulted in greater hydrophilicity. XPS measurements revealed that the enhanced hydrophilicity in UVO-treated plastics was due to a higher oxygen content in the polymer surface. Long-term contact angle measurements showed that UVO-treated samples exhibit significant hydrophobic recovery when stored in air, especially for PC and COC, whereas surface stability could be maintained for up to 16 weeks when stored in dehumidified or vacuum conditions. Lastly, we demonstrated the effectiveness of UVO treatment on PMMA microchannels for generating capillary-driven flows. With further work, this approach can facilitate the development of low cost plastic microfluidic devices for important biomedical applications.

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STICTION AT HIGH TEMPERATURE (>250°C) IN ENCAPSULATED MEMS DEVICES FOR HARSH ENVIRONMENT APPLICATIONS

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ABSTRACT

We demonstrate the stiction properties of ultra-clean, waferscale, vacuum encapsulated MEMS devices at elevated temperatures from 30-325°C. A modest increase in the sidewall adhesion (stiction) force between the pure, single crystal silicon surfaces is observed as temperature is increased, but no fundamental change in behavior occurs. Repeated cycling at high temperature demonstrates that no additional degradation results from the high temperature conditions over thousands of contact impact cycles. This is indicative of the suitability of these encapsulated devices for harsh environment applications.

INTRODUCTION

Static surface adhesion (stiction) is a common problem and failure mode in MEMS/NEMS devices [1] due to the scaling of surface and volume forces. At the small length scales found in micro-sensors and devices, the magnitude of surface forces such as Van der Waals force, or capillary attraction can be of comparable magnitude to the mechanical, piezoelectric and electrostatic forces which permit the device to operate. The failures may occur either during fabrication (process stiction), or during use (in-use stiction). Harsh environment applications make additional demands on device design that can further complicate the problem posed by stiction.

As a result of the significant problem posed by stiction in MEMS devices, numerous methods of mitigating surface adhesion have been developed. By using strict design rules it is possible to design devices with sufficiently high restoring forces, or large gaps to prevent contact from occurring under any circumstances, but this often severely limits device performance [2]. Typical solutions to prevent device failure from adhesion during planned or unplanned surface contact include anti-stiction surface treatments [3], such as polymer self-assembled monolayers (SAMs) [4], and over travel stops to reduce contact area.

High temperature harsh environments present a particularly difficult challenge with regards to stiction. Typical anti-stiction SAMs are polymer based materials with limited thermal budgets. Decomposition of SAMs may begin at temperatures as low as 200°C [5], and few of the materials can withstand temperatures above 300°C for any extended period of time. In addition, critical stiction-related surface properties of silicon, such as roughness and asperity distribution can be modified by exposure to elevated temperatures for extended periods of time [6]. Additionally, harsh environments frequently involve vibration and shock events that make inadvertent surface contact more likely. Such environments include down-hole oil drill monitoring, and combustion engine measurement and control.

The epitaxial encapsulation process is natively hightemperature compatible due to the 1100°C encapsulation process step [7]. The completed device is finalized in an epitaxial furnace, so all aspects of the device are designed to withstand extreme temperatures. As a result, this process has unique implications regarding stiction. Use of polymer anti-stiction materials is impossible, and the sidewalls are smooth, pure silicon, but the environment is free of moisture and hydrogen bonding. Previous work has examined the stiction properties of encapsulated devices [8], and the possibility of mechanical anti-stiction solutions [9]. In addition, previous investigation has demonstrated devices fabricated in this process to be suitable for high temperature use in cases where the behavior is dominated by current flow through the contact [10]. Given the native high-temperature compatibility of these devices, they present an intriguing possibility for use in harsh environments, conditional upon successfully negligible stiction failure modes.

EXPERIMENT DESIGN

Fully encapsulated, electrostatic test structures were fabricated in the standard epitaxial process. The test structures were designed to have anti-stiction spring bump stops, guided by previously observed results [11]. These devices were mounted on polyimide printed circuit boards (PCBs) to allow for high temperature testing.

Device Design and Fabrication

The test structures were designed to allow for a precise and repeatable test of the surface adhesion properties under various conditions. In order to achieve this goal, the following specific features were prioritized in the design:

- Electrostatic pull-in actuation
- Capacitive position sensing
- Released mass, spring constant similar to typical inertial sensors
- Anti-stiction bump stops

A schematic of the final device design is shown in figure 1. Several variants of this device were fabricated with slightly different bump stop configuration, but the basic operation is identical.



Figure 1 - Schematic of test structure device design

The devices were fabricated on 40μ m heavily doped single crystal silicon (both n and p type), and encapsulated at the wafer level in a pure, native oxide-free environment. The encapsulation

978-1-940470-02-3/HH2016/\$25©2016TRF DOI 10.31438/trf.hh2016.52 Solid-State Sensors, Actuators and Microsystems Workshop Hilton Head Island, South Carolina, June 5-9, 2016 process which enables this environment was developed at Stanford University, in cooperation with Robert Bosch GmbH, and has been reported upon extensively in previous publications [7]. Over 100 devices were fabricated, with a yield of about ~75%.

Experimental Procedure

The finalized test structure dies were mounted and wirebonded directly to a high-temperature compatible, polyimide PCB. The PCB was mechanically mounted to an aluminum block heater. This setup, shown in figure 2, avoided the use of solder and most polymer materials, thus allowing the devices to be heated to temperatures in excess of 300°C during long-term testing. A platinum resistive thermometer was used to monitor the temperature, and for the proportional-integral feedback controller, which maintained a desired temperature within ± 0.15 °C. The maximum testing temperature of ~325°C was constrained by the PCB substrate material, but is still well within the range qualifying as a harsh environment.



Figure 2 - High-temperature, long-term test set up

An LCR meter with a DC bias tee was used to simultaneously actuate surface contact and measure the behavior. This quasi-static process allows the proof mass to be pulled in to make contact with the bump stop and released by sweeping the bias voltage. The position of the proof mass may be calculated by measuring the parallel plate capacitance. An example measurement is presented in figure 3. Some fraction of the pull-in/pull-out hysteresis is accounted for by the non-linear nature of the electrostatic force. The remainder of that hysteresis is contributed by the stiction force between the bump stop and the proof mass.

Previous investigation has shown that this quasi-static testing does not utilize the stored energy in the bump stop spring to promote release from stiction [11]. In addition, this test method forces the contact to occur for at least one second on each cycle. For these reasons, this testing does not provide a "best-case" measurement, and is in fact quite conservative. Allowing the spring bump stops to be utilized to their full potential has been shown to improve stiction performance by >50% [11].



Figure 3 - Capacitive measurement of electrostatic pull-in test for a typical device

RESULTS AND DISCUSSION

By repeating the basic pull-in test at a series of elevated temperatures we were able to observe the temperature dependence of stiction. The pull-in and pull-out voltage results shown in figure 4 demonstrate that the stiction force increases modestly at high temperature. Assuming the bump stop deflection is small, we may calculate the stiction force, using the method reported previously [11]. At ambient temperature the measured force was 9 μ N, and it increased to 19 μ N at 250°C.



Figure 4 - The pull-out voltage decreases at higher temperature, indicating that the stiction force is stronger.

The data in figure 4 is assembled from several separate temperature sweeps on a single device. This indicates that the temperature dependence of stiction is both repeatable and reversible. In addition, this same behavior was repeated for all the tested devices.

The most important noteworthy feature of this result is the

stable and smooth nature of the temperature dependence. As compared to devices utilizing a polymer anti-stiction coating, which may abruptly and irreversibly switch from relatively low stiction to much greater stiction, the epitaxially encapsulated devices display a monotonic, and approximately linear increase in stiction. In addition, the devices are not affected by repeated or extended exposure to high temperatures. This behavior imposes only simple design restrictions on devices intended for use in high temperature harsh environments.

The physical mechanism resulting in the increased stiction is likely to be the increased surface energy of silicon, which has been reported elsewhere [12] for bonding of silicon-on-insulator wafers. In addition, although 250°C still well below the brittle-to-ductile transition temperature of silicon [13], there may already be some slight softening of the silicon surfaces. This is additionally suggested by the slight decrease in pull in voltage, as the pull in voltage is entirely determined by the material properties and geometry of the device. It is important to remark that the larger change in observed stiction force cannot be accounted for solely by the reduced mechanical restoring force brought about by the softening of the suspension springs, as this is a much smaller change.

Repeated Cycle Testing

To ensure the results reported above are truly repeatable (and may thus form the basis for future design guidelines), it was necessary to perform the pull-in experiment repeatedly over hundreds or thousands of cycles at a constant high temperature. Subjecting the same test device from figure 4 to over 5,000 contact cycles produced the result shown in figure 5. The device continued to operate normally at the conclusion of the test, and the number of cycles was constrained only by the time required to perform the testing.



Figure 5 - Results of repeated stiction test cycles at 250°C, collected over 3 hours. The measurement was collected every 20 contact cycles for the first thousand cycles, and then with decreasing frequency for the remainder of the test.

The repeated cycling clearly does not cause significant degradation in the stiction behavior of the device. In addition, the non-contact behavior of the device does not change, as demonstrated by the constant pull-in voltage. Further, the data indicates that there is some initial "break-in" stiction force, which is reduced by the repeated cycling at elevated temperature. Before cycling, the stiction force was measured to be 19 μ N, and after 5000 contact cycles, it was reduced to 7 μ N. This same trend is reproduced across several similar devices with different spring bump stop variants.

There are several possible mechanisms for this improvement. A reduction in surface energy, or decrease in contact area could come about during the impacts, and the addition of more thermal energy at the contact could exaggerate these effects. This data alone, however, is insufficient to form a strong conclusion. Further investigation of the surface topology and bonding properties is required to definitively determine the mechanism.

Though the mechanism for the reduction in stiction force seen in figure 5 is not directly observable from this data, it is clear that the modification to the surface adhesion properties is permanent. After cycling, the devices were cooled, and allowed to rest for several days. The original temperature sweep experiment was subsequently repeated for several devices, with the result shown in figure 6.



Figure 6 - Temperature dependence of stiction before and after high temperature cycling for devices A (upper) and B (lower), with slightly different bump stops

The improvement caused by the high temperature cycling process is seen to persist during subsequent testing once the device has cooled, and in additional high temperature sweeps. The base stiction force at ambient temperature is substantially reduced as a result of the cycling, and at 250°C, the stiction force was seen to decrease by as much as 55%. Repeating the measurement of stiction force at ambient temperature three weeks later revealed no change, further indicating that the modifications are permanent.

CONCLUSIONS

High temperature harsh environments present a unique series of challenges to reliable MEMS device design. Many of the basic materials used to construct MEMS devices, such as silicon and silicon dioxide, have high melting temperatures and are relatively insensitive to changes in temperature, which makes them promising candidates for this application. Unfortunately, nearly all of the self-assembled-monolayers and some of the chemical getters used to combat stiction are completely incompatible with high temperatures.

The wafer scale encapsulation process is natively compatible with high temperatures, as a result of the 1100°C encapsulation step. Since typical anti-stiction methods are unavailable, alternative, mechanical anti-stiction bump stops were previously developed. In this work we demonstrate that these anti-stiction bump stops perform well at high temperature. Though the effective stiction force does increase modestly with increasing temperature, the force remains manageable, and this increase is both predictable and reversible. Further, since no destructive processes occur at elevated temperature, the stiction performance does not degrade over time, and it appears that devices may be held at high temperature indefinitely with no negative consequence. Repeated contact cycles at high temperature do not damage the anti-stiction performance, and in fact may serve to reduce the stiction forces. These results demonstrate the suitability of epitaxially encapsulated MEMS devices for use in high temperature harsh environments.

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THE MEMS HAMMER, A TOOL TO STUDY MICROFRACTURE

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ABSTRACT

A MEMS actuator, dubbed the MEMS hammer, capable of storing and rapidly releasing mechanical energy has been designed, built and tested. The hammer is fabricated using a single mask silicon-on-insulator (SOI) process. These devices have been used to study fracture in both lateral and vertical regimes. The lateral tests have shown excellent agreement with shear fracture theory. Using either a mechanical or an electrostatic latching mechanism, the hammers are capable of storing energies up to 3.3μ J. The hammers have been shown to displace up to 36μ m, exert a maximum force of 240 mN, move at speeds exceeding 50m/s, and deliver at least 330 mW of mechanical power.

INTRODUCTION

The goal of this research is to create a microactuator capable of fracturing barriers made of silicon and silicon dioxide. At its core, the MEMS hammer is a device capable of storing and releasing mechanical energy. This system could be used for applications ranging from jumping microrobots [1] to needle-free delivery of drugs and vaccines [6]. In this work, the MEMS hammer is used as a tool to study fracture at the microscale. Additionally, we show promising steps towards creating a self-destructing sensor.

MEMS devices are commonly used in energy harvesting applications to transduce mechanical energy into electrical energy, often using piezoelectric materials [11]. However, limited work has been done at the microscale to store energy in a mechanical state. One way to store energy mechanically is in tension in a beam. The maximum energy stored in a beam of length *l*, cross-sectional area *A*, and Young's Modulus E is given by the following equation where ϵ_{max} is the maximum strain the material can undergo before fracture

$$E_{max} = \frac{1}{2} F_{max} x_{max} = \frac{1}{2} A l E \epsilon_{max}^2$$
(1)

Bergbreiter *et al* [1,2] have successfully stored and released 100uJ of mechanical energy using polydimethylsiloxane (PDMS) as their energy storage medium. These devices were designed to work with micromechanical motors which have an output force of 10mN. This relatively small force was the driving factor for using an elastomer based energy storage system. To store large energies with a small applied force, large displacements had to be achievable.

Rogers *et al* [3] created a mechanical energy storage and rapid release system in polysilicon using Sandia's SUMMIT-V process. This device was capable of using microfabricated electrostatic motors coupled to a 20,000:1 gear reduction ratio to launch a projectile with 19nJ of energy.

Both of the abovementioned systems were created with the intent of using a MEMS motor to store the initial mechanical energy, and the designs were constrained accordingly. These motors have an inherently low force output, so the researchers were forced to use a large mechanical advantage or a material with a low Young's modulus. In this research, the initial mechanical energy is stored in the device in a post fabrication assembly step, allowing us the freedom to choose a simple design and a material more suitable for microfabrication.

DESIGN

The MEMS hammer is fabricated in a single mask SOI process

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Figure 1: The layout of a MEMS hammer capable of storing $0.8\mu J$ of energy.

with a 40μ m device layer, a 2μ m buried oxide, and a 500μ m handle wafer. The MEMS hammer, shown in Figure 1, is composed of three main components: a cylindrical impactor, energy storing beams and a latching mechanism. In standard operation, the lever arm is pushed towards the right using a probe tip. As the lever arm rotates about the pin, it catches the hammer and starts loading the beams. The mechanical latch is moved to the side to allow the lever arm to pass by. Finally, the mechanical latch is moved back into its resting position and the hammer is latched in a high energy state. A latched hammer can be seen in Figure 3b, left.

To successfully build a device capable of fracturing silicon, it is crucial to identify and reinforce all load bearing features in the design. The beams are the main energy storing elements in this device; without filleting the interface between these springs and the anchoring silicon, they will fail at deflections of less than $15\mu m$, as opposed to $40\mu m$ with the fillets.

Additionally, the pin must be able to withstand the maximum force of the springs, which can be up to 240mN. The pin is anchored to the substrate via the buried oxide layer, and should theoretically fail when the shear stress induced by the hammer exceeds the fracture stress of the oxide. The literature on fracture stress of thinfilm silicon dioxide varies from 0.77 MPa [12] to 364 MPa [13]. In earlier versions of the MEMS hammer, a fracture stress of 364 MPa was used to design these pins. Every pin tested failed catastrophically. It was determined that a pin of radius 46µm can withstand forces up to ~300mN, putting the estimated fracture stress of our oxide at 54 MPa. It is important to make this pin radius as small as possible to keep the force required to latch the hammer low. The distance from the center of the pin to the end of the lever arm divided by the distance from the center of the pin to the hammer is roughly equal to the mechanical advantage scale factor seen by the mechanical and electrostatic latches. That is to say, the smaller the pin radius is, the easier it is for these latches to restrain the hammer. Therefore, the pin radius was optimized to allow for ease of latching while still being robust enough to withstand the high forces from the hammer.

Lastly, the piece of the lever arm that catches and loads the hammer must be designed carefully. The first iterations of the hammer used square etch holes. These stress concentrations caused the lever arm to fracture at its contact point with the hammer. In the current design, this was mitigated by moving to circular etch holes.

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Figure 2: MEMS hammer force vs displacement data. The empirical force vs displacement data is fit to the theoretical curve given by equation 5 and is in agreement with Coventor FEA.

Nonlinear Beam Model

In many MEMS applications, small displacement approximations are made to simplify calculations. These approximations are valid if the angle of beam deflection is small, such that the small angle approximations hold [4]. In this small displacement regime, the force displacement relationship of a centrally loaded fixed-fixed beam can be described using standard Euler-Bernoulli beam theory:

$$F(x) = 16 \frac{Ew^{3}t}{l^{3}}x$$
 (2)

This equation accounts for the bending strain energy stored in the beam. The MEMS hammer design uses beams which are 1000μ m long and deflect more than 35μ m at their center. The angles reached are large enough that nonlinear beam theory must be used, which adds a third order term corresponding to the axial stretching of the beam. A simple derivation of this non-linear term is done by assuming the springs stretch linearly, like a rubber band. By ignoring the boundary conditions at the fixed and guided edges, the derivation becomes a simple geometry problem. The non-linear term can be shown to be:

$$F_{stretch}(x) = 8\frac{Ewt}{l^3}x^3 \tag{4}$$

Combining this with the linear force-displacement relation given by equation 2, we arrive at the following equation which approximates the force-displacement relation of a fixed-fixed beam

$$F(x) = 16\frac{Ew^3t}{l^3}x + 8\frac{Ewt}{l^3}x^3$$
(5)

This equation corresponds to the force output for a single beam. In most hammer designs, multiple beams are used in parallel to increase the maximum force output by the device. This manifests itself in equation 5 with the addition of a factor *N*, equal to the total number of beams, in front of both terms. The integral under this force curve is equal to the energy stored by the hammer.

The energy storing characteristics of the beams used in this work were calibrated with a Dage 4000 multipurpose bondtester. This tool applies displacement at a fixed rate and measures the resulting reaction force. Figure 2 shows this data plotted against a finite element analysis (FEA) performed using Coventor. The data from the bondtester was then fit to equation 5 to determine how closely this simplified beam model describes our system. The coefficients of the best fit curve are 24.3 and 7.4 for the linear term and the third order term respectively. We see excellent agreement with our theory for the non-linear stretching term, and fairly poor agreement with the linear bending term. This is in part due to the inability of our bondtester to accurately measure forces of less than \sim 5mN. Figure 2 shows that not a single data point was acquired until the beams had displaced 12µm, well outside the linear range of these beams. Additionally, the critical region of operation is at high displacements where the most energy is stored. In this region, we see less than 5% deviation from theory and simulation.

MICROFRACTURE

Once the energy storage and force output of the hammer are characterized, the device can be used to study microfracture. By fabricating the hammer in front of fracturable structures, we can study in-plane lateral fracture. This lateral fracture can be described by a simple shear model or a Hertzian model. With the addition of one more lithography step, we create individual hammer chiplets, shown in Figure 4, top. These chiplets allow us to study out of plane vertical fracture, and open up the possibility of interfacing the MEMS hammer with non-microfabricated structures.



Figure 3a: A layout representation of a lateral barrier with two triangular stress concentrations. The width of the semi-circular hammer head is 40µm.



Figure 3b: A mechanically latched MEMS hammer. The mechanical latch restrains the hammer [left], until the latch is released allowing the hammer and lever arm to accelerate towards their respective barriers. Close up views of the fractured barriers for the hammer [top] and lever arm [bottom] are shown on the right.

Lateral Shear Fracture

In the lateral regime, hammers with varying stored energy are used to determine the minimum energy necessary to fracture a given barrier. Figure 3b shows that each hammer contains two identical barriers, shown in the dashed boxes, that can potentially be fractured by the device. A zoomed in picture of the barrier before fracture is shown in Figure 3a. The original intent was to use the hammer itself to fracture these barriers, however once the latching mechanism was added to the design, it became apparent that the lever arm absorbs more of the stored energy than the hammer. This is explained by noting the relative distance to the pivot point of the hammer versus the end of the lever arm. This 10x difference in radius turns into a 100x difference in kinetic energy, assuming the masses are identical. The hammer is roughly 10x as massive as the end of the lever arm, so the lever arm winds up with about 10x the kinetic energy as the hammer.

The energy stored in either the hammer or the lever arm must be greater than some critical value for the barrier to fracture. A simple shear force model states that as soon as the applied force causes the induced stress in the material to exceed the fracture stress, the barrier will fail. The energy that caused this fracture event is equal to the critical force times the displacement, where the critical force is equal to the fracture stress (1.3 GPa [5]), times the crosssectional area A

$$E_{shear} = F_{crit} x_{max} = \sigma_{max} A x_{max} \tag{6}$$

Lateral Hertzian Fracture

Shear theory is not the only describing physics for this lateral fracture mode. Hertzian fracture, first described by Hertz in 1881 [7], is the phenomenon in which a cone of material is ejected during a fracture event. This fracture pattern is most commonly seen in brittle amorphous materials, though it has been studied in crystalline materials as well [9].

From [8,9], the Hertzian fracture criterion is described by the following equation, where E, k, and v are material properties, and r is the radius of the impactor:

$$E_{hertzian} = \frac{2}{5} \left[\frac{2\pi \sigma_{max}}{1 - 2\nu} \right]^5 \left[\frac{4k}{3E} \right]^4 r^3 \tag{7}$$

In the above equation we see the characteristic dependence on impactor radius; small-radius impactors should require less energy to produce Hertzian fracture than large-radius impactors.

Vertical Fracture

Lateral fracture is advantageous due to its simplicity in layout and ease of testing, however it limits the potential applications of the hammer. The barrier test structures must be fabricated directly in front of the hammer, and typically from the same material. With the addition of a second lithography and etch step, we can create more useful barrier structures as well as released hammer chiplets.

The reinforced membranes, shown in Figure 4, middle bottom, are simple to fabricate and straightforward to model. The structure is made of a thin circular oxide membrane with a 40 μ m reinforcing silicon plate at the center. We are interested in the applied force and displacement required to fracture these reinforced membranes. From Roark [10], the critical force and critical displacement required to fracture this membrane follow these proportionalities, where *a* and *b* are the outer and inner radii of the membrane respectively, and *t* is the oxide thickness:

$$F_{crit} \propto \frac{t^2 b}{a} \tag{8}$$
$$dy_{crit} \propto \frac{a^2}{t} \tag{9}$$

Relations 8 and 9 help provide intuition for reinforced membrane fracture. The full equations are plotted in Figure 5; each curve represents a plate of different radius, swept from a 1 μ m oxide at the left to a 20 μ m oxide on the right. The required forces and displacements to fracture these membranes are well within the capabilities of the MEMS hammer. The black curve on this plot shows the maximum forces and displacements achievable by one of our hammers. The X's on this plot represent three different reinforced membranes that have been successfully fractured by our hammers.



Figure 4: A $2x2mm^2$ hammer chiplet before oxide release as fabricated [top] and in cross section [middle top]. A 600 μ m reinforced circular membrane as fabricated [middle bottom] and in cross-section [bottom].



Figure 5: Reinforced circular membrane fracture theory. Each curve holds fixed the outer radius, a, while sweeping the oxide thickness, t, from $1\mu m$ to $20\mu m$. The black curve shows maximum forces and displacements achievable by the MEMS hammer. Black X's indicate membranes that have been fractured by hammers in testing.

RESULTS

Arrays of hammers were fabricated with stored energies from 0.2μ J to 3.3μ J and cylindrical impactors of 20μ m, 40μ m, and 60μ m in radius. Lateral fracture studies were performed with these arrays at a probe station. Hammers were latched into place and released to test which barriers they were capable of fracturing.



Figure 6: Lateral fracture characteristics. The superimposed Hertzian and Shear Theory lines show the minimum energy required to produce fracture in each of those regimes.

Figure 6 shows the results of the lateral fracture experiments. The two curves on the plot show the minimum energy required by the impactor to induce fracture under the two fracture theories, shear and Hertzian. It is clear from the data that this system is well described by a simple shear force analysis. The threshold for fracture was not a function of the impactor radius, as would be the case in Hertzian fracture. However, in barriers with no stress concentrations, as opposed to those with stress concentrations seen in Figure 3a, we start to see something akin to Hertzian fracture. Figure 8 shows a barrier after a 3.3μ J impact from a 60 μ m radius lever arm. The fracture pattern on the opposite side of the barrier looks like it could be conical in form. More experiments will need to be run to determine if this fracture was in fact Hertzian, but this is a promising first result.

In the vertical fracture mode, the most robust membranes we fabricated only required an energy of 0.1μ J to fracture. Every hammer we tested with these membranes fractured them easily. The weakest hammer used on the membranes was 0.8μ J. The test setup is shown in Figure 7. A latched MEMS hammer chiplet is picked up with tweezers and rotated until the direction of actuation is normal to the membrane surface. It should be noted that once latched, these hammers are very secure. They remained latched in place even when dropped to the floor (an all too common event during testing). The hammer is then fixed in place using a UV curable epoxy. The mechanical latch is disengaged using a probe tip and the hammer accelerates forward, breaking through the reinforced oxide membrane.

When designing an energy storage device, it is important to keep in mind the maximum theoretical energy one could store with that material. Equation 1 gives the maximum energy that can be stored in a beam. By dividing this value by the volume of that beam, and assuming the maximum strain is 1%, we arrive at the maximum mechanical energy possibly stored in silicon of 8 mJ/mm³. Using this figure, the total possible energy our beams could store would be 1.6μ J. Each beam actually stores 0.21μ J, just over 13% of the maximum possible energy that could be stored in this volume of silicon. When these beams are arrayed together to increase the stored energy, the gaps between adjacent springs are roughly the

width of the springs, dropping the total energy storage figure to 6.5% of the theoretical maximum. After including the mass of the hammer, latch, and anchoring structures, this figure plummets to less than 1%. However, these structures have not been optimized for high energy densities, and with a few simple layout modifications this number could easily be pushed to greater than 20% of the theoretical maximum.





Figure 7: [Top] Cartoon version of hammer chiplet interfaced with reinforced circular membrane. Fabricated hammer and membrane before fracture [bottom left] and after fracture [bottom right].



Figure 8: A potential Hertzian fracture cone. Circular etch holes are $4\mu m$ in diameter.

ELECTROSTATIC LATCHING

The use of a mechanical latch to release stored energy prohibits the MEMS hammer's use in any application outside of a lab with a probe station. To address this, we designed and tested an electrostatic latch, seen in Figure 9. This electromechanical latch relies on a relatively weak electrostatic attraction between two plates and amplifies it through one or more mechanical advantage stages. This electrostatic force is governed by the following equation, where V is the applied voltage across the plates, ϵ_0 is the vacuum permittivity, A is the overlap area, and d is the gap between the plates:

$$F_{es} = \frac{1}{2} \epsilon_0 V^2 \frac{A}{d^2}$$
(10)



Figure 9: Single stage electrostatically latched hammer.

This electrostatic force is then multiplied by the mechanical advantage factor mentioned earlier. This mechanical advantage factor is equal to half the distance from the pin to the tip of the lever arm divided by the distance from the pin to the hammer. The factor of 0.5 shows up because this electrostatic force is distributed along the entire length of the of the lever arm. To first order, if there are multiple stages of the electrostatic latch, as can be seen in Figure 10, each stage contributes a mechanical advantage factor. The mechanical advantage factor in our system is roughly 5, so we see an enhancement of 5 in the single stage and an enhancement of 125 in the three stage latch. Using this theory, the single stage latch should require 60 V.

Figure 9 shows a very weak hammer which can store roughly 0.05μ J of mechanical energy. A voltage of approximately 160 V was needed to close this latch. The three stage latch is shown in Figure 10. This hammer stores 3.3μ J of energy, and the actual voltage required to keep the latch closed was 120 V. Both of these devices required roughly twice the theoretical voltage to remain latched. This deviation from theory may be due to an error in the gap, which was designed to be 0.25μ m but was not measured. Adding more stages than this, we believe we can design an electrostatic latch that works on as little as 5 V.

CONCLUSION AND FUTURE WORK

We have developed and tested a MEMS device capable of storing and rapidly releasing up to $3.3 \,\mu J$ of energy. After simulation of the dynamics of this system, it has been shown that the device releases its energy in 10µs. This means the hammer can deliver in excess of 330mW of mechanical power. The hammer has been used to study fracture in both lateral and vertical regimes. The fracture of the lateral barriers was shown to be well described with a shear force model. Electrostatic latches have also been designed and verified.

The MEMS hammer is a versatile device, with target applications spanning multiple fields. This project is funded under the DARPA Vanishing Programmable Resources program. The goal of which is to create a sensor capable of complete and irreversible self-destruction. We propose using the MEMS hammer to fracture a reinforced circular membrane which covers a cavity of xenon difluoride. This powerful silicon etchant would sit dormant in an inert environment until the time comes for the sensor to destroy itself. At this point, the MEMS hammer triggers, the membrane is fractured, and the xenon difluoride is free to sublimate and attack any silicon it contacts. One additional application we are pursuing is developing a system to convert the stored mechanical energy of the hammer into electrical energy.

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Figure 10: A three stage electrostatically latched MEMS hammer. Device too large to fit in field of view; figure stitched together from multiple images. Lever arm shadows are caused by an optional oxidation step before releasing the structures.

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TIME-DEPENDENT GATING IN NANOFLUIDIC CHANNELS FOR ACTIVE ELECTRIC DOUBLE LAYER MODULATION FOR ION TRANSPORT CONTROL

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ABSTRACT

A nanofluidic field effect device was experimentally demonstrated with DC and time-dependent gated transport. Time-dependent transport was shown with sine and square waveforms over two orders of magnitude frequency range (0.1–10 Hz) over four specific electrolyte concentrations in the 1×10^{-4} – 1×10^{-1} M range. Time dependent transport was found to have a higher degree of current modulation and a higher amount of total charge transfer through the nanofluidic channels compared to DC gating. An equivalent circuit model was developed to show the RC time constants for the PDMS, Stern, and diffuse layers which are hypothesized to be excited at the frequencies used in this experimental evaluation leading to enhanced ionic transport.

INTRODUCTION

Field effect nanofluidic devices (FENDs), geometrically analogous to solid-state field effect transistors, have been reported as diodes, rectifiers, and switches [1-3]. We report a FEND through a systematic study of sine and square wave gate modulation where, for the first time, time-dependent gating allows for active control over the electric double laver (EDL) for significant enhancement in measured current compared to purely DC gating at similar gate voltages. Time-dependent gating was selected for investigation the because introduction of time dependent signals are thought to require lower driving voltages than the DC counterpart [4] for equivalent device performance in microchannels. Moreover, we hypothesize that the active EDL modulation will permit enhanced total charge transfer through the nanochannels when applied frequencies are of similar magnitude to the native RC time constants for the EDL layers suggesting new technology for better pumping and ionic flow control. Technology development interest in FENDs arises from the operational length scales (sub-20 nm) at the level of fundamental biology with potential applications in water desalination [5], drug delivery [5], and DNA sequencing [2].

AC Electroosmosis (ACEO [6]) in microfluidics, relies on generating an effective electric field at a lower voltage than an equivalent DC signal by using an AC amplitude and frequency. The lower voltages limit faradaic reactions such as electrolysis and reduce Joule heating in microchannels. Moreover, as ACEO became better understood and the new name induced charge electroosmotic flow (ICEO) was proposed [7], it became evident that AC signals provides a component of the electric field arising from the induced charge to generate a body force on the ions and fluid. These ideas helped developed travelling wave electroosmotic pumps and separators [8, 9] which have been useful in exploiting the non-linear electrokinetics in microfluidics due to use of AC signals.

It is worth noting that when a time-varying (i.e., AC) signal is used as a gate potential, at low frequencies (and hence the DC limit) nearly all the potential drop occurs across the EDL. In microfluidics, since most devices operate in a thin EDL regime ($\kappa h >> 1$; where κ is the inverse Debye length and h is the channel half-height) the transverse component of the electric field due to the AC potential at low frequencies (1-10 Hz) is too small to generate an appreciable body force and cause motion. At higher frequencies (10^3-10^5 Hz) , [10, 11] ACEO occurs and with increasing frequencies approaching the charge relaxation time limits the stable formation of the EDL. With a disrupted EDL, the space charge in the fluid near the wall is insufficient to generally induce net fluid motion [12]. Therefore, in microfluidics the 'sweet spot' for operating ACEO is in few kHz frequency ranges at voltages in the few V range (<10 V). [10, 11] Nanofluidics presents a different aspect and provides a new avenue to exploit surface charge for a new generation of fluidic platforms as opposed to the traditionally studies ACEO flow seen in microchannels.

At the nanoscale, with critical device dimensions below 100 nm (here at 16 nm), $\kappa h \leq 1$ or $\kappa h \ll 1$, depending on electrolyte concentrations. Under such conditions, a significant portion of the nanochannel is occupied by the EDL. [13, 14] Therefore, at low frequencies, it is expected that an AC signal cause ion and fluid motion leading to quantifiable changes in measured current. Therefore, not only do we expect a much higher modulation of the field effect compared to a microchannel, we also expect a much broader range of frequencies to manipulate charge and mass transfer through the nanochannels while gaining in reduced joule heating and faradaic reactions, even with a single gate electrode instead of the array of gate electrodes seen in microchannels.

In microchannels, where almost all experimental work examining AC driven flow has been focused to date, direct influence of wall surface charge is limited in controlling bulk fluid flow. However, the use of multiple electrodes with high gating voltages (\sim 10–100 V) for velocity control [6] led to development of AC electroosmotic pumps. Additionally, a velocity dependence on frequency has been experimentally shown in microfluidic channels [10]. Yet, for nanochannels, frequency dependent gating is nearly non-existent with only two other limited experimental studies on frequency dependent fluid velocity [4, 15] and our previous report on measured current dependence on frequency (0.1-0.5 Hz) for a sine waveform and concentration. [16]

METHODS

Here, for the first time, we systematically investigated over a 2 orders of magnitude frequency range (0.1-10 Hz) for both sine and square gating, a RMS matched comparison against DC gating for aqueous KCl $(1x10^{-5} - 0.1 \text{ M})$ in 16 nm deep nanochannels. The FEND comprises a bank of three 2.5 mm long, 30 µm wide, and 16 nm deep nanochannels connected to two microfluidic channel reservoirs 3 cm long, 50 µm wide, and 8 µm deep. The channel network was wet-etched into a borosilicate substrate with a gold gate electrode embedded in the nanochannel 'roof' and insulated fluidically and electrically by a polydimethylsiloxane (PDMS) layer (Figure 1). The entire device assembly was bonded with an O₂ plasma bonding process. Fabrication methods for these long, ultra-low aspect ratio (ULAR) nanochannels has been reported by us previously [3, 16]. Channels were filled with aqueous KCl (1x10⁻⁵-0.1 M) at pH 7±0.02. Independent voltage sources were used to generate the axial (V_a) and gate (V_g)

978-1-940470-02-3/HH2016/\$25©2016TRF DOI 10.31438/trf.hh2016.54 Solid-State Sensors, Actuators and Microsystems Workshop Hilton Head Island, South Carolina, June 5-9, 2016 potentials with current measured through a Keithley 6485 picoammeter. All testing equipment shared a common ground.



Figure 1: (Left) An exploded device schematic showing the three layer structure and locations of applied potentials and measured current. Only one of the 4 shown gate electrodes was active. (Top Right) a nanochannel AFM scan showing the 16 ± 3 nm depth of the nanochannel. (Bottom Right) A cross sectional schematic view of the device. V_a (purple) is the axial current driven through the nanochannels while V_g (red) is applied signal to the gate electrode insulated from the nanochannels by the PDMS dielectric layer.

RESULTS

After verifying device operation [3, 16], the AC conductance using the I_{RMS} (RMS current) from sine wave applied axially at 0.1 Hz showed the nanochannel conductance was higher than the purely DC axial signal for dilute electrolytes (≤ 1 mM) or in the surface charge governed regime (SCGR) [2] (Figure 2). Notably, this result is in agreement with our hypothesis that at low frequencies, electroosmotic flow will occur and be more modulated than the equivalent (RMS voltage matched) DC counterpart since the total electric field is expected to be much higher in the transverse direction. The nanochannel conductance was to estimate the nanochannel depth to be ~19 nm [3, 5] in reasonable agreement with the etched channel depth of 16 ± 3 nm.

A representative plot of current vs. time for $V_g = \pm 2$ V DC and $V_g = 2$ V_{RMS} for AC with axial potential $V_a = 3$ V is shown in Figure 3. *I_{RMS}* was 66 pA for sine gating, 73 pA for square, and *I_{DC}* was 6.3 pA i.e., RMS matched AC provides nearly an order of magnitude higher measured current than DC. The rapid decay for the square wave (Figure 3B, shown with orange arrow) suggests the charging and discharging of the fluidic capacitors at the dielectric-fluid interface and subsequent control over the EDL. Since the flow is driven electrokinetically and is governed by ion transport, the measurement current over time was used to estimate the total charge, *Q* that has passed through the nanochannel. Therefore, the area under a current-time plot (i.e.,

 $Q = \int_{0}^{T} Idt$) where, *I* is the current and *T* is the measurement time.

For all cases in this work, a given gate voltage was applied over a 30 second period before the voltage was stepped up or down resulting in total charge values that can be directly compared (Figure 4).

Figure 4 shows Q for all three tested frequencies (0.1, 1, and 10 Hz) across the 2 orders of magnitude range for both the sine and square wave. Q increases with frequency for all measured concentrations in a sine wave while for a square wave gate input, the 1 Hz frequency shows the highest Q in the square wave case, the 10 Hz signal that produces the highest current response in the



Figure 2: The black DC data points show device conductance in the surface charge governed (purple shading) and bulk transport governed regimes (no shading) [3]. The blue data shows nanochannel condutance using the I_{RMS} for a 0. 1 Hz sine wave. The red line is the bulk fluid conductance for a 19 nm channel which was seen to be in reasonable agreement with measured data. Dashed lines are provided as eve guides.



Figure 3: Current vs. time for the DC and 0.1 Hz AC cases at $1x10^{-4}$ M as a representative plot with $V_a = 3$ V. (A) Sine waveform at the gate. (B) Square waveform at the gate. The plots show the charging and discharging cycle evident for square wave gating and also the increase in RMS current compared to DC only for similar gate voltage.

sine case shows the lowest charge transfer indicating an optimal frequency for charge transfer in the 0.1 - 10 Hz range, but requires further investigation. Additionally, in the sine wave cases, the total charge through the device remains relatively constant across frequency (with a slight drop moving from the SCGR to the BTGR) while in the square wave case, the charge transfer in the SCGR is constant but drops off in the BTGR. The differences in transport (both between frequencies and between



Figure 4: Average charge flowing through the nanochannels for both (A) Sine and (B) Square waveforms. $V_a = 3$ V in all cases. Dashed lines are eye guides. The pink data shows comparative DC charge transfer. For sine gating, charge transfer increases with frequency and is relatively invariant across concentrations. But, for square wave gating, both a frequency and concentration dependence was seen, with charge transfer decreasing with increasing concentration.

AC and DC signals) decrease as the concentration increases and moved into the BTGR. All AC cases were shown to have higher average charge than the RMS matched DC case where charge through the device for the $V_g = 2$ V case was added to the $V_g = -2$ V case.

A FEND equivalent circuit model was developed (Figure 5) for fitting to the measured current data. The model assigns a series of R/C circuit elements to the PDMS isolation layer, Stern layer, and the fluid diffuse layer and also accounts for the bulk fluid resistance. Each layer was modeled as a capacitor and resistor in parallel to allow for varying RC time constants for each component. The model solves the series of RC elements assigned to the three layers seen in Figure 5 for the total impedance according to the expression,

$$Z_{\rm T} = \frac{R_{\rm PDMS}}{1+j\omega C_{\rm PDMS}R_{\rm PDMS}} + \frac{R_{\rm S}}{1+j\omega C_{\rm S}R_{\rm S}} + \frac{R_{\rm D}}{1+j\omega C_{\rm D}R_{\rm D}} + R_{\rm b}$$
(1)

where Z_T is the total impedance, R_{PDMS} is the resistance of the PDMS, R_S is the resistance of the Stern layer, R_D is the resistance of the diffuse layer, R_b is the bulk resistance, ω is the frequency of the applied signal, C_{PDMS} is the capacitance of the PDMS layer, C_S is the capacitance of the Stern layer, and C_D is the capacitance of the diffuse layer.



Figure 5: An equivalent circuit model for sine gating as a representative case. PDMS, Stern, and diffuse layers were modeled as R/C circuits.

Since the R and C values were assumed to stay constant for a given electrolyte concentration, the bulk resistance value for each concentration (found from the DC conductance data in Figure 2) was also held constant. The model shows RC time constants for the PDMS layer was ~97 s and diffuse layer was ~0.02 s with all RC constants shown in Table 1. Therefore, the gate voltage frequency can modulate diffuse layer ion distributions. Moreover, the Stern layer modulation would require frequencies on the order of 100 kHz (based on RC time constants) so for the frequencies studied here, it would remain relatively in the steady state. The stability of the Stern layer throughout this process supports the claims this term can often be neglected when modeling similar systems. [17]

Table 1: A summary of the time constants from the Equivalent Circuit Model. Across three concentrations examined for each of the layers, the RC time constant was evaluated. The PDMS time constant ($R_{pdms}C_{pdms}$) remained constant with concentration change, the Stern Layer time constant (R_sC_s) remained constant with concentration change, and the diffuse layer time constant (R_dC_d) changed with concentration as the layer thickness was altered. The model and data agree within 3% and is well within the experimental error.

	Time Constant (s)		
	1*10 ⁻⁴ M	1*10 ⁻³ M	1*10 ⁻² M
R _{pdms} C _{pdms}	97	97	97
R _s C _s	7.5E-5	7.5E-5	7.5E-5
R _d C _d	0.021	0.019	0.026

CONCLUSIONS

An AC gated FEND was demonstrated with two applied waveforms over a range of frequencies (0.1 - 10 Hz). AC gated FENDs were shown to have a more than an order of magnitude higher of charge transfer than the equivalent DC system while in the surface charge governed regime. An equivalent circuit model shows RC time constants for the PDMS layer was ~97 s and diffuse layer was ~0.02 s. Therefore, the gate voltage frequency appear to modulate diffuse layer ion distributions based on the choice of gate frequencies used here. Moreover, the Stern layer modulation would require frequencies on the order of 100 kHz and does not currently appear to be affected by our current gating platform.

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TWO-LAYER PLATE MECHANICAL METAMATERIALS

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ABSTRACT

We present the fabrication and characterization of plates made from two ultrathin layers and capable of withstanding repeated extreme mechanical deformation. The ultrathin layers, one flat and the other corrugated, are less than 100 nm thick and created using atomic layer deposition, forming a joined structure similar to conventional honeycomb sandwich plates. The structures exhibit a high bending stiffness while still possessing a low area density of approximately 0.5 g/m². Finite element simulations show that the bending stiffness initially increases quadratically with the height of the plate, but saturates at very large heights. Measurements of bending stiffness using an atomic force microscope agree qualitatively with the simulations.

INTRODUCTION

Cellular solids offer low weight and unique mechanical and thermal properties, which explains their longstanding use for a variety of structural applications in the transportation and construction industries [1]. In the last decade, a novel class of cellular solids known as mechanical metamaterials has been introduced [2-3]. These solids have carefully designed and tightly controlled periodic 3D geometries, usually at the micro- or nanoscale, which lead to unique combinations of mechanical properties, allowing them to occupy new areas on the Ashby charts [4-6].

Recently, we introduced the concept of *plate mechanical metamaterials* [7]—cellular plates with carefully controlled periodic geometry and unique mechanical properties—as well as its initial realization in the form of freestanding corrugated plates made out of an ultrathin film. In particular, we used atomic layer deposition (ALD) and microfabrication techniques to make robust plates out of a single continuous ALD layer with cm-scale lateral dimensions and a thickness as low as 25 nm, creating the thinnest freestanding plates that can be picked up by hand [7]. Our continuous plates differ from "bulk mechanical metamaterials" reported by other groups (e.g., Refs. [2-6]), which form a lattice that is easily penetrated by air and are typically made using much slower fabrication techniques, such as nanoscale 3D printing [4-5] or evaporation-driven self-assembly [6].

At the macro scale, many types of periodic cellular plates are used. In particular, honeycomb lattices and sandwich structures, which consist of two face sheets attached to a periodic core, have become ubiquitous in construction, aerospace, scientific instrumentation (e.g., optical tables), and other industries that require lightweight rigid plates [8,9]. Sandwich structures possess a high bending stiffness and very low areal density (mass per unit area). However, macroscale sandwich plates generally cannot sustain sharp bending deformations without permanent damage [9].

Here, we report fabrication and characterization of fully suspended plate metamaterials made from <u>two</u> layers of nanoscale thickness, whose geometry and properties are reminiscent of honeycomb sandwich plates (Fig. 1). The two layers are offset from each other but at the same time are connected using hexagonal vertical walls, which prevent shear of the two layers with respect to one another. As a result, the two-layer plates offer much higher bending stiffness than the single-layer structures we reported earlier [7], while still possessing extremely low weight and mechanical robustness. The increase in the bending stiffness is expected, and its mechanism is similar to that used in conventional honeycomb sandwich plates. However, in contrast to sandwich composite plates, our nanoscale two-layer mechanical metamaterials can sustain extremely large deformations without fracture, fully recovering their original shape and not displaying any signs of internal damage.



Figure 1: (a) SEM image of a 1-mm-long and 0.5-mm-wide cantilevered plate made from a two-layer mechanical metamaterial. Because the freestanding ALD layers are highly transparent in SEM, they are colored for clarity. (b) Schematic illustrating the periodic geometry of the two-layer plate. The bottom layer is planar and continuous while the top layer is corrugated and includes etch holes for release. (c) SEM showing the detail of the cantilever edge. The layers are approximately 60-nm-thick and are colored differently for clarity. The plate height, i.e., the spacing between the two layers, is nominally 2 microns, but in practice varies slightly in a fully released structure.

FABRICATION

The plates were fabricated from ALD aluminum oxide (alumina) as illustrated in Fig. 2. First, we deposited a 60-nm-thick planar layer of aluminum oxide (Al₂O₃) on a cleaned double-side polished silicon wafer. The deposition was performed at 250 °C using water and trimethylaluminum precursors in the ALD tool. Next, a sacrificial layer of amorphous silicon (a-Si) was deposited at 175°C on the front side using plasma-enhanced chemical vapor deposition (PECVD). The thickness of the a-Si layer, which determines the height of the finished plate and the spacing between the ALD film layers, varied between 1 and 3 microns in different fabrication runs. The a-Si layer was then patterned using photolithography and anisotropic reactive ion etching (RIE) to obtain the hexagonal honeycomb pattern shown in Fig. 1b. The hexagonal honeycomb geometry was chosen because it exhibits approximately isotropic bending stiffness [1,7]. Subsequently, another 60-nm-layer of ALD alumina was deposited to form the corrugated layer. The second ALD layer was then patterned using photolithography and inductively coupled plasma (ICP) RIE to define the width and length of the cantilever as well as to open the etch holes for removing the a-Si sacrificial layer later on. Next, an approximately 500-nm-thick layer of silicon nitride was deposited

978-1-940470-02-3/HH2016/\$25©2016TRF DOI 10.31438/trf.hh2016.55 Solid-State Sensors, Actuators and Microsystems Workshop Hilton Head Island, South Carolina, June 5-9, 2016 on the back side of the wafer and patterned to serve as a hard mask for KOH wet-etching. The wafer was then backside-etched in KOH, stopping 10-50 μ m before reaching the ALD layers. Finally, the device was suspended using isotropic XeF₂ etching or isotropic CF₄+O₂ plasma RIE, which also removed the sacrificial a-Si trapped between the ALD layers. The SEM images confirm the presence of two alumina layers and the removal of the sacrificial layer (Fig. 1c).



Figure 2: Schematic of the fabrication steps.



Figure 3: Photographs of partly (a) and fully (b) etched cantilevers under an optical microscope. The remaining a-Si trapped between the ALD alumina layers appears brown in the partly etched cantilever (a). In contrast, the completely etched cantilever without residual amorphous silicon (b) is completely clear (transparent).

The complete removal of the trapped Si is critical because the unique mechanical properties of plate mechanical metamaterials depend on the extremely low thickness of the structural ALD layers. Any residual a-Si dramatically changed the mechanical properties of the cantilevers, leading to easy fracturing during sharp bending deformations. Therefore, the etching of a-Si was continued until all evidence of a-Si disappeared in optical and scanning electron micrographs. Because the ALD alumina is highly transparent compared to a-Si, the presence of the a-Si could be easily detected in optical micrographs (Fig. 3).

SIMULATIONS OF BENDING STIFFNESS

The two-layer geometry described above is much easier to realize using MEMS fabrication processes than the exact analog of a standard honeycomb sandwich composite [8,9], which would consist of two planar layers (faces) and a corrugated honeycomb layer in between them. However, based on our finite element simulations and experiments, the stiffness of the fabricated twolayer structures is comparable to that of a full honeycomb composite.

The small-deformation response of the two-layer plate structures was studied using the shell interface of COMSOL Multiphysics (version 5.0). The layers were modeled as alumina films with isotropic linear elastic properties, a Young's modulus of 130 GPa, and a Poisson ratio of 0.22 [7,10]. Mesh convergence was achieved using triangular elements with a minimum and maximum element size of 0.005 and 10 microns respectively. Geometric parameters were set to coincide with those of the experimental cantilever. To find the spring constant of a cantilever and the corresponding bending stiffness of the plate material, one end of the cantilever was fixed while a normal, out-of-plane load was applied at the other end. Mirror symmetry conditions were applied to the edges along the cantilever's length.



Plate height *h* (microns)

Figure 4: The symbols show the simulated effective bending stiffness K_{eff} of the two-layer plates as a function of the plate height. For comparison, the solid line shows the bending stiffness of a perfect sandwich plate of the same height (h) made from the films of the same thickness (t): $K_{sandwich} = Y th^2/2$, where Y = 130 GPa is the Young's modulus of the ALD aluminum oxide.

Figure 4 shows the bending stiffness K_{eff} of two-layer plate metamaterial as predicted by the finite element simulations. We note that the spring constant k of the cantilever with a width W_{cant} and length L_{cant} is related to the plate bending stiffness by the formula $k = 3W_{\text{cant}}K_{\text{eff}}/L_{cant}^3$. The simulations show that for low heights, the bending stiffness of the fabricated two-layer plates increases

quadratically with the plate height. It also remains approximately a factor of two lower than the bending stiffness of a perfect sandwich plate, i.e., a plate consisting of two face sheets and a corrugated layer between them that completely prevents the lateral shear of the face sheets with respect to one another. The fact that the bending stiffness is lower than that of a perfect sandwich structure is not surprising, given that the top layer of our plates is not perfect: it contains etch holes and is completely absent inside the hexagons. At heights above ~10 μ m, the bending stiffness saturates as the vertical hexagonal walls that connect the top layer with respect to the bottom layer.

Still, even for the relatively low heights reported here (1-3 µm), the bending stiffness of this two-layer plate mechanical metamaterial is many orders of magnitude higher than that of singlelayer plate mechanical metamaterials reported earlier. From Fig. 3, the effective bending stiffness is predicted to be between $K_{eff} \approx$ 2×10^{-9} N·m for h = 1 µm and $\approx 10^{-8}$ N·m for h = 3 µm. In contrast, single-layer plate metamaterials with similar in-plane geometry and the same 60-nm thickness of the ALD film have bending stiffness of only 3×10^{-11} N·m. By adding a second layer, we therefore achieved 2-3 orders of magnitude improvement in the bending stiffness of the plate material while the mass per unit area is less than doubled. The areal density of the two layer-structures can be easily calculated from the geometry and the density of the amorphous aluminum oxide ($\rho = 4000$ kg/m³) and is approximately 0.5 gram per square meter.

MEASUREMENTS OF BENDING STIFFNESS

To validate the predictions of the finite element simulations, we determined the spring constants of the cantilevers that were made from two-layer plate mechanical metamaterials (shown in Fig. 1 and 2). In particular, force-displacement curves were obtained at the tip of each cantilever using an Asylum atomic force microscope (AFM). Given the relatively low spring constants of the two-layer plates, we used soft AFM cantilever tips with a spring constant of $k_{AFM tip} \approx 0.01$ N/m and 0.26 N/m, which were determined using the thermal noise method. By modeling the plate as a spring in series with the AFM cantilever, the slope of the force displacement curve corresponds to the effective spring constant K_{total} , which satisfies $k_{total}^{-1} = k_{AFM tip}^{-1} + k_{plate}^{-1}$.



Figure 4: Force-displacement curves of two-layer plate cantilevers obtained at the tip of each cantilever using an atomic force microscope. The 1-µm-tall cantilevers were measured using an AFM tip with $K_{AFM tip} \approx 0.01$ N/m, while the 3-µm-tall cantilever was measured using an AFM tip with $K_{AFM tip} \approx 0.26$ N/m.

Using this equation, we determined that the spring constants were 1.5 \pm 0.3 mN/m and 5.4 \pm 1.1 mN/m for the 1-µm-tall and 3µm-tall cantilevers, respectively. Using the cantilever width of $W_{\text{cant}} = 0.45 \text{ mm}$ and the length of $L_{cant} = 1 \text{ mm}$, we could also determine the effective bending stiffness of the plate material: $K_{\rm eff} = kL_{\rm cant}^3/3W_{\rm cant}$, which yielded bending stiffness of $(1.1 \pm 0.2) \times 10^{-10}$ N·m and $(4\pm 1) \times 10^{-10}$ N·m for the 1-µm-tall and 3-µm-tall cantilevers, respectively. These measured bending stiffnesses are approximately a factor of two lower than those predicted by finite element simulations (Fig. 4), which could be attributed to the fact that the actual spacing between the top and bottom layers is lower in experimental fully released devices than the nominal design values (Fig. 1). Despite this discrepancy, the predicted trend of increasing bending stiffness with increasing plate height is fully supported by the experiments. In the future, we plan to fabricate structures with even larger heights to test the prediction that the bending stiffness saturates at large heights.

RECOVERY FROM EXTREME BENDING

The most surprising property of our two-layer plates is their ability to sustain sharp bending without deformation. We observed the mechanical robustness of the two-layer plates using a micromanipulator probe while imaging the device in an FEI Strata focused ion beam (FIB) tool. Similar to the single-layer plates reported earlier [7], the two-layer plates showed a remarkable ability to recover from several extreme deformations without any apparent fracture or permanent deformation (Fig. 5). This is strikingly different from the behavior of macroscale multilayer composites, such as honeycomb sandwich plates, which typically fracture, delaminate, or irreversibly buckle after such sharp bending [8,9].



Figure 5: Sequential screenshots from a live video feed of an FIB tool, showing a 1-µm-tall two-layer cantilevered plate undergoing extreme bending deformations and then recovering its original shape.

The exact nature of this observed robustness is not fully clear at this point. As discussed in our previous publication [7], sandwich plates that undergo very sharp bends are expected to develop strains of the order of 1 unless the structure of plate changes. Real sandwich plates undergoing sharp bending or folding therefore either fracture completely or irreversibly change their structure [8,9]. For example, the face sheets of a conventional sandwich composite can delaminate from the hexagonal webbing that connects the two face sheets. Alternatively, the hexagonal webbing can be crumpled by irreversible buckling. In any of these cases, the sandwich plate typically remains permanently deformed and weakened in the area that underwent sharp bending or folding.

Yet, high resolution scanning electron micrographs do not reveal any evidence of irreversible changes after extreme bending of our two-layer plates (Fig. 6). While the plates clearly change their internal structure by locally buckling the vertical walls during sharp bending, the deformations appear to be fully elastic, reversing after the external force is removed. While the exact form of this elastic local buckling deformation needs to be investigated further, we note that similar recoverable deformations have been observed in "bulk mechanical metamaterials" and, in particular, nanolattices made from ultrathin ALD aluminum oxide [5].



Figure 6: Sequential scanning electron micrographs illustrating a 2-µm-tall two-layer cantilevered plate undergoing extreme bending deformations and then recovering its original shape. After the recovery, the plate did not display any signs of damage even in high-resolution SEM images of the areas where sharp folding occurred.

CONCLUSION

We have demonstrated shape-recovering, flat, ultrathin, continuous plate mechanical metamaterials made out of two ALD layers. Relative to previously reported single-layer plates [7], the bending stiffness of two-layer plates is increased by 2-3 orders of magnitude and is, in fact, within a factor of two of that of an ideal sandwich plate. At the same time, the two-layer plates still feature ultralow areal density (~0.5 gram per square meter) and can be made using a relatively high-throughput fabrication method that can be easily scaled to cm-scale plates. Like the single-layer plates reported earlier, the two-layer plate mechanical metamaterials can pop back elastically to their original position after extremely sharp bending deformations without any apparent damage. This is in striking contrast to macroscopic sandwich composites, which typically fracture, delaminate, or irreversibly buckle after sharp bending. The observed robustness and the apparent lack of irreversible deformation are likely due to reversible buckling of ultrathin structures [5].

Ultralow weight, mechanical robustness, thermal insulation, as well as chemical and thermal stability of alumina make two-layer plate metamaterials attractive for numerous applications, including structural elements in flying microrobots, high-temperature thermal insulation in energy converters, testing of nanoscale strength enhancement, new types of optical and acoustic metamaterials, as well as ultra-lightweight hollow MEMS sensors and resonators.

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A LOW-COST PAPER-BASED MODEL FOR ON-CHIP HUMAN RESPIRATORY SYSTEM STUDIES

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ABSTRACT

We present the use of hydrophobic paper as an effective semipermeable membrane, ideal for air-liquid interface cell culture applications. The surface properties of the paper is modified through selective CO₂ laser-assisted scrubbing to create a unique porous substrate with hydrophilic regions that regulates fluid diffusion and cell attachment. To select the appropriate model, four promising hydrophobic films were compared with each other in terms of gas permeability and long-term strength in an aqueous environment (wet-strength). Among the investigated substrates, parchment paper showed the fastest rate of oxygen diffusion. Mechanical analysis revealed superior dry and wet tensile strength of 69.5 MPa and 29 MPa for parchment paper which remained virtually unchanged after 7 days of submersion in PBS. The final platform with a constant flow of media in the basal lumen was found to show confluent coverage of Calu-3 (airway epithelial cells) with a mature ZO1 expression at the air-liquid interface after 7 days of cell culture.

INTRODUCTION

Respiratory diseases (e.g., pneumonia, tuberculosis, and lung cancer) are among the leading causes of mortality worldwide [1], [2]. The scarcity of current therapies calls for the development of more clinically relevant models to facilitate the study of the respiratory system and drug testing. To aid such understanding and to develop appropriate therapeutics, researchers are limited to animal and transwell-based in-vitro models for epithelial tissue. The restricted control of experimental variables and biological irrelevancy of animal models can result in both time consuming and unreliable data. Therefore, in-vitro models that mimic the functionality and physiological properties of the respiratory epithelia can provide a better understanding of the drug efficacy at the cellular level [3]. Currently, the most common in-vitro approach is the use of commercially available semipermeable hanging film for creating an air-liquid interface (ALI) in transwell flasks [4]. This technique has proven to be physiologically invaluable for the development of epithelial therapeutics; however, it does not constitute an ideal (and practical) epithelial model due to its inability to faithfully recapitulate the mechanics of epithelial tissue (e.g., most are brittle and too impermeable to gases, or not sufficiently strong).

Recently, many researchers have attempted to fabricate microengineered models that mimics the structure and functionality of the various physiological environments by using micro-fabrication technology. These efforts have resulted in the development of several examples of organ-on-a-chip technologies including lung, brain, kidney, and cancer [5]–[7]. However, many of these require time consuming fabrication processes and high-cost cleanroom manufacturing, limiting the translation of the technology to high-throughput pharmaceutical research industry. A more practical approach (clinically and financially) would be one

which takes advantage of modern rapid prototyping equipment for low-cost production and customization as well as suitable commercially available materials which are inexpensive, ubiquitous, and biocompatible [8].

As a more cost effective approach, many researcher have successfully shown the use of paper as an alternative material for conventional cell culture substrates (e.g., polystyrene, and PDMS) [9], due to its naturally biocompatible 3D cellulose fiber composition and its webbed architecture for efficient cell attachment [10], [11]. However, the inherent hydrophilic nature and low mechanical strength when wet, prevents it's prolong use in aqueous environments [12]. Nevertheless, impregnating paper with hydrophobic materials (e.g., wax, silicone) imparts on it unique mechanical and fluidic properties which mimic those of epithelial tissue and which can be conveniently integrated with BioMEMS devices [13]. In this study, we expand the benefit of paper-based biological assays by presenting a model for epithelial tissue consisting of a commercial hydrophobic paper (parchment paper) which is further customized via laser machining to create a unique substrate for epithelial cell culture and attachment, Figure 1a.

A schematic of the working mechanism of the microfluidic airway systems is demonstrated in Figure 1b. The system consist of an upper and a lower laser cut acrylic chambers corresponding



Figure 1: (a) Illustration of the respiratory epithelial tissue and paper-based microfabricated lung-mimicking device, (b) schematic of the air-liquid-interface (ALI) platform.

978-1-940470-02-3/HH2016/\$25©2016TRF DOI 10.31438/trf.hh2016.56 Solid-State Sensors, Actuators and Microsystems Workshop Hilton Head Island, South Carolina, June 5-9, 2016 to the apical (airway lumen) and basal compartments of airway epithelium. The laser-treated hydrophobic paper provides a semipermeable support for the cell attachment with regulated permeability of both oxygen and nutrition, mimicing the *in-vivo* basement membrane.

EXPERIMENTAL

To compare different substrates for the described airway model, we characterized four different promising common films in terms of their wet-strength (standard tensile strain as a function of wetting duration) and gas permeability. These films were wax paper, copy paper, parchment paper, PDMS, and a paper-PDMS composite prepared by placing a sheet of filter paper on a thin layer of 50 µm spin-coated PDMS pre-polymer. Standard tensile stress-strain test was used to characterize how well the fibers in different papers hold together after the wetted for various number of days. In this test, the papers were submerged in buffer saline solution (PBS) for various durations (0-7days) and the ultimate tensile strength (UTS) and Young's modulus were subsequently measured using a universal testing machine (Admet[®], model eXpert 1000). All specimens were laser cut to the same (5 mm \times 20 mm) dimensions. The tensile strength measurements were performed by fixing the two ends of the film and stretching from 0 % to 12 % strain at a constant extension velocity of 10 mm/min.

A customized setup was used to measure the gas permeability through different films, Figure 2. The structure consisted of a cylindrical chamber filled with 20 ml deoxygenated DI water and covered by 20 mm in diameter circular sample of the film. For all tests, the dissolved oxygen was removed by purging the water for 8 h with nitrogen. The oxygen permeability was confirmed with real time measurement of the dissolved oxygen using an optical oxygen sensor positioned in the DI water chamber. All measurements were performed at room temperature and atmospheric pressure. We evaluated the oxygen permeability of different membrane via the rate of dissolve oxygen increase in the water. Figure 3 shows the optical photographs of the paper-based microfluidic airway systems. In this platform, the laser patterned hydrophobic paper with hydrophilic traces were placed between to laser-cut acrylic chambers (1.5 mm deep and 15 mm long channels). As the first step, each component of ALI platform including parchment paper were sterilized by irradiating with ultraviolet (UV) light at a distance of 8 cm for 15 min. The ALI platform was assembled as shown in the Figure 3 inset. The inletoutlet tubing were connected to platform after sterilization with 70% ethanol. An antibiotic/antimycotic solution was placed in the upper/ lower chambers and interface tubing overnight at room temperature inside the tissue culture hood. The sterilizing solution



Figure 2: Gas permeability test setup.

was removed and the chambers and tubing were washed with PBS twice. Next the top open chamber was filled with media and seeded at a density of 1.5×10^6 cells/ml in 200 µl with Airway Epithelial Cells (Calu-3) and the bottom chamber was infused with a constant supply of media at a flow rate of 8 µl/min. The whole assembly was incubated at 37 °C, 5% CO2 under static condition. Once the cells exhibited a confluent monolayer coverage on the hydrophilic regions of the paper (5 mm × 15 mm) an air-liquid interface was established by removing media from the top chamber while maintain a constant flow of media in the bottom chamber for 7 days. Live-dead assay was performed with the biohazard cell viability kit (Life technologies) which contains SYTO 10 (green fluorescent nucleic acid stain for live cells) and DEAD Red (ethidium homodimer-2, nucleic acid stain for dead cells). The dye mixture was prepared in 1:500 dilution of SYTO 10 and 1: 1000 dilution of DEAD Red. Before addition of dye mixture, all samples were washed with PBS for 1 time. In order to assess the airway epithelium's differentiation that closely resembles native airway tissue, the cells were stained for ZO1 (bright green), pancytokeratin marker (panKRT-green) and DAPI (nuclear staining blue) at 5days and 7days in ALI under static and flow conditions. The static condition was used as a model for studying lung injuries induced by thromboembolism (pulmonary embolism), which the pulmonary arteries are blocked and blood flow is in a static condition.



Figure 3: Photograph of components and final assembled onchip microfluidic respiratory epithelia tissue model (inset). All scale bars: 10 mm.

RESULTS AND DISCUSSION Mechanical characterization

Figure 4 shows the stress-strain curve for filter paper and various hydrophobic papers before and after 24 h submerging in PBS. All the samples in dry states show a linear stress-strain profile with a small strain (2.5 %) before rupture. The sharp drop in the plot specifies the strain level at which the paper splits in half. The commercial parchment paper and wax paper have the highest mechanical properties in dry state with the UTS of 69.4 MPa and 73 MPa and Young's moduli of 33.2 GPa and 33.4 GPa, respectively. Filter paper impregnated with PDMS has a dry UTS of 21.4 MPa which is three time higher than the pristine filter paper (7.6 MPa). This increase in mechanical strength is explained by the presence of the PDMS filler in the network fiber of the paper forming a stronger composite film. Wet tensile strength results, for all the specimens, show an increase in elasticity but decrease in mechanical strength.

Unlike the dry state, the wet papers show and an average 6 % strain followed by necking before rupture. This is due to the diffusion and plasticizing effect of water molecules in the paper film. The results show that the hydrophobic papers retain some of their mechanical strength after 24 h submerging in PBS, whereas the filter paper wet UTS drastically decreases to 1.2 MPa and starts to disintegrate in the solution. Among the investigated hydrophobic papers, parchment paper retains more than 40 % of its original dry UTS strength followed by PDMS/paper and wax paper with retentions of 31% and 14 %, respectively. The trend in change in UTS for different films as a function of wetting duration is shown in Figure 5. The parchment paper and wax paper show a stable retention of mechanical strength (UTS) of 29 MPa and 8.9 MPa for 7 days. However, for filter paper and PDMS/paper, a longer wetting duration reduces the mechanical strength (UTS) down to 0.4 MPa and 5.4 MPa, respectively.



Figure 4: Stress vs. strain of dry and wet paper films after 24 hours of PBS immersion (a) parchment paper, (b) wax paper, (c) paper-PDMS, and (d) filter paper.



Figure 5: Ultimate tensile strength for different papers as a function of wetting duration.

Oxygen permeability characterization

Figure 6 shows the gas permeability results for different films using the aforementioned setup (Figure 2). For all measurements the initial dissolved oxygen of the DI water was close to zero (~0.5 ppm) and increased with time up to the oxygen saturation level in the water (8 ppm). The increase is due to the diffusion of the oxygen gas in ambient condition through the membrane and its dissolution in the water. Without any membrane, the water equilibrates to its steady-state saturation level of about 8 ppm in less than 140 min. However, when the chamber is covered, the time required for oxygen saturation increases. The results show the longest oxygen equilibration time occurs for a pristine 100 µm membrane of PDMS (720 min), and the shortest time occurs for parchment paper (210 min, with an average rate of 2.4 ppm/h). No signs of water leakage were observed with the hydrophobic films during any of the measurements. The mechanical strength and gas permeability results show the superior performance of the parchment paper, providing a suitable substrate for the proposed ALI platform.



Figure 6: Oxygen permeability of different hydrophobic paper membranes.

Cell culture results

As shown in Figure 7a, with the aid of live (green) and dead (red) staining the CALU3 cells exhibited a confluent monolayer coverage with high cell viability (>90%) after 3 days. In continuous flow condition the mature ZO1 expression (chicken wire appearance) was more prominent after 7 days culture compared to the 5 day ALI samples, Figure 7b. ZO1 is the tight junction protein that is mainly expressed in the differentiated airway epithelium. The results show a much faster epithelium differentiation compared to commercial transwell plates which often require 30 days for complete differentiation. However, in static ALI condition, there is no sign of ZO1 staining, and most of the cells die rather than fully differentiating, Figure 7c. The results show that parchment paper provides an ideal, robust, and inexpensive platform for generating epithelial cells on-chip in a high-throughput fashion for disease modelling and in-vitro drug testing.



Figure 7: (a) Assessment of cell attachment and viability after 3days, (b) ZO1 expression (bright green) on differentiated CALU3 cells at Air Liquid Interface for 5 and 7days under flow condition and (c) under static condition.

CONCLUSIONS

In this study, we investigated the potential use of a paperbased microfluidic system as an in-vitro model for human respiratory system studies. Among different papers, parchment paper showed superior performance in terms of wet strength and gas permeability. Furthermore, by selectively treating the surface of this hydrophobic paper with a CO₂ laser, we were able to produce hydrophilic regions with effective cell attachment and to regulate fluid diffusion which mimics the semi-permeable properties observed in basement membrane of human respiratory system. The final device, composed of parchment paper and acrylic microfluidics, was able to maintain long-term stability under constant flow of media with faster differentiation rate of airway epithelium compared to conventional transwell methods. This cost-effective platform can facilitate new drug discovery and improve the understanding of the etiology for different diseases by providing a model that better mimics the respiratory surface at the cellular level.

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DISTRIBUTED AND THERMO-ACOUSTICALLY COUPLED MODELING FOR ACCURATE PREDICTION OF THERMAL NONLINEARITY IN PIEZOELECTRIC MEMS RESONATORS

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ABSTRACT

In this paper, we employ for the first time a distributed and thermo-acoustically coupled modeling technique for thermal nonlinearity. The technique captures the true distribution of temperature throughout the resonator and its surrounding support structure. The resultant non-uniform temperature distribution modifies the stiffness coefficients in an acoustic simulation to directly compute the sensitivity of the frequency to power input. The technique results in new insights with respect to geometry, and enables geometric design tradeoffs to mitigate the effects of thermal nonlinearity. Our technique is compared to the lumped method in prior work and verified by measurements on two aluminum nitride resonators.

INTRODUCTION

Thermal nonlinearity has been identified as one of the key limiters on power handling and the primary mechanism for producing intermodulation in piezoelectric MEMS resonators [1][2][3]. Previous techniques to model thermal nonlinearity are either analytic [4] or lumped [1], neither of which can account for geometric details, and thus are limited in capturing detailed thermoacoustic coupling. For instance, the lumped model from [1], as shown Fig. 1, assumes that the resonator body is at a single temperature and the only significant thermal resistance to heat flow is through the anchors. This assumption breaks down as the length of the resonator grows such that there is a significant thermal resistance from the center of the resonator to the anchors. Similarly, if the anchors are widened to be on the order of the resonator width, the resonator body's thermal resistance is also on the same order as the anchors and cannot be neglected. While the thermal resistors could be adjusted or a different thermal circuit topology could be used, both require heuristic knowledge, and are approximations at best. Thus, it is challenging for heuristic models to capture the true thermal paths and be predictive for device performance optimization.

To improve, we employ a distributed and thermo-acoustically coupled modeling technique via the finite-element analysis (FEA). The coupled analyses determine the power coefficient of frequency (PCF) which directly relates the change in resonance frequency to the absorbed power. It is a more precise method because the nonuniformed heat generation and temperature profile across the resonator body is explicitly determined from the acoustic modeshape. The derived temperature profile is subsequently used to modify the stiffness for each meshed element and compute the resonance change via frequency domain FEA. Similar to the technique in [1], this work also captures the thermal nonlinearity by realizing that the shift in resonance frequency due to self-heating not only changes the amplitude of oscillation and thus the heat generated at a given frequency, but also alters the impedance mismatch seen by the source, further changing the heat generated. By coupling the feedback effect in thermal nonlinearity [1] with the PCF-based modeling method, the impact of thermal nonlinearity on the resonator admittance response is determined. Two aluminum nitride (AlN) resonators similar to those in [5] are measured, and results are compared with our modeled results to demonstrate the effectiveness



Figure 1: Lumped thermal model applied in [1]. The resonator body is at a single temperature, T, thermally coupled to ambient, To, through thermal resistors for the anchors.



Figure 2: Equivalent Butterworth Van-Dyke model for the resonator showing power dependent circuit components.

of the developed model.

MODELING METHODOLOGY

In this section a top-down description of the modeling methodology is presented, starting with the origin of the nonlinearity.

Absorbed Power

The self-heating that shifts the resonance frequency and thus the impedance of the resonator, is a function of the steady-state absorbed power, *P*. That is, $Z = Z(\omega, P)$ where ω is the radian drive frequency. The absorbed power also obeys the standard relation for absorbed power of the load at an impedance mismatch:

$$\frac{P}{P_{avs}} = 1 - \left| \frac{Z(\omega, P) - Z_0}{Z(\omega, P) + Z_0} \right|^2 \tag{1}$$

where P_{avs} is the power available from a source with an impedance of Z_0 . If the functional relationship $Z(\omega, P)$ can be determined, then Eq. 1 can be solved numerically for each frequency, ω , to determine $P = P(\omega)$. The desired admittance from thermal nonlinearity of selfheating is $Y(\omega) = Z(\omega, P(\omega))^{-1}$. In the next section we describe the determination of the function $Z(\omega, P)$.

Resonator Input Impedance

The impedance of a piezoelectric resonator near resonance is well modeled by the Butterworth Van-Dyke (BVD) circuit model shown in Figure 2. The components of the BVD circuit without selfheating (P=0) can be determined from simulation or measurement of the resonance frequency (ω_0), the electromechanical coupling coefficient, k_r^2 , the static capacitance, C_0 , and the quality factor, Q. To determine the circuit components under self-heating, note that

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Figure 3: AlN laterally vibrating resonator used in this study and similar to [5].

for a given *P*, the self-heating in steady-state shifts the resonance frequency, thus $\omega_0 = \omega_0(P)$, which implies the motional resistance, R_m , and motional inductance, L_m , are functions of absorbed power:

$$R_{m}(P) = \frac{\pi^{2}}{8} \frac{1}{\omega_{0}(P)C_{0}k_{t}^{2}Q}$$

$$L_{m}(P) = \frac{\pi^{2}}{8} \frac{1}{\omega_{0}(P)C_{0}k_{t}^{2}}$$
(2)

Note that we assume k_t^2 and C_0 to be independent of self-heating as the temperature coefficients of piezoelectric coupling and permittivity of AlN are insignificant. Q, despite being observed to a weak function of power prior to thermal bifurcation [2], is also treated as a constant versus power for simplifying the discussion in this work.

Since the heat equation is linear, and assuming the temperature variation is small enough that the elastic stiffness varies linearly, the resonance frequency is linear with power and can be written in terms of a *power coefficient of frequency* (PCF):

$$\omega_0(P) = \omega_0^0 (1 + PCF \cdot P) \tag{3}$$

where ω_0^0 is the resonance frequency at ambient temperature and zero input power (*P*=0). In the next section, we describe the determination of PCF with distributed thermo-acoustic modeling via the finite-element method.

Power Coefficient of Frequency

In order to complete the determination of $Z(\omega,P)$, the power coefficient of frequency (PCF) as defined in Eq. 3 must be determined. To do so, we use the finite element method to compute ω_0^0 and $\omega_0(P')$ for a given P' and use Eq. 3 to compute PCF. For simplicity of explanation, we assume the power lost to acoustic radiation to the substrate is negligible (we will address this in more detail below). Under this assumption, the absorbed power, P, generates heat proportional to the square of the magnitude of the local displacement. This is consistent with effects such as interfacial loss and thermoelastic damping. The local displacement will be proportional to the local eigenmode displacement and can be scaled appropriately to produce the correct net generated heat, P', for the local heat generation used in the thermo-acoustic modeling. The six steps of the method are as follows:

- Compute zero-power (ambient temperature) resonant mode frequency ω₀⁰, and mode shape displacements, *u(r)*, where *r* is a point inside the resonator
- 2. Apply volume heat flux, $q(\mathbf{r})$, to the thermal model $q(\mathbf{r}) = k|\mathbf{u}(\mathbf{r})|^2$ with k such that the total heat applied is the given P'
- 3. Solve the heat equation in the resonator, support structure, and substrate to determine temperature field, T(r)
- 4. Modify the local stiffness tensor: $c = c(T(\mathbf{r}))$
- 5. Recompute mode frequency using the distributed stiffness $c(T(\mathbf{r}))$ to determine $\omega_0(P')$



Figure 4: Half of the solid model to be used in Finite-element analysis. The model includes the resonator, support structure, undercut substrate, and portion of full thickness substrate.

TABLE I. Key characteristics of the two resonators				
Device Parameters	Symbol	Device 1	Device 2	
Frequency (MHz)	f_0	456	567	
Bottom Electrode Thickness (nm)	t_{Pt}	150	150	
AlN Thickness (µm)	t _{AIN}	1	1	
Top Electrode Thickness (nm)	t_{Al}	100	100	
Pitch Width (µm)	W_p	10	8	
Resonator Length (µm)	L	210	210	
Number of Electrodes	п	7	7	

6. $PCF = (\omega_0(P')/\omega_0^0 - 1)/P'$

The assumption of negligible acoustic loss to the substrate was shown to be valid for the AlN resonators in [6] resonating near 1 GHz. If that is not the case then in step 2 the total heat applied, P', can be replaced with $P''=P'(1 - \alpha)$ where α is the fraction of acoustic power loss to the substrate.

RESULTS

We will demonstrate the methodology on two aluminumnitride (AlN) Laterally Vibrating Resonators (LVR) operating in their S0 mode similar to those described in [5] and shown in Fig. 3. The dimensions of the two resonators are shown in Table I.

The methodology described in the previous section was implemented in the CoventorWareTM commercial software suite [7]. Most of the steps in the power coefficient of frequency calculation as well as the nonlinearity modeling of Eq. 1 were automated within CoventorWare with the exception of a few steps as will become clear below.

The modeling domain shown in Fig. 4 includes not only the resonator, but also the support structure and undercut substrate. Modeling beyond the resonator is required because the etch that releases the resonator also undercuts the substrate and this undercut region has significant thermal resistance.

Next, we will demonstrate the details of the PCF method using the 567 MHz resonator, and then present a comparison to measurement of the nonlinear response for both resonators.

Modeling of PCF

The model of Fig. 4 was meshed into 10555 parabolic piezoelectro-mechanical elements. 10215 of these elements were on the resonator and 340 on the support structure and substrate. For step 1 of the method described in the previous section, CoventorWare's FastPZE module was used to rapidly determine both the resonant frequency and its related displacement mode shape in the admittance response for the resonator at ambient temperature. Fig. 5(a) shows the S0 displacement mode shape, |u(r)|. As expected, Fig. 5(b) shows the mode has a much larger displacement response near the center of the resonator compared to the portion near the anchors.



Figure 5: (a) Top view of the S0 operating mode of the LVR (top); (b) modal displacement amplitude along the indicated line.



Figure 6: Approximate power absorption profile and boundaries of discretized zones for power input.

For step 2, a volume heat flux, $q(\mathbf{r})$, proportional to the square of the amplitude of the modal displacement field should automatically be applied to the model for the steady-state heat simulation. At the time of this writing, this automatic step was not available so a piecewise constant heat flux on 6 regions was applied that matches the square of the displacement field in form as shown in Fig. 6. This loss in detail did not significantly impact the PCF computation.

To compute the temperature distribution for step 3, the outer boundary of the substrate region is fixed at the ambient temperature of 293 K and the steady-state heat equation is solved with the heat flux of Fig. 6 scaled to give a total heat flux over the entire resonator of P' = 1 mW. The thermal conductivity for thin film aluminum nitride was used from [8]. The resulting temperature distribution shown in Fig. 7 shows a significant variation in temperature over the body of the resonator. This variation could not be modeled using a lumped method of a single temperature for the entire resonator.

For step 4, the thermo-acoustic coupling is obtained by using this temperature distribution to locally modify the stiffness tensor of aluminum nitride based on the temperature coefficients of stiffness from [9]. The resonance frequency is then recomputed with these distributed stiffness using the FastPZE module. The result for power levels of 1, 2, 4 mW is shown in Fig. 8 normalized to the resonance frequency. Based on P = 1 mW, the PCF is -360 ppm/mW. The slight nonlinearity after 2 mW may be the result of thermal expansion or



Figure 7: (a) Temperature field, $T(\mathbf{r})$, for 1mW power absorbed; (b) temperature profile extracted along the indicated line.



Figure.8: simulated frequency variation of the S0 mode as a function of the absorbed power for device 2. Frequency deviation is normalized to $f_0 = 566$ MHz.

thermal stress that shifts the mechanical operating point of the structure.

The total computation time to perform this PCF calculation for P=1 mW was 15 minutes on a four-core computer with a clock speed of 2.70 GHz.

Modeled and Measured Thermal Nonlinearity

The PCF from the previous section was used to define $\omega_0(P)$ from Eq. 3. This definition of $\omega_0(P)$ was used in Eqs. 2 to determine $Z(\omega,P)$ from Fig. 2. Values for, ω_0^0 , k_t^2 , C_0 , and Q for Eqs. 2 were taken from measured data rather than simulation to focus the following results on thermal nonlinearity modeling rather than the slight mismatch in simulated resonance frequency.

To determine $P(\omega)$ for a given $P_{\alpha\nu s}$ and Z_0 , Eq. 1 was solved for P with Newton's method separately for each frequency point of interest. To capture hysteresis, the initial guess for P is the solution for P from the previous frequency point. Results for $P_{\alpha\nu s} = 1$ dBm are given in Fig. 9. As long as the first point in the upward or downward frequency sweep is outside the multi-valued region, the hysteresis will be properly captured.

With this $P(\omega)$, the admittance is computed as $Y(\omega) = Z(\omega, P(\omega))^{-1}$. The results for the two resonators are shown in Fig. 10 for $Z_0 = 50\Omega$ and a series of increasing values of P_{avs} . For both resonators the lumped method severely under predicts the thermal



Figure 9: Power absorption as a function of frequency.



Figure 10: Measured admittance of device 1 compared with the (a) thermal-acoustically coupled model and (b) lumped model. Measured admittance of device 2 compared with (c) thermal-acoustically coupled model and (e) lumped model. The measurement was done with an forward frequency sweep.

nonlinearity because it does not account for the higher thermal resistance for heat generated near the middle of the resonator. In contrast, the method of this paper shows a similar nonlinear response as the measurement, with some over prediction. The over prediction is expected since the portion of energy that is acoustically radiated into the substrate has not been subtracted from P in the computation of PCF. Also, thin film thermal conductivity used from [8] is for deposition at 20 °C, but the actual film was deposited at 300 °C. Thin film sputter at higher temperature is expected to have better orientation and thus have conductivity closer to the higher bulk value than the value from [8].

CONCLUSIONS

In summary, this work presents a distributed and thermalacoustically coupled approach for accurate prediction of thermal nonlinearity in piezoelectric MEMS resonators. The methodology and detailed steps involved in the model have been outlined and explained. Two AlN resonators were used as the experimental testbed to validate the accuracy of the model. In comparison to the methods in prior work, the modeled results have shown closer match with the measurements. The modeling accuracy can be further improved by using measured thermal conductivity of the sputtered AlN thin-film and determining the proportion of energy acoustically radiated to the substrate. Despite being only showcased in AlN devices, the model herein can be readily extended to model thermal nonlinearity in any micro-scale resonators with temperature instability.

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A COMB-DRIVE ACTUATOR ENABLED DYNAMICALLY TUNABLE TERAHERTZ METAMATERIAL

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ABSTRACT

This paper reports our recent progress on a dynamically tunable terahertz metamaterial based on broadside-coupled split ring resonators (BC-SRRs) and a comb-drive actuator. In the structure, the coupling between two arrays of SRR is modulated with the lateral displacement exerted by the comb-drive actuator. In the spectrum of BC-SRRs, two resonant modes can be observed, i.e. symmetric mode and anti-symmetric mode, due to the strong inductive and capacitive coupling. Over 20 μ m lateral displacement, the symmetric mode blueshifts ~60 GHz and the anti-symmetric mode redshifts ~50 GHz. The amplitude of the transmission at 1.03 THz is modulated by 69%. We can realize a large tuning range with structure optimization, thereby enabling the applications including THz modulators and chemical sensing.

INTRODUCTION

Over the past decade, metamaterials (MMs) have been proved to be a scheme to engineer the electromagnetic response of materials through resonance excitations [1]. They have many remarkable applications, such as negative refractive index [2], super lensing [3], perfect absorption [4], and cloaking [5], in information processing [6], chemical and biology sensing [7], and security [8, 9].

In order to enhance the functionalities of MMs, a number of approaches have been employed to make the MMs reconfigurable. These include electrical gating [10], photo-excitation [11], thermal tuning [12], modulation of near-field coupling [13] and structural and mechanical reconfiguration [14]. Among these methods, tuning the near field coupling is of interest because of its high efficiency, stability and ease of controlling [15]. One coupled MM structures is broadside-coupled split ring resonator (BC-SRR), including two split ring resonators (SRRs) arrays are stacked vertically and rotated 180° to each other. Recent study shows that the lateral displacement between the two SRR layers will tune the MM resonance frequency [13].

Micromachined electrostatic actuators provide an intuitive path to realize the functional broadside-coupled tunable devices. We demonstrated implantation of the real-time tunable metamaterial based on the BC-SRRs and comb-drive actuators in [16].

In this paper, we report our recent process of the BC-SRRs based metamaterial at THz frequencies. The coupling between the SRRs is optimized with new geometry design and the fabrication process is improved to achieve good alignment in the flip-chip bonding. With the new mechanical structure design, the yield of the functional device is increased as well. With this improvement, the tunability is improved significantly compare to our previous work [16]. The first resonant mode redshifts 60 GHz and the second mode redshifts 50 GHz with 20 μ m lateral displacement achieved by the comb-drive actuator. Meanwhile, the amplitude at 1.03 THz is modulated by 69%.

DESIGN AND FABRICATION

The illustration of the designed tunable MMs is shown in Figure 1. The MMs are composed with two layers of SRRs separated by $20 \ \mu m$ air gap. One layer is on $10 \ \mu m$ thick silicon with

978-1-940470-02-3/HH2016/\$25©2016TRF DOI 10.31438/trf.hh2016.58 electrostatic comb-drive actuators, while the other is fixed on 500 nm thick SiN_x thin film. The unit-cell of the MMs with different x-axis displacement is shown in Figure1 (b). The rings are rotated 180° to each other, forming a broadside coupled configuration. Since the two layers of MMs are closed to each other, the near-field inductive and capacitive interactions between them are strong. The relative shift along x-axis between the arrays, controlled by the applied voltage, can change the coupling between the SRRs. Thus, it modulated the frequency response of the MM.

In this device, the geometries of the two SRRs in each coupled pair are not identical to compensate for the effect of the different substrates. We determine the parameters, as listed in Table 1, using numerical simulation in order to match the resonant frequency of the uncoupled SRRs. The frequency match can improve the coupling efficiency between the SRRs and tunability comparing to the unmatched condition [16]. The simulated transmission spectrum of SRR1 and SRR2 are shown in Figure 2. The dip in the each spectrum corresponds to the LC resonant frequency of the SRR, which is determined by the self-inductance and capacitance of each ring. When the two rings are stacked together, the mutual inductance and capacitance will show up and lead to the frequency split in the spectrum [17], as shown with the red curve in Figure 2.



Figure 1: Illustration of the broad-side coupled tunable metamaterials: (a) the exploded diagram of the tunable metamaterials, in which two chips were bonded together with polyimide spacer; (b) and (c) the unit-cell of the metamaterials with different x-axis shift.

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Table 1: The dimensions of SRRs (unit in µm)

	Periodicity	Side-width	Linewdith	Gap
	(P)	(l)	(w)	(g)
SRR1	58	40	11	16
SRR2	58	40	11	2

The tunable metamaterial was fabricated with micromachining processes as shown in Figure 3, which include two wafer processes and flip chip bonding. An SOI wafer with a 10 µm thick device layer and 2 µm buried oxide layer was used as the first wafer. Both sides of the wafer were coated by 400nm thick low-stress SiNx films grown by LPCVD. With subsequent photolithography and reactive ion etching (RIE), the SiN_x film on the top side was patterned [Figure 3 (a)]. Then, the electrodes were deposited on the exposed silicon using e-beam evaporation of 400 nm Al. The ohmic contact was achieved by annealing at 450 °C in H₂/N₂ ambient. The electrodes were coated with a 150 nm gold thin film after annealing [Figure 3 (b)]. Subsequently, 150 nm thick gold SRRs were patterned via a lift-off process and the device layer was etched using deep reactive ion etching (DRIE) to form the comb-drive actuators and supporting beams [Figure 3 (c) and (d)]. A 600 nm thick SiO₂ passivation layer was deposited on the front side with PECVD in order to protect the patterned device layer in the following steps [Figure 3 (e)]. Next, backside alignment and photolithography using AZ9260 (MicroChemicals, Inc.) was performed and the SiN_x film was etched with RIE, followed by etching through the Si substrate using DRIE [Figure 3 (f) and (g)]. The last step [Figure 3 (h)] was to etch the passivation layer and buried oxide layer with Silox Vapoxy III etchant (Transene, Inc.) to release the movable structure. The second wafer, a single crystal <100> silicon wafer, was coated with low-stress SiN_x film on both sides using LPCVD. Then, 150 nm thick gold SRRs were patterned on the top side with lift-off process [Figure 3 (i)]. Then, the silicon wafer was etched through with KOH wet etching from the window opened on the backside SiNx film [Figure 3 (j)]. After the wafer-level fabrication, the two wafers were diced into chips. For clarity and conciseness, we name the chips from the SOI wafer as chip 1 and the chips from the silicon wafer as



Figure 2: The simulated response of the SRR1, SRR2 and BC-SRRs with the geometries listed in Table 1. The resonant frequencies of the SRR1 and SRR2 are matched. The vertical distance between the SRRs is $20 \ \mu m$ in the BC-SRRs. Frequency splitting shows up in the BC-SRRs.

chip 2. Polyimide bonding pads were patterned on chip 2 via photolithography of the photosensitive polyimide (HD8820, HD Microsystems). Finally, chip 1 and chip 2 were bonded together using a flip chip bonder (FC150, Suss MicroTec AG) under 200 g force and 175 °C temperature. The bonded chips were connected with a printed circuit board via bonding wire to apply the voltage.

The fabricated device is shown in Figure 4 (a). Details of the chip 1 are shown in Figure 4 (b) and (c). The microscope images of the bonded device [Figure 4 (d) and (e)] show that the SRRs are well-aligned in y direction and the misalignment in the x direction can be controlled with the comb-drive actuated lateral displacement.



Figure 3: Fabrication process of the broad-side coupled tunable metamaterials: (a)-(h) the first (SOI) wafer process; (i)-(j) the second (silicon) wafer process; (k) the flip-chip bonding. The bonded chips were connected with a printed circuit board via bonding wire to apply the voltage



Figure 4: Fabricated device.(a) The flip-chip bonded tunable metamaterial; (b) and (c) SEM images of the comb-drive chip;(d) the SRRs with 0μ m lateral displacement, where the SRRs are aligned; (e) the SRRs with 20μ m relative lateral displacement, where the SRRs are misaligned.

EXPERIMENT AND RESULTS

The electromagnetic response of the real-time tunable metamaterial was characterized by THz time domain spectroscopy (THz-TDS), as shown in Figure 5. A 1 kHz Ti:sapphire femtosecond laser producing 1.55 eV near-infrared pulse (800 nm, 3 mJ, 35 fs) was utilized to pump the ZnTe crystal to generate THz pulses. The THz pulses were focused on the sample or reference with a parabolic mirror. The electric field of the THz pulses was polarized perpendicular with the gaps of the SRRs. The transmitted THz pulses were collimated and focused in the detection ZnTe crystal, with which the electro optical (EO) sampling based on Pockels effect can detect the electric field strength of the THz pulses. We can obtain the time domain signal of the sample and reference individually with this scheme. Then, a Fourier transform was performed on the time domain signal to obtain the frequency spectrum of the reference and metamaterial samples. The transmission spectrum of the sample was calculated by normalize the spectrum of metamaterial sample to the reference. A DC voltage power supply was used to drive the comb-drive actuator. By sweeping the applied voltage, the spectra of the metamaterial were measured at different lateral displacements.

The measured transmission spectra of the metamaterial with different lateral displacements are shown in Figure 6 (a). When the lateral displacement is 0 μ m, there are two resonant modes with resonant frequencies of 1.03 THz and 1.23 THz. With the increasing lateral shift, the first resonant mode shifts higher frequency (blueshift) while the second mode shifts to lower frequency (redshift). With the lateral displacement up to 20 μ m, the first mode blueshifts by 60 GHz and the second mode redshifts by 50 GHz. At the same time, the transmission amplitude at 1.03 THz is tuned from 0.2 to 0.65, as shown in Figure 6 (b).

DISCUSSION

The behavior of the BC-SRRs can be described with different theories [18], including coupled mode theory and mutual capacitance and inductance model [19]. We will use the third method to interpret the presented tunable metamaterial.

We simulated the transmission spectra and surface current distributions of the BC-SRRs with different lateral displacements using CST Microwave Studio. In the simulation, unit-cell boundary condition was applied. The spectra agree [Figure 7 (a)] well with the experimental results [Figure 6 (a)]. When the lateral shift is 0 μ m, the surface currents are in the same direction at the first mode and in the opposite direction at the second mode, as shown in Figure 7 (b) and (c). We define the first mode as symmetric mode and the second mode as the anti-symmetric mode. The mutual inductance and capacitance are positive for the symmetric mode but negative for the



Figure 5: Terahertz time domain spectroscopy for the tunable metamaterial characterization.

anti-symmetric mode due to the surface current and charge distribution. With increasing lateral displacement, the absolute value of the mutual inductance and capacitance decrease. As a result, the total inductance and capacitance of the symmetric mode decrease, leading to a blueshift in the resonant frequency; at the same time, the total lumped L and C increase, resulting in the redshift of the second mode. When the lateral displacement is about 20 μ m, the two resonators oscillate individually without any coupling as shown in Figure 7 (d) and (e). The simulated transmission spectra agree well with the experimental data and support our qualitative analysis.

CONLCUSION

We presented our recent process on the dynamically tunable terahertz metamaterial based on broadside-coupled split ring resonators (BC-SRRs). The BC-SRRs are with a symmetric mode and an anti-symmetric mode. With the optimization of the structural design, we achieved 60 GHz redshift of the symmetric mode and 50 GHz blueshift of the anti-symmetric mode. At the same time, 69% modulation depth was demonstrated at 1.03 THz. The tunable metamaterial has potential applications for dynamic modulation of the THz radiation and spectral sensing of chemicals.



Figure 6: (a) The experimental transmission spectra of the real-time tunable metamaterial. With the increase of the lateral displacement, the two resonance modes merge together due to the decrease of the coupling. (b) The magnitude of transmission at 1.03THz with different lateral displacement. The transmission is tuned by 69%.



Figure 7: (a) The simulated transmission spectrum at different lateral displacement. (b) - (e) the surface current at the corresponding resonance frequency as shown in (a). Strong coupling exists between the broadside coupled SRRs in (b) and (c), while little coupling in (d) and (e).

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A HIGHLY SENSITIVE SMALL-FOOTPRINT VOX-BASED MICRO-PIRANI GAUGE FOR IN-SITU MONITORING OF VACUUM WAFER-LEVEL PACKAGED BOLOMETERS

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ABSTRACT

We have designed, fabricated, and tested micro-Pirani gauges, optimised for hermeticity monitoring in wafer-level packaged bolometer dies. Tests show that the gauges have a resolution of at least 1 mTorr. Furthermore, our gauges have a significantly smaller footprint than gauges previously published which achieve a similar resolution. This is due to the use of high TCR (temperature coefficient of resistance) vanadium oxide and an optimised compact design. These features make our Pirani gauges ideal for hermeticity monitoring in bolometer dies, and we have demonstrated this capacity by performing leak rate tests on wafer-level packaged dies with our gauges.

INTRODUCTION

Bolometers and infrared (IR) imaging, initially developed for military applications, are currently attracting ever-greater interest for consumer applications, as evidenced by recently released cameras, designed to be connected to a smartphone [1], [2]. A bolometer is essentially an IR-sensitive thermistor, so it must be packaged under vacuum to suppress heat loss through air, generally at a pressure on the order of 10 mTorr [3], [4].

Cost of the device is critical, and one of the key enabling technologies for reducing fabrication costs of bolometers is to replace die-level packaging with wafer-level packaging (WLP) [3], [5]. The seal must be hermetic to avoid leaks and the ensuing reduction of performance, so a method of monitoring pressure inside the bolometer throughout the lifetime of the device is extremely useful. Pirani gauges are a good choice for vacuum monitoring of bolometers: being pressure-sensitive thermistors, they are functionally similar to bolometers, so can easily be integrated in the same device using the same fabrication process.

Microfabricated Pirani gauges have been demonstrated before, but those with the required resolution have a footprint greater than 100 x 100 μ m². Given that another key factor for costs is the device size, it is essential that the Pirani gauge has as small a footprint as possible to avoid wasting valuable die real-estate. We have designed, fabricated, and tested micro-Pirani gauges with a resolution of at least 1 mTorr and sizes between 22 x 22 μ m² and 42 x 45 μ m². These gauges are significantly smaller than any other previously published gauges with similar resolution. We have also used these micro-Pirani gauges to monitor hermeticity inside WLP dies.

THEORY OF PIRANI GAUGES

Micro-Pirani gauges are generally implemented using a suspended platform. On this platform there is a resistive sensing element which is heated by the Joule effect. The equilibrium temperature of the gauge depends on the heat loss, which occurs mainly via solid conduction through the structure suspending the gauge, and through the surrounding gas. The thermal conductance of the surrounding gas varies with pressure, as long as the mean free path is greater than the gap between the gauge and the substrate, so the temperature of the gauge depends on pressure. The gauge temperature is determined by measuring the electrical resistance of the suspended heating element, which varies due to its TCR (temperature coefficient of resistance). In our gauges there is a layer of vanadium oxide on the suspended platform which serves as the resistive sensing element, and since vanadium oxide has a negative TCR, resistance decreases as pressure decreases.

Research on micro-Pirani gauges initially focussed on addressing one of the main disadvantages of conventional Pirani gauges, by extending their upper pressure limit up to and beyond atmospheric pressure. This was achieved by reducing the gap between the suspended platform and the substrate [6], down to values as low as 50 nm [7]. More recently, it has been proposed to



Figure 1: a) Optical microscope image showing a top-view of a 22 x 22 μ m² Pirani gauge. The dotted lines show the limits of the device. b) Tilted scanning electron microscope (SEM) image of a 26 x 29 μ m² gauge.

978-1-940470-02-3/HH2016/\$25©2016TRF DOI 10.31438/trf.hh2016.59 use micro-Pirani gauges to monitor hermeticity of vacuumpackaged MEMS [8], so sensitivity down to the mTorr range is required.

EXPERIMENTAL DETAILS

Fabrication and design of the micro-Pirani gauges

We designed micro-Pirani gauges with four different sizes, between 22 x 22 μ m² and 42 x 45 μ m². The legs suspending the platform and sensing element are long and narrow to reduce thermal conductance of the gauges, thus increasing their resolution at low-pressure. The gauges were successfully fabricated on 200 mm wafers using an industrial CMOS-compatible process.

The main materials used were silicon nitride as the structural material, and high-TCR vanadium oxide as the sensing material on the suspended platform. The latter material was chosen as it is the most commonly used material for commercial micro-bolometers. Certain wafers were then wafer-level packaged by bonding them to a second wafer using a metal solder, in order to enclose the Pirani gauges in a cavity under vacuum. Fig. 1 shows images of two examples of Pirani gauges which were fabricated, with footprints of $22 \times 22 \ \mu\text{m}^2$ and $26 \times 29 \ \mu\text{m}^2$.

Pirani gauge characterisation methods

All electrical tests were carried out by driving the Pirani gauges with a current source and measuring their output voltages. To test the response of the gauges as a function of pressure, the gauges were placed in a vacuum prober, in which the pressure could be controlled between 10 μ Torr and atmospheric pressure. Pressure was measured using an ion gauge or a capacitive diaphragm gauge, depending on the vacuum level.

Pirani gauges can also be used to measure leak rates into a WLP die, by measuring the evolution of pressure inside the die versus time. This test is usually accelerated using "bombing", in other words, by placing the die in a high pressure environment. We carried out leak rate measurements in a custom-built bombing chamber, pictured in Fig. 2, under a helium atmosphere.



Figure 2: 200 mm bombing chamber used for accelerated leak rate measurements at the wafer-level with our micro-Pirani gauges.

RESULTS AND DISCUSSION

Electrical measurements of unpackaged micro-Pirani gauges

The resolution of the Pirani gauges was determined by measuring the voltage across the gauges driven at constant current, while increasing pressure in the vacuum prober. Fig. 3 shows these measurements for two different gauge sizes and we can observe that in both cases the resolution is better than 1 mTorr.

Fig. 4 shows resolution versus device footprint for the smallest Pirani gauge which we fabricated ($22 \times 22 \mu m^2$), compared to other Pirani gauges published in the literature [4], [6], [7], [9]–[16]. This clearly shows that a resolution of 1 mTorr has never been achieved



Figure 3: Measurement of the response of two Pirani gauges at constant current as a function of pressure. The 22 x 22 μ m² gauge has a resolution of 1 mTorr and the 26 x 29 μ m² gauge has a resolution of less than 1 mTorr.

with such a small footprint; indeed the footprint of devices with a comparable resolution is at least one order of magnitude higher. Previously published work has focussed on either achieving very high resolution gauges at the cost of a larger footprint, or gauges with very small size, but which had lower resolution. The performance of our gauges was largely achieved through the use of vanadium oxide as a sensing material. Indeed, the TCR of vanadium oxide is an order of magnitude higher than that of metals, which are the most commonly-used material for sensing elements in previously published work. The footprint of the gauges was reduced by optimising their design to make them as compact as possible.



Figure 4: Resolution versus footprint for our Pirani gauge and a selection of gauges from the literature. Our gauge achieves a resolution of 1 mTorr, but with a significantly smaller footprint than other published devices. The colour of the symbols indicates the sensing material which was used.

Fig. 5 shows the results of resistance versus current measurements performed at different constant-pressure values. These measurements demonstrate the wide operation range of the Pirani gauges, which have an upper detection limit of around 100 Torr. While this is much greater than the desired operating pressure for bolometers, this can be useful for troubleshooting hermeticity problems caused by leaks or outgassing for example.



Figure 5: Pirani gauge resistance versus driving current, for different pressures. This illustrates the typical pressure-dependent behaviour of a Pirani gauge: as pressure decreases, a given current will cause a greater temperature rise, leading to a greater resistance decrease (due to the negative TCR of vanadium oxide).

Using the micro-Pirani gauges to monitor hermeticity of WLP dies

Fig. 6 shows resistance versus current measured on a WLP Pirani gauge before and after bombing for 9 days under 5 atm of helium. We can see that for a given current, resistance is higher after bombing, and as explained earlier, this means that the pressure inside the die has increased, due to helium leaking through the seal. Based on the estimated pressure values, the standard leak rate is on the order of 10^{-12} Torr.L/s.

The maximum bombing pressure recommended to avoid creating additional leakage paths in the seal is 5 atm [17], but bombing time can easily be increased. Furthermore, the use of helium also significantly increases the sensitivity of the measurement. Leak rates are usually reported as *standard leak rates*, i.e. the leak rate which would be measured if the die was exposed to air under a ΔP of 1 atm. To convert a standard leak rate, \mathcal{L} , to a leak rate, R, obtained with another gas at a different pressure and temperature, the following equation is used [18]:

$$R = \mathcal{L} \sqrt{\frac{M_{air}}{M_{gas}} \frac{T_{gas}}{T_{293K}}} \frac{P_{ext} - P_{die}}{P_{atm}}$$
(1)

where M_{air} and M_{gas} are the atomic weights of air and the gas used, T_{gas} is the temperature of the gas, P_{ext} is the external pressure, P_{die} the pressure inside the die, and P_{atm} the standard atmospheric pressure. Therefore, by using helium at 5 atm, the leak test is accelerated by a factor of 13. Furthermore, the sensitivity of the Pirani gauges is improved, given that the thermal conductivity of helium is six times higher than that of air. Therefore, by carrying out bombing during one month, we estimate that we will be able to measure leak rates as low as 10⁻¹⁵ Torr.L/s, which compares favourably with RGA (residual gas analysis) measurements.



Figure 6: Resistance versus current for a WLP Pirani gauge, before and after bombing during 9 days at 5 atm of helium. After bombing, the curve has shifted upwards due to the pressure increase inside the die caused by helium leaking in.

CONCLUSIONS

We have successfully designed and fabricated micro-Pirani gauges on 200 mm wafers, using a CMOS-compatible industrial process. We also packaged devices under vacuum, using waferlevel packaging. Electrical testing shows that the gauges have a resolution of at least 1 mTorr, which has never been achieved previously with such a small footprint. This performance was obtained thanks to the use of high-TCR vanadium oxide and the use of this material also makes these Pirani gauges an ideal choice for in-situ monitoring of WLP bolometers. We have also used these gauges to carry out leak rate measurements to evaluate the hermeticity of WLP dies. Pirani gauges are usually calibrated as a function of pressure before vacuum packaging, or one die is calibrated and used for all other dies from the wafer [4]. Both approaches are time-consuming, and even with a well-controlled process, the latter approach increases measurement uncertainty [4]. Therefore, we are currently developing methods for measuring pressure within a packaged die which do not require prior calibration versus pressure.

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ALL FLEXIBLE GRAPHENE STRAIN SENSORS WITH LIQUID METAL INTERCONNECTORS FOR EMBEDDED STRUCTURAL HEALTH MONITORING AND SKIN TACTILE SENSING

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ABSTRACT

This paper reports on the development of graphene strain sensors made of all flexible materials, including graphene, liquid metal, and elastomer. The sensors are featured with a flexible interconnector design using liquid metal as an interconnecting material for graphene-based sensing elements. This design allows enhancing flexibility and reliability of the sensors compared with existing hard metal based interconnection methods. A unidirectional strain sensor and a multidirectional rosette-type strain sensor are developed by encasing patterned graphene and liquid metal in an elastomer. We explore the applications of the unidirectional sensor for structural health monitoring by attaching the sensor on the surface of concrete, and for tactile sensing by attaching the sensor on human skin. This work represents an exploratory effort on using liquid metal interconnectors for graphene-based sensors to obtain full structural flexibility.

INTRODUCTION

Strain sensors are used to monitor structural damage, evaluate material fatigue properties, and track human body movement. Recently, graphene has been investigated as a strain sensing material due to its high piezoresistivity, carrier mobility, and Young's modulus [1-3]. Significant efforts have been made to develop various graphene-based flexible strain and pressure sensors [4-8]. While these existing graphene-based sensors are regarded as flexible devices, electrical interconnections for these devices are generally obtained using hard metal/alloy materials [9]. This has compromised overall structural flexibility of the devices and has caused a long-term reliability issue due to possible local strains introduced to the interconnecting areas during sensing and/or actuation.

Many liquid metals and their alloys are found to be non-toxic, non-evaporative, and melt at room temperature. They usually have comparable electrical conductivities and better wetting properties compared with mercury [10]. As an electrical interconnecting material, liquid metal has been used in skin type temperature and pressure sensors with a mixture of PDMS and carbon nanotubes [11-14], and also has been reported to accommodate thermal stresses and provide re-workability in case of chip failure or upgrade [15]. In addition, liquid metals have been used as electrical interconnects for integrated circuits to overcome some packaging challenges in wire bonding and flip-chip bonding [16].

In this paper, we reports flexible graphene strain sensors using liquid metal as interconnectors for enhanced structural flexibility and reliability. The sensors are formed by encasing graphene sensing elements in an elastomer with built-in microfluidic channels filled with liquid metal. By integrating graphene and liquid metal in an elastomer, the sensor gains full structural flexibility. By stacking multiple flexible sensors layer by layer together with a rosette structure, the obtained rosette sensor can measure strains coming from multiple directions. To explore potential applications of these sensors, we demonstrate feasibility to monitor strain of concrete using a surface attached unidirectional sensor, and to track motion behaviors of human body using a sensor attached on human skin.

DEVICE FABRICATION

Three major steps are involved in fabricating graphene sensors, including patterning and transferring of graphene, formation of microfluidic channel, and formation of liquid metal interconnectors (Fig. 1). First, photoresist was spin coated on the surface of a nickel foil deposited with ~105 nm thick graphene, with 500 rmp for 5 sec and then 4000 rmp for 45 sec. Subsequently, photolithography was used to form patterns in photoresist. Then, oxygen plasma was used to etch away unwanted graphene from the nickel foil, with the etching time of 50 mins. After that, polydimethylsiloxane (PDMS, Sylgard, 15:1 part A:B) was degassed to remove bubbles. 15 g of the PDMS precursor was then poured into a 3 inch diameter petri-dish and baked at 85 °C for 40 min. The cured PDMS slab was cut and peeled out of the petri dish and then immediately stamped onto the fresh surface of the patterned graphene. Then, the entire substrate was immersed into 5 wt % FeCl3 solution for 24 hours or until the nickel foil was fully etched.



Figure 1: Process flow for an array of graphene-based strain sensors. The process mainly involves embedding patterned graphene into PDMS, and flowing liquid metal into microfluidic channels to realize interconnection.

In order to allow liquid metal to access and directly contact the patterned graphene, microfluidic channels were formed by soft lithography. In this process, photoresist patterns were fabricated on a glass slide as a master mold, followed by pouring PDMS solution (Sylgard, 15:1 part A: B) and baking at 70 °C for 40 min. After the PDMS layer with patterned channels was peeled off from the mold, accessing holes were manually punched through the PDMS channel layer. Subsequently, the PDMS channel layer and the previously formed PDMS layer with graphene patterns were bonded with the

978-1-940470-02-3/HH2016/\$25©2016TRF DOI 10.31438/trf.hh2016.60 Solid-State Sensors, Actuators and Microsystems Workshop Hilton Head Island, South Carolina, June 5-9, 2016 help of oxygen plasma treatment. Then, Gallium-Indium eutectic or EGaIn was injected into the channels using syringe needles. Here, EGaIn is considered as a promising substitution of mercury because of its liquid state in room temperature and low toxicity. The liquid metal was able to reach and cover the contact pads of the graphene patterns. Lastly, metal wires were inserted into the channels through the accessing holes to realize electrical connections to external circuits and these accessing holes were sealed by thermally curing PDMS precursor solution dropped at the holes.

By using this fabrication method, a graphene based strain sensor capable of detecting strain in a single direction was formed. A multidirectional rosette type strain sensor (Fig. 2) employed three unidirectional strain sensors arranged with 60 degrees angle between neighboring sensors. To reduce the total thickness of the rosette sensor, instead of simply stacking three unidirectional graphene strain sensors vertically, the PDMS layer for the liquid metal channels and the other PDMS layer for the patterned graphene were combined together. In other words, the PDMS layer containing the liquid metal channels which conduct strain signals for the lowest layer graphene, also served as the middle layer graphene substrate.



Figure 2: Process flow of a stacked rosette strain sensor

RESULTS AND DISCUSSION

Due to the flexible nature of silicone, liquid metal, and graphene, the sensors could be deformed into different shapes without sacrificing durability, conductivity, and robustness of the sensors (Fig. 3). For example, mechanical twisting or bending at the sensing area did not affect electrical interconnection between liquid metal and graphene patterns.



Figure 3: Photos showing interconnecting, external wiring, bending, and twisting of the graphene-liquid metal strain sensors.

Different tensile strains were applied to the sensor by stretching the sensor using a motored linear stage. The resistance value of the sensor at a given strain was recorded when it became stable. By releasing the sensor to its starting zero strain state, the resistance of the sensor went back to its initial value. Fig. 4a shows the resistance is varied between ~245 and ~295 Ω as the applied strain increases up to 9%.

Fig. 4b shows the resistance responses of both liquid metal and graphene elements to the same levels of applied strains. We used gauge factor to evaluate how sensitive the two elements responded to applied strains. Here, gauge factor is defined as (dR/R)/(dL/L), where R and L represent the resistance and length of the sensor, respectively. The results demonstrate that the gauge factor of the graphene element of the sensor is ~1.5. As the strain increased, the resistance of the sensor grew in a linear fashion. However, due to possessing an almost zero gauge factor, the liquid metal element of the sensor to applied strain.



Figure 1: (a) Resistance response of the unidirectional strain sensor to applied strains during the cyclic stretch and release measurement. (b) Relative resistance change of the graphene sensor as a function of applied strain.

For the multidirectional rosette sensor (Fig. 5a), the normal strain ε_{θ} at any angle θ from the major principal axis can be expressed as [17]:

$$\varepsilon_{\Theta} = \frac{\varepsilon_{x} + \varepsilon_{y}}{2} + \frac{\varepsilon_{x} - \varepsilon_{y}}{2} \cos 2\Theta. \tag{1}$$

The strain on each rosette gauge can be represented in terms of the principal strain on x and y axis with corresponding rotation angles θ , θ +60 degrees, and θ +120 degrees. Using the following strain transformation equations (Eq. 2a-2c), it is possible to determine the directionality of the strain applied.

$$\varepsilon_{s1} = \frac{\varepsilon_x + \varepsilon_y}{2} + \frac{\varepsilon_x - \varepsilon_y}{2} \cos 2\Theta$$
(2a)

$$\varepsilon_{s2} = \frac{\varepsilon_x + \varepsilon_y}{2} + \frac{\varepsilon_x - \varepsilon_y}{2} \cos 2(\Theta + 60^0)$$
(2b)

$$\varepsilon_{s3} = \frac{\varepsilon_x + \varepsilon_y}{2} + \frac{\varepsilon_x - \varepsilon_y}{2} \cos 2(\Theta + 120^0)$$
(2c)

Therefore, by measuring the strain values of ε_{s1} , ε_{s2} , and ε_{s3} , we could find the angle of θ with the following equation [18]:

$$\theta = \left(\frac{1}{2}\right) \tan^{-1} \left(\left(\sqrt{3}(\varepsilon_{s2} - \varepsilon_{s3})\right) / (2\varepsilon_{s1} - \varepsilon_{s2} - \varepsilon_{s3}) \right)$$
(3)



Figure 2 (a) S1, S2, and S3 graphene sensors stacked as a rosette gauge system. (b) Schematic representation of graphene strain sensor in rosette manner indicating the direction of principle axis (c) Variation of normalized resistance with respect to stretching up to 2.1% by a force applied in the direction 3° to Direction 1.

Fig. 5c demonstrates resistance responses of the three sensing elements in the rosette sensor to applied strains. The three sensing elements, S_1 , S_2 , and S_3 were arranged along the directions 1, 2 and 3, respectively. When the external strain of 2.1% was applied to the device in the direction 3° to Direction 1, the resistance of S_1 , S_2 , and S_3 was changed by 21.5%, 6.2% and 4.5%, respectively. As discussed, by knowing the strain values of the three elements, the principal strain and its orientation with respect to the rosette axis could be estimated. Here, the principal strains ϵ_x and ϵ_y and their angle of orientation were calculated by inserting the three strain values of the three elements to Eq. (3). The value of θ was found to be 0.045 (rad) or 2.604° to Direction 1.

The use of liquid metal interconnects in this graphene based strain sensor has several advantages. Firstly, the entire flexible structure allows the sensor to be installed in varies environments, such as the curved surface or moveable object. Also, its flexible structure decreases the fail rate caused by additional applied force on contact. Secondly, the liquid metal interconnectors keep the high flexibility around the detecting area, thus keeping the sensor's max sensitivity with minimal effect of less deformation due to solid contacting wires. To demonstrate these capabilities, liquid metal interconnects graphene film sensor were embedded inside concrete and bonded on human skin as two specific application examples.

Fig. 6 demonstrates the installation and testing of the unidirectional graphene strain sensor with liquid metal interconnect on concrete strain sensing application. The sensor was horizontally attached at the surface of a concrete cylinder. An external force was vertically loaded onto the cylinder. With a higher load, the concrete cylinder react an expansion in the horizontal plane. Therefore, the sensor felt a stretch strain while the concrete was expanded. The resistance of the sensor and the force load on the concrete were both recorded. The testing result indicates our sensors reacts in the similar pattern as the given load.

(a)



Figure 3: (a) Setup for monitoring strain changes of a concrete cylinder during loading and unloading processes. (b) Relative resistance change and load on the concrete cylinder over a period of time (\sim 140 sec).

Fig. 7 provides the demonstration of the unidirectional sensor for tracking wrist motion. As the wrist bent up and down, the strain sensor changed its resistance. A larger bending degree led to a higher peak on resistance. The repeatability measurement (~200 times) for this tactile sensor indicates that the device could provide good repeatability with a low relative standard deviation (RSD: 2.5%) of peak resistance, which is believed due to good structural durability of the graphene sensing element and the liquid metal interconnectors.



Figure 7: (a) Strain sensor bonded to waist to monitor waist motion. (b) Resistance response to waist motion. Resistance reflects the tensile strain and further indicates the different angles of waist movement.

CONCLUSIONS

This paper demonstrates a graphene based all flexible sensor which is built by: multilayer of graphene, liquid metal, and PMDS. We take advantage of graphene with its piezoresistive properties, which translates a mechanical displacement into an electrical signal. We take benefit from liquid metal interconnect for the better flexibility. Due its good repeatability and its linear strain response, this sensor will be useful for multiple applications, such as monitoring structural deformations on concrete, and tensile measurement on human skin.

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AN ANISOTROPIC-WET-ETCHED PITCH OR ROLL MODE-MATCHED GYROSCOPE WITH SLANTED QUADRATURE-CANCELLATION ELECTRODES

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ABSTRACT

This paper presents the design, fabrication, and preliminary characterization of a novel anisotropic-wet-etched silicon pitch or roll gyroscope fabricated on a silicon-on-insulator (SOI) wafer without using the deep reactive ion etching (DRIE) process. The novel gyroscope features slanted electrodes with sub-micron gaps, which for the first time, enables electrostatic quadrature tuning in out-of-plane gyroscopes. Complete quadrature cancellation and perfect mode-matching of the drive and sense modes at 200 kHz are demonstrated in fabricated devices. Sensitivity characterization measures a scale factor of 35.4 pA/°/s under quadrature-cancelled mode-matched condition, showing ~10× improvement in scale factor compared to operation without quadrature compensation.

INTRODUCTION

MEMS gyroscopes offer self-contained rotation measurement of an object. The most critical application of MEMS gyroscope is to enable miniaturized inertial navigation systems (INS) for personal navigation [1-3]. Personal INS requires tri-axial rotation sensing with high-performance gyroscopes. Different types of MEMS yaw gyroscopes have demonstrated promising performance [4-6]. However, developing high-performance out-of-plane pitch and roll gyroscopes is known to be very challenging.

The performance of a gyroscope can be evaluated through its signal to noise ratio (SNR). The SNR of a gyroscope can be increased by noise reduction and improvement in rate sensitivity. The best way to achieve large sensitivity is through mode-matched operation, with the drive and sense modes having the exact same resonance frequency. When mode-matched, Coriolis force excites the sense mode at its resonance frequency, leading to a Qamplified sense response. However, perfect mode-matching may not be possible due to cross-coupling of resonance modes (i.e. quadrature error) caused by fabrication non-idealities. Quadrature error breaks the eigenvalue-degeneracy of the equations of motion, resulting in a veering phenomenon [7], which appears as a minimum obtainable frequency-split between the drive and sense modes of a gyroscope. In addition, quadrature error provides a path through which drive-loop noise is carried to the sense mode and becomes a major noise contributor in sense output signal.

Considering both effects, quadrature error significantly degrades the output SNR and must be minimized to achieve best performance in a MEMS gyroscope. This is generally done by electrostatic quadrature tuning in mode-matched yaw gyroscopes [8]. However, in out-of-plane gyroscopes that are used for pitch and roll detection and fabricated using wafer level processing and DRIE techniques, mode alignment or quadrature tuning electrodes are typically unavailable, which makes quadrature error the biggest obstacle in realizing high-performance pitch and roll gyroscopes.

In this paper, we show that slanted electrodes along wetetched (111) surfaces can be used for efficient quadrature nulling in out-of-plane pitch and roll gyroscopes. In addition, we extend the use of anisotropic-wet-etching to the fabrication of the entire vibrating body. By doing so, we not only make the frequency split between drive and sense modes insensitive to thickness variations of the SOI device layer, but also eliminate the use of expensive DRIE in device fabrication, reducing both process variation dependency and fabrication cost of the gyroscope.



Figure 1: Electrodes in out-of-plane gyroscopes. (a) Conventional horizontal and vertical electrode and (b) novel slanted electrode.

DESIGN AND SIMULATION

Slanted electrode for quadrature tuning

A general MEMS gyroscope with stiffness non-ideality can be represented by a two-degree-of-freedom spring-mass-damper system with the equations of motion described by:

$$m\begin{bmatrix} \ddot{q}_1\\ \ddot{q}_2 \end{bmatrix} + \begin{bmatrix} \dot{q}_1\\ \dot{q}_2 \end{bmatrix} \begin{bmatrix} b_{11} & 2\lambda m\Omega\\ -2\lambda m\Omega & b_{22} \end{bmatrix} + \begin{bmatrix} q_1\\ q_2 \end{bmatrix} \begin{bmatrix} k_{11} & k_{12}\\ k_{21} & k_{22} \end{bmatrix} = \begin{bmatrix} F_d\\ 0 \end{bmatrix}, (1)$$

where q_1 , q_2 , k_{11} , k_{22} are displacements and effective stiffness of the drive and sense modes, respectively; k_{12} and k_{21} are cross-coupling spring constants, causing quadrature errors. The eigenfrequency of Eqn. (1) is given by:

$$\omega_{1,2} = \sqrt{\frac{1}{2m} \left(k_{11} + k_{22} + 2k_{12} \pm \sqrt{\left(k_{11} - k_{22}\right)^2 + 4k_{12}^2} \right)}$$
(2)

For an ideal mode-matched gyroscope, the stiffness matrix K_m is a scalar matrix with $k_{11}=k_{22}=k$ and $k_{12}=k_{21}=0$, which gives degenerate eigen-frequencies $\omega_1=\omega_2=(k/m)^{1/2}$. In practice, $k_{11}\neq k_{22}$ and $k_{12}=k_{21}\neq 0$, which results in divergence of frequencies. To compensate for this non-ideality, electrostatic tuning is usually used. In capacitive transduction, electrical energy stored in the capacitor between vibrating structure and a fixed electrode is a function of q_1 and/or q_2 , which gives rise to an electrostatic stiffness matrix:

$$\boldsymbol{K}_{e} = -\begin{bmatrix} \frac{\partial^{2} U_{e}}{\partial q_{1}^{2}} & \frac{\partial^{2} U_{e}}{\partial q_{1} \partial q_{2}} \\ \frac{\partial^{2} U_{e}}{\partial q_{1} \partial q_{2}} & \frac{\partial^{2} U_{e}}{\partial q_{2}^{2}} \end{bmatrix} = \frac{-V^{2}}{2} \begin{bmatrix} \frac{\partial^{2} C(q_{1}, q_{2})}{\partial q_{1}^{2}} & \frac{\partial^{2} C(q_{1}, q_{2})}{\partial q_{1} \partial q_{2}} \\ \frac{\partial^{2} C(q_{1}, q_{2})}{\partial q_{1} \partial q_{2}} & \frac{\partial^{2} C(q_{1}, q_{2})}{\partial q_{2}^{2}} \end{bmatrix}$$
(3)

The overall stiffness of a gyroscope is the superposition of K_m and K_e from all electrodes. By changing tuning voltages at different electrodes, the overall stiffness can be adjusted. However, electrostatic tuning in pitch and roll gyroscopes using in-plane and out-of-plane modes has limited efficiency. Conventional gyroscopes fabricated with directional etching allow only vertical and horizontal electrodes (Fig. 1a). Vertical capacitance has negligible change under out-of-plane displacements and horizontal capacitance has negligible change under in-plane displacements. Consequently, any electrode combination gives a capacitance as an uncorrelated function of in-plane and out-of-plane displacements, namely, $C(q_1,q_2)\approx C_1(q_1)+C_2(q_2)$. For example, in a right-angle electrode, the electrical energy neglecting the small contribution from fringing field is given by:

$$U_e \approx \frac{V^2}{2} \left(C_{vertical} + C_{horizontal} \right) = \frac{\varepsilon_0 V^2}{2} \left(\frac{A_h}{g_h} + \frac{A_v}{g_v} \right)$$
(4)

978-1-940470-02-3/HH2016/\$25©2016TRF DOI 10.31438/trf.hh2016.61 Solid-State Sensors, Actuators and Microsystems Workshop Hilton Head Island, South Carolina, June 5-9, 2016 In order to achieve efficient tuning, small gap sizes and large transduction areas are generally used for the electrodes. Compared to the gap size, the area has negligible change under resonant deformations. Assume without the loss of generality that both horizontal and vertical capacitors have same rest gap size g_0 . A series expansion of the electrical energy gives:

$$U_{e} = \frac{\varepsilon_{0}V^{2}}{2} \left(\frac{A_{h}}{g_{0} - q_{1}} + \frac{A_{v}}{g_{0} \mp q_{2}} \right)$$

$$\approx \frac{\varepsilon_{0}V^{2}}{2g_{0}} \left[A_{h} + A_{v} + A_{h} \frac{q_{1}}{g_{0}} \pm A_{v} \frac{q_{2}}{g_{0}} + A_{h} \left(\frac{q_{1}}{g_{0}} \right)^{2} + A_{v} \left(\frac{q_{2}}{g_{0}} \right)^{2} + \cdots \right]^{(5)}$$

According to (3), the corresponding K_e is diagonal:

$$\mathbf{K}_{e} = \begin{bmatrix} k_{e11} & k_{e12} \\ k_{e21} & k_{e22} \end{bmatrix} = \frac{-\varepsilon_{0}V^{2}}{g_{0}^{3}} \begin{bmatrix} A_{h} & 0 \\ 0 & A_{v} \end{bmatrix},$$
(6)

which means in-plane or out-of-plane frequency can be tuned independently, but no quadrature tuning is achieved.

In contrast, anisotropic wet etching of (100) SCS forms slanted sidewalls bounded by (111) crystal planes, which offer a well-defined correlation between in-plane and out-of-plane degrees of freedom. Slanted electrodes along wet-etched (111) surfaces have capacitive gaps affected by both in-plane and out-of-plane motions (Fig. 1b). The energy stored in a slanted electrode is:

$$U_{e} = \frac{\varepsilon_{0}AV^{2}}{2g_{s}} = \frac{\varepsilon_{0}AV^{2}}{2\left[g_{0} - \left(q_{1}\sin\theta \pm q_{2}\cos\theta\right)\right]}$$

$$\approx \frac{\varepsilon_{0}AV^{2}}{2g_{0}}\left[1 + \frac{q_{1}\sin\theta \pm q_{2}\cos\theta}{g_{0}} + \left(\frac{q_{1}\sin\theta \pm q_{2}\cos\theta}{g_{0}}\right)^{2} + \cdots\right]$$
(7)

Correspondingly, the electrostatic stiffness matrix K_e is:

$$\boldsymbol{K}_{e} = \begin{bmatrix} k_{e11} & k_{e12} \\ k_{e21} & k_{e22} \end{bmatrix} = \frac{-\varepsilon_{0}AV^{2}}{g_{0}^{3}} \begin{bmatrix} \sin^{2}\theta & \pm\sin\theta\cos\theta \\ \pm\sin\theta\cos\theta & \cos^{2}\theta \end{bmatrix}$$
(8)

As shown in (8), the electrostatic stiffness matrix has non-zero offdiagonal terms that are suitable for quadrature cancellation. In addition, anisotropic-wet-etching is a self-bounded process. It provides precise control of the surface finish and final geometry, which is critical for gyroscope fabrication.

Gyroscope design and simulation

A novel single-crystal silicon (SCS) pitch or roll gyroscope with slanted quadrature electrode is designed, in which the entire vibrating structure is formed by anisotropic-wet-etching of SCS.

The anisotropic-wet-etched gyroscope features an isosceles trapezoid cross-section (Fig. 2). The top width of the trapezoid is defined by lithography, whereas the bottom width is determined by both the top width and the thickness of the device, which introduces thickness dependency to the in-plane resonant frequency of the gyroscope. As a result, the in-plane and out-ofplane modes track each other over device thickness variations, making the frequency split thickness insensitive.

Figure 2 shows the electrode configuration and mode shapes of the gyroscope. Horizontal and slanted electrodes with submicron gap sizes are used for capacitive transduction. A pair of slanted electrodes is used to actuate the in-plane drive mode at 200 kHz while excitation of out-of-plane sense mode is avoided due to opposite mode symmetry at the electrodes. Another two slanted electrodes are used for drive mode current output and electrostatic quadrature tuning. Horizontal electrodes on top of the device are used for differential sense output and sense mode frequency tuning. The sense mode is designed to have a slightly higher frequency than drive mode to ensure mode-matching capability.



Figure 2: Anisotropic-wet-etched gyroscope geometry and mode shapes. Cross-section shows the shape of the gyroscope body and poly electrode at different locations. The width of the trapezoid structure is coupled to its thickness through the slanting angle.



Figure 3: FEM simulation results: (a) frequency variation with different device thickness; (b), (c) frequency tuning w/o and with electrostatic quadrature cancellation.

The frequency behavior of the gyroscope is simulated using COMSOL Multiphysics. Figure 3a shows the thickness dependency of the operation modes. Across 2μ m thickness variation, the resonance frequencies change significantly, whereas the frequency difference remains relatively unchanged. The mode-matching behavior is shown in Fig. 3b and 3c. Small trench tilting is added intentionally to simulate imperfections causing quadrature. In simulations, the sense mode frequency split. As shown in Fig. 3b, in the presence of quadrature, frequency veering phenomenon is seen and the two eigen-modes cannot be matched. In contrast, with proper quadrature tuning voltage V_Q , quadrature is compensated and perfect mode-matching is achieved.

FABRICATION

Figure 4 shows the fabrication process flow of the anisotropic-wet-etched gyroscope. During the anisotropic wet etching, convex corners need to be protected. A two-mask LOCOS method [9] is used with modifications made to eliminate effects of misalignment. In the self-aligned process, the entire gyroscope pattern is defined on a first thick nitride mask layer. A second thin nitride layer partially covers the pattern and avoids the exposure of convex corners in the first wet etching. The thin nitride mask has irregular-shape openings. Wet etching undercuts the thin nitride and forms trenches aligned to the thick nitride masks will not affect the final device geometry. Following Ref. [9], local oxidation and a second wet-etching step is performed after the first wet-etching,

forming the final SCS structures with intact convex corners. After the device geometry is defined, polysilicon and sacrificial oxide surface micro-machining steps [10] are used to form horizontal and slanted electrodes with sub-micro gaps.



(a) Deposit and pattern thick LPCVD nitride layer to define trench shapes; deposit and pattern thin LPCVD nitride to form self-aligned openings; first KOH wet-etching.

(b) Wet oxidation of SCS to form thick thermal oxide for (111) sidewall protection; blank etch nitride using RIE to remove thin nitride layer; second KOH wet-etching.

(c) Remove nitride and oxide with RIE; deposit and pattern TEOS; wet oxidation to form sacrificial thin oxide.

(d) Pattern thin oxide; deposit and pattern LPCVD polysilicon.

(e) Release device in HF solution and dry with super critical dryer.

TEOS

Figure 4: Fabrication process flow for anisotropic-wet-etched pitch or roll gyroscope.

Prototypes of the gyroscope are fabricated on a 40 μ m (100) SOI wafer. KOH is chosen as the etchant for its high crystalline selectivity. Gap sizes of ~500 nm are defined with sacrificial thermal oxide for top surface and slanted electrodes. Figure 5-7 show the SEM pictures of a fabricated gyroscope. The selfconfined nature of anisotropic wet etching results in very smooth slanted sidewalls. Convex corners are mostly protected although small imperfections are found due to excessive KOH etching (Fig. 6). Sub-micron transduction gaps are successfully formed both on top and on slanted sidewalls of the device with polysilicon electrodes stiff enough for designed operation voltages (Fig. 7).



Figure 5: SEM picture of the wet-etched gyroscope. Electrical connections are labeled on the polysilicon pads, indicating the electrode configuration used in experimental measurements.



Figure 6: Close-up SEM picture of convex corner structure and wet-etched surface finish.



Figure 7: Close-up SEM picture of electrodes and sub-micron capacitive gaps on top and slanted sidewall of the gyroscope.

MEASUREMENTS

The quadrature tuning and mode-matching of fabricated gyroscope is characterized using a 4-port Agilent network analyzer with V_P =23.35V. Drive and sense modes are actuated using separate channels, and the frequency response of each channel is recorded. Figure 8 shows the stiffness tuning behavior of the gyroscope without quadrature cancellation. As expected, veering phenomenon is observed where the eigen-modes push each other away when getting close and switch place without crossing at a common frequency. A minimum frequency-split of ~60 Hz is found by tuning down sense mode frequency with V_T .



Figure 8: Measured sense mode frequency tuning behavior without quadrature tuning, showing frequency veering. (a) Frequency spectrum of drive and sense channels. (b) Peak frequencies.



Figure 9: Measured quadrature tuning behavior of the gyroscope. (a) Coarse quadrature tuning with the presence of large spring mismatch. (b) Fine quadrature tuning after V_T is adjusted to reduce spring mismatch.



Figure 10: Mode-matched drive (blue) and sense (red) resonance peaks at 200,578 Hz.



Figure 11: Measured rate response with mode-matched condition and scale factor comparison between mode-matched and modesplit conditions. TIA with 510 k Ω resistor is used for sense output.

Figure 9 shows the quadrature tuning effect using the slanted electrode. When proper quadrature tuning voltage is applied, the cross-coupling peak drops and the frequency-split reduces. Perfect mode-matching is achieved at 200 kHz by fine tuning V_T and V_Q , showing quality factors of 20k and 13k for drive and sense modes, respectively (Fig. 10).

Angular rate response of the gyroscope is also characterized (Fig. 11). The mode-matched scale factor of the gyroscope is measured to be 35.4 pA/°/s. For comparison, sensitivity is also measured with a minimum frequency-split without quadrature nulling, which gives a scale factor of $3.8 \text{ pA}/^\circ$ /s. Table 1 lists the measurement results for the anisotropic-wet-etched gyroscope.

Table 1: Measured performance parameters

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Mode-matched frequency	200.578 kHz		
Mode-matched Q	$Q_d \sim 20 \mathrm{k}, Q_s \sim 13 \mathrm{k}$		
Δf_{min} without V_Q	~ 60 Hz		
Δf_{min} with V_Q	0 Hz		
Scale factor (w/o V_Q)	3.8 pA/°/s		
Scale factor (with V_Q)	35.4 pA/°/s		

CONCLUSIONS

Quadrature error significantly limits the performance of outof-plane pitch and roll MEMS gyroscopes. Through the use of slanted electrodes enabled by anisotropic wet etching of SCS, we successfully demonstrate electrostatic quadrature cancellation in out-of-plane resonant gyroscopes. The elimination of quadrature allows perfect mode-matching of the gyroscope, resulting in $\sim 10 \times$ improvement in sensitivity. In addition, the anisotropic-wet-etched design reduces the thickness dependency of frequency-split and eliminates expansive DRIE steps, showing promises in improving the yield and fabrication cost of pitch and roll resonant gyroscopes.

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ANALYSIS AND EXPERIMENTAL RESULTS OF UNTETHERED FLIGHT OF STEREOLITHOGRAPHICALLY PRINTED MEMS MICROFLIERS

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ABSTRACT

This paper presents modeling and experimental results of untethered flight of stereo-lithographically printed MEMS microfliers propelled by radiometric forces. Analysis of the physics behind radiometric microscale flight is presented, followed by detailed experimental results. The present work further enhances our understanding of the underlying principles of these microflier designs as well as demonstrates methods to build designs using 3-D stereo-lithography. This paper verifies that the *perimeter* and not the *surface area* of the flier is the key factor in determining the force experienced during flight. Despite differences in underlying physics, similar preference (perimeter over surface area) is observed in biological microscale flight.

INTRODUCTION

Microrobotics is a new field entailing the development of microscale robotic systems, i.e. robotic devices with dimensions on the order of tens or hundreds of micrometers. Due to recent advances in micro electro mechanical systems (MEMS), this last decade has seen the development of several novel microscale mobile robotic systems. These systems include devices such as electrostatically driven stress-engineered microrobots (MicroStress-Bots) [1], resonating stepping magnetic robots [2], stick-slip magnetic walkers [3], rolling magnetic walkers [4], microscrew-based swimmers [5], and biologically-propelled swimmers [6]. Despite the successful development of several mobile microrobotic systems, untethered aerial microrobots with a maximum size of one millimeter cubed have not yet been successfully developed.

While successful aerial microrobots have not been developed in the past, the concept of microscale aerial nodes is not a new idea. Microscale surveillance nodes floating in the air was proposed (but never implemented) as early as in the mid 1980's [7]. Microscale levitating gyroscopes have been implemented using magnetic [8] and electrostatic [9] forces, but these structures are constrained to reside in well-defined sites above a substrate. Radiometric levitation of microstructures has been also proposed and theoretically analyzed [10]. Recently, untethered flight of surface micromachined structures powered by radiometric forces [11, 12] have been demonstrated. These MEMS microfilers are $300\mu m \times 300\mu m \times$ $1.5\mu m$ in size and are actuated by a temperature gradient from microfabricated heaters situated underneath them. Movies of representative flight experiments for radiometric microfiliers can be found here [13].

However, in order to fully investigate the physics of microscale flight, design parameters must be explored that make the use of traditional surface micromachining difficult. The microfliers discussed in this paper have been fabricated using 2-photon polymerization, allowing potentially unrestricted 3D geometry. The devices are lighter than their surface-micromachined counterparts, and have achieved untethered flight to a distance of several mm, reached heights of up to a 1 mm above the substrate, and velocities of up to 10 mm/s. An in-depth analysis of the radiometric forces, combined with our experiments, provides evidence that the overall perimeter of the flier, rather than its surface area, is the key factor affecting its thrust. This result seems to further be corroborated by the flight surfaces of microscale insects found in nature.

Future aerial microrobots, if successfully developed, have

many applications in areas such as microscale payload delivery, radar deflection, and medicinal and sensing.

RADIOMETRIC MICROFLIER ACTUATION

Radiometrically powered microscale flight is achieved by the use of thermal transpiration effect, which is a force generated by a thermal gradient in the air surrounding the microflier. The thermal transpiration effect is caused by momentum transferred from the impact of faster moving gas particles on the underside of the microflier as shown in Fig 1. If the thermal gradient is oriented in the vertical direction, the thermal force F may counteract the force due to gravity, propelling the microflier upwards. Higher momentum of gas particles below the microflier is achieved by a heating the underside gas particle using a MEMS micro-heater.



Fig. 1. The mechanism of actuation of microfliers using thermal transpiration effect.

Previous analytical treatments of the radiometric force have been performed by Einstein [14], Maxwell [15], and others. These treatments have been expanded on recently by Scandurra [16], where relationship for the net force on a thin plate in a thermal gradient was derived. In Scandurra's paper the force is divided into two components: the shear and normal components. These components are combined into a single formula (1): the force generated by the thermal transpiration effect:

$$F_r = -l \cdot \Delta T \, \frac{(2\alpha\lambda - \alpha\tau - 4\lambda)}{\sigma^2 \tau} \frac{15k}{\pi 64\sqrt{2}} \, . \tag{1}$$

In Eq. (1), α is the thermal accommodation coefficient of the surrounding air to the material, k is the Boltzmann constant, τ is the thickness of the plate, ΔT is the difference in temperature between the top and bottom of the plate, λ is the mean free path of the surrounding gas, σ is the radius of the surrounding gas, and l is the length of the perimeter around the edge of the plate. A key observation that can be made is that in (1), the force produced is directly proportional to the perimeter of the plate.

The following corollary is proposed based on Scandurra's radiometric force analysis for thin plates based in equation (1):

Corollary 1: Given two microfliers with the same horizontal crosssectional area but different perimeters, assuming all other factors remain the same, the microflier with the larger perimeter experiences greater radiometric force, i.e. $F_r \propto l$.

Measuring forces at the microscale is challenging. Instead of directly measuring the force, the vertical and horizontal distance travelled by the microflier can be analyzed. This is assuming that a

Solid-State Sensors, Actuators and Microsystems Workshop Hilton Head Island, South Carolina, June 5-9, 2016 higher actuation force leads to a greater vertical and horizontal travel distance. Using this, Col. 1 is extended for the horizontal distance travelled by the microflier:

$$D_h \propto F_r \propto l, \tag{2}$$

where D_h is the horizontal distance covered by the microflier. We designed microfliers and experiments to test Col 1.

MICROFLIER DESIGN

To show that the perimeter is an important factor in the radiometric force, microfliers were designed with similar horizontal cross-sections and varying perimeters. Two microfliers (Flier A and B) were designed with the perimeter of the one twice that of the other, respectively. Fig. 2 shows the CAD models of the microfliers with 4 wings (Flier A) and 16 wings (Flier B) which has twice the perimeter of 4-wing (Flier A). Other parameters for the two designs are given in Tab 1.



Fig. 2. CAD models of (a) 4 winged and (b) 16-winged 3D printed microfliers.

Tab. 1. Microfliers' features with horizontal surface area of 26000 μm^2 and ρ (density) = 1370 kg/m²

Туре	Wings	Perimeter (µm)	Volume (m ³)	Weight (nN)
Flier A	4	1516	7.44e-14	0.9988
Flier B	16	3135	7.10e-14	0.9532

FABRICATION

Previously reported silicon microfliers used stress-engineering to angle the wings such that in-flight stability is ensured. In this work, two photon lithography is used in an additive manufacturing process to fabricate our microfliers.

Multiphoton lithography is a technique for creating small features in a photosensitive material without the use of complex optical systems or photomasks [17]. Multi-photon lithography has been successfully used in the Nanoscribe Photonic Professional GT tool for 3D microprinting and maskless lithography. Fig. 3 shows the scanning electron micrograph (SEM) of the designs for Flier A and B using stereolithography.



Fig 3. SEM Pictures of 4 wing (Flier A) and 16 winged (Flier B) stereolithograpically printed microfliers.

Two photon absorption [18] is the simultaneous absorption of

two or more photons in order to excite a molecule from a ground state to a higher excitation state. Two photon absorption has a general advantage over single-photon absorption in the fact that its resolution has a quadratic dependence on the intensity. The intensity level is just enough to polymerize the photoresist which acts as the basic material for microstructures created by this process. For creating MEMS microfliers, a stereolithography technique enables creation of a large variety of microflier geometries, enabling the evaluation of the effects of flier geometry on flight performance. This in turn enabled the investigation of the hypothesis from Col 1.

EXPERIMENTAL RESULTS

Experiments were conducted to test our hypothesis presented in Col 1. For this experiment, the 3-D printed microfliers were transferred to MEMS heaters. The heaters were fabricated from polycrystalline silicon using a surface micromaching foundry process [18] on a die with 10 nm of chrome metal deposited to increase the conductivity.



Fig. 4. Isothermal profile around the MEMS heater. Microflier flight path normal to the isothermal contour is indicated by the arrow and superimposed CAD model.

Fig 4 presents the isothermal contours around the heater as generated by FEA model. In all the experiments, the microflier is observed to fly normal to the isothermal contours.

For each experiment, a single microflier was placed at the center of a heater. A high speed optical camera (Basler Ace acA2040-180kc) connected to a microscope recorded the flight at a frame rate of 620 fps. Fig. 5 show flight sequences of a 4 wing and 16 wing microflier captured using the high speed optical camera during the activation of the heater. The location of the microflier is highlighted in red. Fig 6 shows a 16-wing microflier take-off imaged using FLIR infra-red imaging, indicating that the heater reaches a temperature of above 700 °C as the flier is propelled upwards.



Fig. 5. Image sequences of a representative untethered flight of a 4wing (top) and 16 wing (bottom) microfliers captured by high-speed optical camera at 620 fps.



Fig. 6. Take-off of 3D printed microflier captured using FLIR thermal camera at 480 fps.

Flight profiles

The vertical and horizontal distance covered by the flier during flight was extracted by comparing the high-speed camera frames to a set of images taken of the microflier at known reference heights. The error to estimate the height of these flights increases with increasing height which is shown with respect to the error bar at the each point. A total of five flight profiles for each of the flier types (Flier A and B) were extracted.

Five representative flight trajectories for 4-winged (Flier A) and 16-winged (Flier B) microfliers are shown in Figs 7 and 8, respectively. The flight profiles indicate a tendency for increase in the horizontal and vertical distance traveled by the 16-winged microflier compared with the 4-winged microflier, as suggested by Col 1.



Fig 7. Flight profiles for five representative experiments with 4winged microfliers.



Fig. 8. Flight profiles for five representative experiments with 16winged microfliers.

According to our hypothesis, it was presumed that a larger force is experienced with a greater perimeter and hence a larger vertical or horizontal flight distance. The extracted experimental data was used to generalize our hypothesis to a larger population of microfliers.

A sampled dataset of fifteen experimental data points for each design is used to test the hypothesis. The statistical data for both microflier experiments are summarized in Tab 2.

Tab 2. Statistical analysis of the microfliers' horizontal flight distance as observed from 15 experiments for two types of microfliers.

Statistics	4-Winged Microflier	16-winged Microflier	
Mean	315.15	426.45	
Standard Dev	65.54	94.60	
Confidence interval 95%	36.29	52.39	

Statistical t-test for fifteen samples was performed with a 95% confidence interval. Fig 9, shows the resulting 95% confidence intervals of the data for the 4 winged and 16-winged microfliers. A paired t-test was used to reject the null hypothesis, up to the confidence of 98.25%, suggesting that Flier A and B indeed fall into different distributions with respect to distance flown. It can be therefore inferred that the perimeter plays an important role in thermal flight performance of stereo-lithographically printed MEMS microfliers, confirming Col 1.



Fig. 9. 95% confidence interval of horizontal distance travelled by two sets of 15 microfliers indicates statistically significant differences between their flight performances.

Biomimetic Significance

Although diametrically different in the physical underpinnings of the forces experienced by our radiometrically powered microfliers to the forces experienced by microscopic flying insects, a convergence to the same underlying geometry can be observed. Fig. 10 shows the body and wing designs of a recently discovered parasitic wasp [19]. The unusual wing structure consisting of fringes (*setae*) favoring large perimeter, can be observed. A simple analysis of laminar flow across the wings' surface at varying scales (ranging from micrometer to cm sized) suggest that the flow in between the gaps of these wings is very small relative to the flow outside of the relative scale is increased, more flow is observed in-between the fringes.

Although it is unclear at this point the exact convergence between the microflier geometries and the wing geometries of the microflying insects, despite the differences in the underlying physical mechanisms, perhaps the similarities in the shear molecular flow across both sides of the wings is an explanation as to this biomimetic coincidence, warranting further investigation.



Fig. 10. (top) (a) Tinkerbella Nana parasitic wasp (b) 2D FEM model of the fringes, approximately based on cross section as shown (bottom) Simplified 2D Finite Element Model laminar flow of parasitic wasp wing fringes at different scales. Arrow indicates the air-flow.

CONCLUSIONS

This paper presents an analysis of the underlying forces that govern radiometrically-powered MEMS microfliers, develops designs based on this understanding using 3-D stereo-lithography, and presents experimental flight profiles extracted using high-speed microscopy. In particular, two microflier designs were tested experimentally and analysis of the results corroborate the theory that the perimeter of the microflier as opposed to the horizontal cross section area is the key factor determining the radiometric forces experienced by the microfliers.

The results indicate similar preference to what is found in nature with respect to flight surfaces of microscale insects. This is surprising, as microscale insects use a different underlying mechanism to generate lift, however it is possible that microfluidic behavior involving gaseous flow at the microscale leads to the emergence of this common feature, warranting further investigation of this phenomenon.

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DEVELOPMENT OF A HYDRAULICALLY SMOOTH WALL SHEAR STRESS SENSOR UTILIZING THROUGH SILICON VIAS

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ABSTRACT

This paper describes the development of a wall shear stress sensor system designed to be fully non-invasive to low-speed aerodynamic flows. The microelectromechanical systems (MEMS) sensor is fabricated with integrated through-silicon-vias (TSVs), eliminating the need for front side wire bonds. Packaged in a modular housing with interchangeable components, the result is a device with less than 50 μ m of deviation over the wetted surface, satisfying the requirements of hydraulic smoothness for flow speeds less than 30 m/s. Sensor performance includes a sensitivity of 1.36 mV/Pa at 1.128 kHz, corresponding to a minimum detectable signal of 1.1 mPa, resonance of 3.4 kHz with a 1.5 kHz flatband, and 72 dB of pressure rejection.

INTRODUCTION

Despite years of effort, the ability to make continuous realtime direct measurements of wall shear stress with both mean and fluctuating components remains elusive [1],[2]. Because of this, investigations into fundamental fluid flow problems are often hindered. An instrumentation-grade tool to precisely measure wall shear stress would enable further research in the areas of skin friction drag and turbulent boundary layer analysis [3].

For accurate measurement of fluid-wall interactions in a turbulent flow field, it is essential that the installed sensor be non-invasive, or hydraulically smooth, so as to not corrupt the flow measurement [4]. Hydraulic smoothness requires a surface roughness less than 5 viscous wall units, which for typical low-speed wind tunnel applications (e.g, Reynolds number ~ 2,500) is ~50-100 μ m. Wall shear stress sensors with integrated TSVs have been previously reported only for polymer extrusion applications [5].

Given this limitation, and considering the small magnitude forces of aerodynamically generated wall shear stress, a MEMSbased differential capacitive floating element design retains the most promise. The work presented here is a continuation of prior efforts from Chandrasekharan et. al[6], and Meloy et al [7]. Previous sensors made use of frontside wire bonds for electrical connections to the silicon electrodes, which require encapsulation for flow protection and extend above the surface well over 500 μ m. Additionally, improvements to the interface circuitry design reduce the electronic noise floor to increase the dynamic range.

STRUCTURE AND DESIGN

The overall sensor structure is composed of three parts: the floating element, interdigitated comb fingers, and supporting tethers. An analytical mechanical model of the dynamic and static sensor response can be found in [6]. An image of a fabricated device under a microscope is seen in Figure 1.

The floating element acts as the primary flow interface, providing a surface for wall shear stress to act upon. Pressure release holes throughout the surface assist in equilibrating normal pressure across the floating element. Their diameter, 5 μ m, is small enough to remain hydraulically smooth and not significantly alter the effective wetted surface.



Figure 1: Microscope image of wall shear stress sensor.

Interdigitated comb fingers on either side of the element act as variable gap capacitors. Set in a differential orientation, when the floating element deflects in a streamwise direction the gap between one set of fingers will shrink and the opposing gap widens. The benefits of a differential setup include rejection of common mode inputs, including pressure, and doubling of the overall sensitivity. Furthermore it allows for proper retention of input phase and thus the single-axis direction of wall shear stress at the sensor location.

The supporting tethers are arranged in an H-bar structure format as seen in Figure 1. Modeled as a clamped-clamped beam, the tethers act as a restoring spring in the primary flow direction, and work to reject normal pressure and cross-axis shear inputs. Optimization of the sensor's operational space focuses primarily on tether width. A narrower tether reduces structural stiffness, increasing sensitivity, while a wider tether increases the resonance and bandwidth.

FABRICATION

The sensor is fabricated using a six-mask process, starting with a silicon-on-insulator (SOI) wafer with an n-doped 50 μ m device layer on top of a 1 μ m buried oxide (BOX) and 400 μ m handle. Integrated poly-silicon (Si) TSVs, doped with red phosphorus to a resistivity of <0.005 ohms-cm, are isolated from the handle layer by 1 μ m of thermal oxide and directly connected to the device layer to improve conductivity. Wafers were sourced from IceMOS Technologies with TSV fabrication already completed.

The remaining fabrication steps are carried out at the University of Florida Nanoscale Research Facility (NRF). A buffered oxide etch (BOE) is first used to clear out backside contact windows over the TSVs, while the frontside oxide is completely stripped. A 0.5 μ m Al/Si (2%) layer is sputtered on both sides of the wafer. Bond pads are patterned and etched as 250 μ m squares over the TSV contacts. A protective layer of silicon nitride, 800 nm thick, is then deposited on the backside using plasma-enhanced chemical vapor deposition (PECVD).

978-1-940470-02-3/HH2016/\$25©2016TRF DOI 10.31438/trf.hh2016.63 To ensure ohmic contact between the TSVs and bond pads, a series of current-voltage (I-V) curve measurements are made with a HP4154 Semiconductor Parameter Analyzer. Test structures include sets of resistive elements in series utilizing 2, 4, 8, 16, 32, or 64 TSVs. Total resistance measurements are normalized by the number of TSVs used. After initial measurements are performed the wafers are annealed in forming gas (95% nitrogen and 5% hydrogen) at 450C for 60 minutes and the resistance measurements are ohmic.

Sequential deep reactive-ion etch (DRIE) masks are then used for frontside floating element definition and backside cavity etching. Device separation is achieved during these cycles as well, with 'dicing' streets included in the exposed areas. A BOE etch releases the floating elements and separates the individual die, which are held to a carrier wafer with Nitto-Denko thermal release tape. Final clearout of the BOX near the base of tethers is completed with a reactive-ion etch (RIE). A schematic of the final device, and its first level of packaging, is shown in Figure 2.



Figure 2: Simplified schematic of six-mask fabrication performed at UF Nanoscale Research Facility.

Die selection is carried out as a two-step process. Physical quality of the sensor is assessed with electrostatic actuation. With the die placed under a microscope, a 2 Hz square wave is placed across each capacitive gap. If structurally sound movement is observed, and there are no cracks or defects in the floating element, the sensor is set aside for electrical impedance testing. A measured phase delay greater than 80 degrees is interpreted as nominally a capacitive impedance, without spiking or shorting through the TSV isolation oxides.

INTERFACE CIRCUITRY

The interface circuitry includes a voltage follower amplifier circuit collocated inside the cylindrical sensor housing, and an external synchronous modulation and demodulation circuit (SMOD/DMOD) [8]. Together, the system allows for real-time data acquisition at baseband frequencies far away from the experimental setup.

Situating a voltage follower directly behind the sensor die reduces the effect of signal degradation from transmission over long distances. The experimentally verified impedance of the sensor electrodes is O(10 pF). An Analog Devices AD8022 operational amplifier is selected for its well-matched input capacitance and overall low noise floor. Theoretical predictions indicate minimal signal loss with cable lengths up to 50 m.

As a capacitive sensor with the intention of measuring both mean and dynamic inputs, a standard DC biasing scheme is unsuitable. To avoid the undefined impedance of a capacitor at DC, a sinusoidal biasing scheme is utilized. The SMOD/DMOD circuit is composed of four main blocks: oscillator and bias generation, timing control, filtering and conditioning, and active rectification.

A crystal oscillator generates a low-amplitude sinusoidal signal at 1 MHz. A series of adjustable gain amplifiers allow for production of two 180° phase-shifted 1 MHz carrier signals, typically set to 8 V amplitude, which are supplied to the sensor's fixed electrodes. Simultaneously, a phase-locked 5 V square wave is generated as a timing control signal for rectification. Both the duty cycle and relative phase offset of this signal are adjustable, and tuned for maximum signal to noise (SNR) performance during calibrations.

As the modulated signal is returned from the sensor amplifier, a bandpass filter removes residual DC components, low noise content, and carrier signal harmonics. A secondary gain stage further increases SNR before demodulation, accomplished with an active envelope detector. The gained signal is split, with one output buffered and the other inverted. A switch, controlled by the timing control signal, incorporates the two split signals. The result is a rectified carrier signal wave, with the envelope defined by the baseband signal content. Mean wall shear stress inputs, resulting in static changes in the nominal capacitive gaps, change the amplitude of the passed carrier wave. Low pass filtering of the rectified signal completes the demodulation, removing the carrier signal component entirely. The final system output is baseband wall shear stress information in real time, containing both mean and dynamic components.

PACKAGING

Packaging is achieved with a three-part modular approach, consisting of the sensor head, amplifier housing, and cabling connector, with a representative schematic seen in Figure 3. External packaging dimensions are 0.5" in diameter and 2" in length for the total aluminum cylinder tube.

The sensor module itself is a four-part assembly, consisting of a sensor die, a supporting substrate for electrical interconnect, a shim cap for flushness, and a metal housing. Figure 4 shows a schematic of the sensor cap attachment. A micro-milling machine is used to recess a rectangular pocket in a circular FR-4 disk. Epoxy film, chosen with a coefficient of thermal expansion minimizing mismatch between the silicon and FR-4 interface, is placed in the recess. A flip-chip bonder places the sensor die into the milled pocket of the FR-4 support substrate, with the heated chuck curing the epoxy film. Electrical connections are made from the backside of the sensor die to the FR-4 bond pads. Shim caps are machined from plastic stock with a picosecond laser system, and affixed around the die. The entire disk is placed into a milled recess of the aluminum end cap, and pressed down for flushness.



Figure 3: Schematic of three part packaging, with sensor cap recessed into removable module housing.



Figure 4: Schematic of sensor cap assembly. The fabricated sensor die is affixed and electrically connected to the PCB, with a surrounding shim cap for flushness.

The buffer amp module is a hollow tube of aluminum which houses the amp board. Included with the AD8022 are power line filtering capacitors and a bias resistor to provide a path for dissipation of DC charge. Wiring connections to the sensor and cabling modules affix to either end of the amp board. Cabling is provided with LEMO style end connectors, with interchangeable total cable lengths, as seen in Figure 5.



Figure 5: Packaged and assembled sensor, ready for installation.

Scanning white light interferometry (SWLI) measurements of this packaged cap, shown in Figure 6, indicate a hydraulically smooth front surface for low-speed flows. Maximum step deviations are roughly 50 μ m, occurring at the junction between the metal housing and shim cap. Overall roughness is below 5 μ m. The majority of this roughness is found on the face of the metal

housing. Higher tolerances during machining, or a secondary polishing stage before assembly, will likely lead to a more planar surface.



Figure 6: SWLI measurement of packaged sensor module, with X-Profile map included. The sensor die surface is used as a zerooffset reference level.

SENSOR CALIBRATION

To characterize the dynamic wall shear stress response of the system, the shear stress field within an acoustic plane wave tube is utilized [9]. This method takes advantage of Stokes' layer excitation to generate a known oscillating wall shear stress based on measurements of maximum end wall pressure.

The wall shear stress sensor is located at a quarter wavelength away from the sound hard termination for the frequency of interest. This situates the sensor at a pressure minima node, corresponding to a maxima in velocity and shear. The result is acoustic excitation of the sensor with tangential shear with reduced normal pressure loading, mitigating cross-axis inputs during testing.



Figure 7: Sensitivity calibration results at 1.128 kHz. Linear fit to points above the noise floor in red correspond to a sensitivity of 1.36 mV/Pa.

By increasing the sound pressure level driven by the speaker, the dynamic sensitivity of the sensor is determined. A calibration frequency of 1.128 kHz is used. At a carrier signal bias level of 8 V amplitude, the sensitivity is measured as 1.36 mV/Pa, shown in Figure 7. The response is linear up to the bounds of the testing platform of 2.18 Pa for 66 dB of experimentally verified dynamic range. Theoretical predictions indicate the onset of non-linear response at 100 Pa, corresponding to a dynamic range of 100 dB.

A noise floor measurement is displayed in Figure 8. Data is sampled with a SRS780 (Stanford Research Systems) dynamic signal analyzer utilizing 800 FFT lines over various frequency spans. To eliminate EMI effects, the wall shear stress sensor and interface circuitry are placed within a double Faraday cage. With a measured noise floor of 1.5 μ V at 1.128 kHz, the corresponding minimum detectable signal is 1.1 mPa.



Figure 8: Noise floor of fully assembled wall shear stress sensor system. At the calibration frequency of 1.128 kHz, the noise floor is $1.5 \mu V$, for a MDS of 1.1 mPa.

A similar methodology is used to characterize the frequency response function of the device. The static end wall termination is replaced with a variable termination that moves axially along the duct. By keeping the wall shear stress sensor at a fixed location along the tube length, and adjusting the axial distance of the end wall, the shear sensor is kept at a pressure node for all frequencies. A frequency response function is constructed using the singlefrequency sensitivity as a reference. The resonant peak of the sensor is identified at 3.4 kHz, with a useable flatband extending to 1.5 kHz.

Sensitivity to normal pressure is characterized by mounting the shear sensor in the end wall of the plane wave tube. In this orientation the floating element is subjected to normally incident sound waves with no tangential shear, and repeating the procedure yields system sensitivity to pressure. A pressure rejection ratio of 72 dB is observed.

CONCLUSIONS

The sensor described herein is the first hydraulically smooth wall shear stress sensor for use in low-speed wind tunnel applications. SWLI measurements verify a maximum step height of less than 50 μ m across the 0.5" diameter package. Specialized interface circuitry leverages an amplitude modulation approach to deliver continuous real-time measurements of wall shear stress with directional mean and dynamic information included. Overall performance specifications are consistent with other MEMS

sensors described in the literature, including a sensitivity of 1.36 mV/Pa, MDS of 1.1 mPa, and bandwidth of 1.5 kHz.

Future work will focus on experimental verification of performance inside full scale wind tunnels, and a transition to commercially available instrumentation. Extension of measurement capabilities to both tangential in-plane directions would provide a true vector measurement of wall shear stress.

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ELECTROMECHANICALLY ACTIVE FLAT OPTICAL DEVICES

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ABSTRACT

We demonstrate an electromechanically controllable reflective flat lens at mid-infrared wavelength. The lens is designed from plasmonic nano-discs, and focuses light at mid-infrared, 45° to the incoming beam. The flat lens is fabricated on a 2.8 μ m thin membrane and integrated with a micro-electro-mechanical (MEMS) device. Mechanical testing of the MEMS device before and after integration of the flat lens confirms that the performance of the MEMS has not been compromised due to addition of a flat lens.

INTRODUCTION

Recently, flat optical devices capable of shaping the wavefront of light have come to the forefront of ongoing scientific research [1]. Planar counterparts of conventional optical devices like lenses [2, 3], beam deflectors [4, 5], holograms [6, 7], and so on have been experimentally demonstrated. These devices, referred to as "metasurfaces", use sub-wavelength dimension metal or dielectric resonators, arbitrarily spaced on a two dimensional plane, mimicking the phase profile of any conventional bulk optical device and beyond. Such metasurface-based flat devices are compact and lightweight compared to the three-dimensional counterparts. However, most of these nanostructured devices have so far been passive; a single device provides a specific output only. For developing these flat optical elements into useful optical systems, we need to incorporate precise mechanical control of the individual elements.

In this paper we introduce a useful concept of actively controlling these flat optical devices by electrostatic actuation. We present a prototype consisting of an electromechanically controlled plasmonic flat reflective lens that focuses in the mid-infrared part of the spectrum. When electrostatically actuated, the MEMS platform controls the tilt angle of the lens along two orthogonal axes by about +-9 degrees. In dynamic actuation the platform can be tilted to larger angles to scan the focal spot. Such actively controlled miniaturized optical devices promise to provide faster, more efficient and often enhanced functionalities.

DESING OF THE LENS

We design a plasmonic lens producing cylindrical focus when illuminated with light at wavelength of 4.58 µm. As the design units, we choose sub-wavelength sized gold resonators in the shape of a disc (Fig. 1a). The reflection from the gold nano-resonators is reinforced by the use of a continuous gold film, separated from the resonator by a 400 nm thick silicon dioxide layer. When signal is incident from the top, it interacts with the nano-disc, passes through the transparent dielectric layer and gets reflected back up from the gold backplane. The reflected signal comes out with a different amplitude and phase compared to the incident signal. By simply changing the radius of the disc, it is possible to realize different values of reflected phase between 0 and 2π (Fig. 1b). To construct a planar lens, we spatially distribute the discs with varying radii to realize the hyperbolic phase profile on a twodimensional surface. Figure 1c shows a schematic of the reflective flat lens; light is incident normal to the lens surface, but reflects at an angle from the incoming beam, while focusing the reflected light at a predetermined distance from the lens surface. To achieve this change in path of the focused beam, we superimpose a planar phase shift on the hyperbolic phase profile. This configuration helps to dissociate the reflected light from the incident light, besides proving the flexibility of our design technique.



Figure 1: Designing the plasmonic flat lens (a) A unit cell consisting of a 50 nm thick gold disc on 400 nm thick silicon dioxide substrate with 200 nm thick gold backplane (b) Simulated values of reflective phase for varying sizes of gold discs. (c) Schematic representation of focusing characteristics of the reflective flat lens.

Figure 2 summarizes the simulated results for a realistic lens design at the wavelength of 4.58 μ m. The cylindrical lens has a square layout, with each side measuring 800 μ m. Figure 2a shows the propagation of the reflected beam along the transverse direction; the incoming beam is not shown. The lens is positioned at y = 0, and about x =0. The axis of reflection makes an angle of 45° to the normal of the lens, i.e. to the y-axis. The focal plane is 2 mm away from the lens surface, when measured along the axis of reflection (Fig. 2a, dotted straight line). The intensity distribution at the focal plane is shown in Fig. 2b. A focal line appears because of the cylindrical focusing characteristics of the flat lens. The spatial distribution of the phase at the plane of the lens is shown in Fig. 2c; the phase changes along x-position of the lens only.

FABRICATION

The flat lens

The flat lens is fabricated using standard photolithography techniques. As the substrate, a silicon-on-insulator (SOI) wafer is chosen with 200 nm buried-oxide layer and 600 μ m thick handle layer. Figure 3 (a – f) shows the process flow for fabricating the lens. In the following section we describe the fabrication steps following the figure numbers: (a) On the topside of the SOI wafer,

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Figure 2. Simulated characteristics of a flat cylindrical lens at $\lambda = 4.58 \ \mu m$. (a) Intensity distribution of the reflected beam at the plane of propagation; position of the lens is shown by the white solid line. (b) The intensity distribution at the focal plane (c) The phase distribution at the surface of the lens.



Figure 3. Fabrication of the planar lens. (a) - (f) Process flow of photolithography as descried in the section **Fabrication: The flat lens**. (g) Optical microscope image of the flat lens (h) Scanning electron microscope images of a part of the fabricated lens.

a 100 nm thick protective layer of silicon nitride (Si_3N_4) is deposited by plasma-enhanced chemical vapor deposition process. On the backside, a 110 nm thick layer of silicon dioxide (SiO_2) is deposited using the same process; this layer would serve as the oxide mask for etching the handle layer at a later stage of the fabrication. (b) A positive photoresist (SPR 700) is coated on the backside and exposed using a stepper tool (Autostep 200 i-line) to print circular windows, which would be perfectly aligned with the flat lenses to be fabricated on the topside. Using the developed photoresist as an etch-mask, the silicon dioxide layer is plasmaetched to produce the windows. (c) Next, the topside protective layer of Si₃N₄ is cleared in hot phosphoric acid (85% H₃PO₄ at 165 °C for 5 minutes). (d) Now we start fabricating the flat lens on the topside. We deposit 200 nm thick layer of gold (using electron beam vapor deposition), followed by 400 nm thick SiO₂ (using PECVD). (e) We choose a bilayer resist for facilitating clean liftoff. The topside is coated with the photoresists LOR 3A followed by SPR 700. The resist bilayer is exposed using stepper, ensuring each lens structure is accurately aligned with the previously etched backside windows. (f) Finally, 50 nm gold with an adhesion layer of 5 nm thick titanium is deposited using e-beam vapor deposition, and lifted-off in remover-PG. This results in the gold disks constituting each lens. Figure 3g shows the optical microscope image of one of such lenses. Figure 3h shows the scanning electron microscope images of a section of the fabricated lens.

Integrating flat lens with MEMS

To integrate the flat lens with an external MEMS device, we try to "tear out" the lens from the thick SOI wafer. To do this, we start by etching the 600 μ m thick handle layer in xenon difluoride (XeF₂). Xenon difluoride etch is a highly selective, isotropic, dryetch process for silicon [8]. The backside window made with SiO₂ provides the entry point for the xenon difluoride gas to react with the bulk of silicon in the handle layer. Due to excellent selectivity of XeF₂ to silicon versus SiO₂, the 200 nm thick buried-oxide layer of the SOI wafer also serves as the etch-stop layer. The etching process is visually inspected in-situ until all the silicon directly beneath the lens is etched out, and the lateral extent exceeds the outer dimension of the lens.



Figure 4. Optical microscope image after etching of the SOI handle layer from the backside and from the topside.

Figure 4 shows optical image of the etched portion from the backside as well as from the topside. From the top, the shadow beneath the square lens structure indicates the area where silicon has been etched out. Figure 5a shows the schematic and the scanning electron microscope image of the different layers building up the lens. After the XeF₂ etch, we are left with a membrane that is only 2.8 μ m in thickness and almost 1.35 mm in diameter (see Fig. 4).



Figure 5. Integrating planar lens with MEMS. (a) Schematic of different constituting layers after etching of the SOI handle layer (left), and (right) SEM of the layers constituting the membrane on which the gold nano-discs are placed. (b) Stages of integration of the flat lens with an external MEMS platform.

For the next stage of fabrication, we use the focused ion beam (FIB) tool integrated with a micromanipulator needle to assemble the lens with the MEMS device. Figure 5b depicts the steps of the process. We trace the focused ions around the periphery of the lens to cut out most of the structure, except a small portion. Here we weld the needle-tip of the micromanipulator by depositing platinum. Now the rest of the structure is released; the membrane is free from the surrounding solid substrate and is held only by the micromanipulator needle. We move the membrane out of the substrate and on to the MEMS platform by controlling the micromanipulator arm. After the lens is placed and aligned with the central platform of the MEMS, it is *glued* or welded in small

patches with ion-beam assisted deposition of platinum. Finally the needle is cut away by milling with focused ion beam.

EXPERIMENTAL CHARACTERIZATION Optical measurement

The optical performance of the lens is measured before integration onto the MEMS actuator, while it is still on the solid SOI wafer. To measure the focusing performance of the lens, we use a quantum cascade laser at $4.58 \mu m$.



Figure 6. Schematic showing experimental arrangement used for characterization of the optical response of a flat lens.

Figure 6 shows the schematic of the experimental arrangement. The laser beam is downsized by 0.5 times, such that the incident beam is only slightly larger than the 800 μ m square lens. The light is incident normal to the lens surface. The lens reflects the incident beam at 45° to the incoming path, and focuses the light at a distance of 2 mm away from its surface. The focused light is magnified two times by the lens pair L3 and L4 (see Fig. 6) and the intensity is detected by a thermo-electrically cooled mid-IR detector through a 10 μ m pinhole.



Figure 7. Focusing characteristics of the lens. (a) Experimental and (b) Simulated intensity profile measured across the focal line.

The detector-arm assembly is mounted on a rotating stage centered about the incident point on the wafer. This ensures we can precisely rotate the arm to detect the reflected light at an angle of 45° to the incoming beam. The telescope (lens L3 and L4) and the detector can separately move along the reflection path so that all the components can be placed to accurately capture the intensity at the prescribed focal length of the lens. After the focal plane is detected, the detector is scanned linearly, across the reflection path,

in 2 μ m steps. This gives us the intensity distribution across the focal line as shown in Fig. 7; the experimentally measured profile agrees well with the simulated result. The asymmetric distribution of low-intensity sidebands, in both the simulated and experimental result, is due to the 45° phase ramp imposed on the lens surface to achieve the non-normal reflective focusing.

Mechanical measurement

To characterize the mechanical response of the MEMS device, we mount it on a dual in-line package. Figure 8a shows the optical microscope image of the flat lens integrated with 2D MEMS scanner; the inset shows a packaged device ready for electrostatic testing. As shown on Fig. 8a, the MEMS device has rotational degrees of freedom about two orthogonal axes: the inner gimbal axis and the outer gimbal axis respectively. The torsional movement of the MEMS platform is achieved by electrostatic actuation of capacitive vertical comb drives [9].



Figure 8. Mechanical characterization: (a) Optical microscope image of the flat lens integrated with MEMS; inset shows the device mounted on a dual in-line package ready for electrostatic actuation. (b) Electro-mechanical response of the MEMS platform with and without the flat lens.

Figure 8b shows the mechanical response of the MEMS platform when a voltage is applied either across the inner or the outer axis. As the applied voltage is increased, the platform starts rotating until a saturating region is reached beyond which the comb drives cannot be moved further. We perform this experiment under

an optical profilometer; for each applied voltage we capture an image of the MEMS surface. The tilt in the MEMS platform is calculated with respect to a rigid part of the peripheral structure. The measurements are taken for bare MEMS, i.e., before the flat lens has been integrated, and also for the final device loaded with the flat lens. As shown in Fig. 8b, even after the addition of the flat lens on the MEMS, the electromechanical response of the MEMS has not been compromised.

CONCLUSION

In summary, we have presented an electromechanically active focusing device in the form of a thin and flat surface. The planar optical device is designed for mid-IR wavelength and successfully integrated with a micro-electro-mechanical platform. The MEMS platform provides mechanical control of the flat lens up on electrostatic actuation. The lens can be tilted by ~9 degrees and hence the focal spot. We plan to test the optical characteristics of the lens while it is being actuated by the MEMS. Also, it would be interesting to study the high frequency scanning of the focal spot, while the MEMS is dynamically actuated. This proof-of-concept integration of flat optical devices with MEMS can be extended to visible and other parts of the electromagnetic spectrum implying potential for application across wider fields.

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FLEXIBLE WIDE-BAND TWO-ARM SPIRAL ANTENNA FOR DIRECTIVITY OPTIMIZATION

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ABSTRACT

This paper reports a directivity-reconfigurable antenna with a wide frequency operation range in microwave regime. The device is formed by encasing liquid metal alloys into a highly stretchable elastomer membrane. The antenna is designed to have a two-arm Archimedean spiral structure and is able to optimize its directivity by inflating the elastomer to form a dome-shaped structure with different heights, pneumatically driven by microelectromechanical systems (MEMS) based microblowers. The antenna can operate in a wide frequency band from 6.9 GHz to 13.8 GHz. The proposed tuning mechanism allows optimizing radiation patterns of the antenna by making radiation become more directive in the inflation direction, while the passing band of the device still remains wide.

INTRODUCTION

Flexible electronics have been intensively studied because they are able to be conveniently integrated on curved surfaces for a broad range of applications such as wearable devices, artificial skins, flexible displays, sustainable energy harvesters [1-3]. Recently, inclusion of liquid metal into stretchable elastomers has opened up many great opportunities to develop tunable and flexible electronic and electromagnetic devices in radio frequency range [4-6]. Generally, liquid metals and alloys can flow and reshape even under large mechanical deformation. Gallium-Indium eutectic (EGaIn) is considered as a promising substitution of mercury because of its liquid state in room temperature and low toxicity. Recently, we have recently developed a fabrication process capable of embedding EGaIn into a stretchable silicone-based elastomer (Ecoflex). This process has allowed us to develop stretchable and tunable microwave meta-atoms [6], and meta-skins for tunable frequency selective surface and microwave cloak applications [7], where an array of EGaIn split ring resonators are encased into Ecoflex.

Reconfigurable antennas have attracted increasing attention due to the explosive growth of wireless communications. Several liquid metal based tunable antennas have been reported based on different tuning mechanisms and designs, including a stretchable dipole antenna or unbalanced loop antenna [8], an electrically small antenna [9], and an electrochemical controlled capillary actuated monopole antenna [10]. All of the existing devices have focused on frequency tuning without paying much attention to optimization of directivity. It should be noted that directivity reconfigurability is highly desirable for applications, such as remote sensing and targeted power transfer, where signal and power strength in a specific direction needs to be improved.

Here, we report a flexible and inflatable two-arm spiral antenna for directivity optimization in microwave regime. The antenna is formed by encasing liquid metal into a highly stretchable elastomer. The antenna can be inflated from its original planar shape to a dome-like shape by blowing high-pressure air into an air cavity below the antenna using microelectromechanical systems (MEMS) based microblowers. The antenna is capable of changing its shape and allows reconfiguring the radiation pattern of the antenna along the main lobe direction or the inflation direction. When the antenna is inflated to the dome-like shape, the radiation pattern of the antenna becomes shaper, thus optimizing its directivity, while its reflection coefficient remains almost unchanged in a wide band range. In addition, the antenna provides good circular polarization.

DESIGN AND FABRICATION

Fig. 1(a) shows a schematic for a planar self-complementary, two-arm Archimedean spiral antenna made of liquid metal encased by a silicone elastomer (Ecoflex, Smooth-On, Macungie, PA). The operating frequency range of the antenna can be determined by the inner radius *a* and the outer radius *b*. In order to operate in X-band regime, *a* and *b* of are set to be 3.5 mm and 18.5 mm, respectively. Each spiral arm has 2.5 turns. The width of the arm is w = 1.5 mm and the gap between two arms is g = 1.5 mm.



Figure 1: (a) Design parameters of the proposed two-arm spiral antenna. (b) Photo of the fabricated planar antenna before assembled to a pneumatic control unit. (c) Schematic of the fabrication process for the planar antenna.

Fig. 1(c) schematically shows the fabrication process of the antenna. First, a SU-8 photoresist (Microchem, Westborough, MA) is patterned to the designed spirals structure on a silicon wafer W1 with traditional photolithography. Subsequently, a 700 µm thick silicone layer L1 is formed by casting a prepolymer solution of silicone Ecoflex on the SU-8 mold, and fully thermally cured at 60 °C on a hotplate for 30 min. The fully cured L1 layer is then peeled from the SU-8 mold with the spiral channels inside it. An inlet and an outlet are mechanically punched at the two ends of each channel as the liquid metal injection vias. Due to the poor adhesion between fully cured Ecoflex, another 100 µm thick Ecoflex layer L2 is spin coated and followed by a partial curing process on a silanized bare Si wafer W2 at 45 °C for 40 sec. After that, the fully cured L1 and the partially cured L2 are immediately transferred to a hotplate at 90 °C for 20 mins. This allows permanently bonding these two layers. Thus, the spiral channels are formed inside the elastomer with inlet and outlet. After EGaIn is manually injected into the channels with a metal needle (#20 Gauge) amounted on a syringe (10 ml; Becton-Dickinson, Franklin Lakes, NJ), residues of liquid

978-1-940470-02-3/HH2016/\$25©2016TRF DOI 10.31438/trf.hh2016.65 Solid-State Sensors, Actuators and Microsystems Workshop Hilton Head Island, South Carolina, June 5-9, 2016 metal around the inlets and outlets are cleaned by a cotton swab dipped with a hydrochloric acid solution (50%, v/v). Lastly, the device is immersed into a thin prepolymer solution of Ecoflex and then is fully cured at 80 °C for 30 min to seal the inlets and outlets. The total thickness of the elastomer is approximately 1 mm. The liquid metal is located in nearly half the thickness of the elastomer (Fig. 1(b),(c)).



Figure 2: (a) The air pumping setup via four MEMS voltage actuated micro-blowers (b) Relationship between the inflation height and micro-blower actuation voltage with error bars.

OPERATION AND MEASUREMENT

In order to inflate the liquid metal antenna to a dome shape and well control its deformation, an inexpensive and accurate control unit is built with MEMS microblowers (MZB1001E00; Murata, Kyoto, Japan). Briefly, the microblower utilizes a piezoelectric based drive method that features small dimensions and is suitable as a source for high-pressure air generation with compact and low profile. To provide a high pressure to deform the antenna, four microblowers are connected in parallel with tubing and three-way hose fittings (Fig. 2(a)). Subsequently, the planar antenna is sandwiched between two 3 mm thick acrylic boards (Total Plastic, Baltimore, MD) with the help of 4 nylon screws, providing a gas tight package. The whole antenna structure is inflated by two lateral air vents pre-machined on two sides of the lower PMMA board. A circular through-hole with the diameter of 50 mm is created in the upper cladding board. When a D.C. voltage is applied to the microblowers, the microblowers generate high-speed airflow to pneumatically push the elastomer into a dome-like cap. The relationship between the applied actuation D.C. voltage to the microblowers and the resulting height H is investigated. As shown in Fig. 2(b), the height increases almost linearly with an applied voltage at low voltage regions (0-10 V) and then increases slightly slowly at high voltage regions (11-21 V). The reliability of the antenna was investigated by repeatedly balloon it and deflate it from 0 V to 21V. After 100 times of ballooning, the antenna was shaped nearly the same cap height with standard deviation less than 1.5%. At the maximum allowed voltage of 21 V for the microblowers, the obtained dome height is 21.8 mm. During the reliability test, the liquid metal inside the channels is able to reshape and respond to the applied pressure and adapt to different cap shapes continuously every time, thus ensure the whole reconfigurable antenna remain functional without fatigue or cracking after repeated measurements.



Figure 3: Time-lapse images (a-d) and FEA simulation results (e-h) for the Ecoflex elastomer containing the liquid metal antenna with different inflation heights: 0 (a, e), 7 mm (b, f), 14 mm (c, g), and 21.8 mm (d, h). Scale bars in (a-d) represent 3 mm.

Fig. 3 shows the FEA simulation result of stress distribution for four instances of the antenna under different actuation voltages applied to the microblowers. The Young's modulus and Poisson's ratio of Ecoflex are 29.5 kPa and 0.43, respectively. The pressure data for the simulation are obtained by using a pressure sensor (SPD015AA; Smartec, The Netherlands). As the Ecoflex membrane is inflated, mechanical restoring force of the elastomer induced by the stress will eventually balance with the air pressure and achieve an equilibrium shape at a certain height of the dome. For H = 0, 7mm, 14 mm, and 21 mm under the applied pressure of 0, 2.2 kPa, 5.2 kPa, and 11.8 kPa, the resulting maximum surface stress in the deformed Ecoflex membrane is found to be 0, 184 kN/m², 491 kN/m², and 915 kN/m², respectively.

The electromagnetic measurement is carried out in an anechoic chamber. The antenna is fed by connecting two pins of a 3-mm SMA connector with the two heads of the spiral arms at the center region of the antenna. A programmable vector network analyzer (VNA, Agilent E8364) is used in this experiment. The radiation pattern is measured by putting the antenna towards to a horn antenna within the far field region using an automatic antenna measurement system (Dimond Engineering, CA). Fig. 4 shows the measurement setup where the antenna rotates around *z*-axis, and the dashed line is in *x*-*y* plane. The rotation angle φ is defined as the angle between the horn antenna (dashed line) and the *x*-axis. The turntable is set to rotate from θ° to 180° clockwise with an angular resolution of 2°. 21 measurements are conducted at each position to minimize the effect of possible mechanical vibration.



Figure 4: Photo of a setup for measuring radiation patterns of the liquid metal antenna with horizontal polarization.

RESULTS AND DISCUSSION

We measured reflection coefficient of the antenna as a function of dome heights (Fig. 5). The result shows that the passing band for this antenna spans from 6.9 GHz to 13.16 GHz with S11 lower than -10 dB. When the antenna is inflated, the frequency band remains in the same wide frequency region, ensuring that the antenna is fully functional in this range.



Figure 5: Measured reflected power with different inflation heights of the liquid metal antenna.

Fig. 6 shows the radiation patterns of the antenna with horizontal polarization at 8.5 GHz for three different inflation heights: 0 mm, 10.9 mm, and 21.8 mm. The result shows that the signal strength

exhibits the strongest along the y axis, which plotted at 90° for y direction and at 270° for -y direction. The planar antenna transmits almost the same signal strength for both y and -y directions, due to the geometrical symmetry at x-z plane along y-axis. As the inflation height increases, the dome shaped antenna tends to increase the radiation strength along y direction (90°), while decreasing the radiation strength along -y-direction (270°). Further increasing the inflation height will further enhance the radiation along y-direction. Therefore, the directivity at the main lobe direction (y-direction) can be optimized by inflating the antenna to higher heights.



Figure 6: Measured radiation pattern at 8.5 GHz of horizontal polarization for three inflation heights.

The radiation patterns of the antenna at different frequencies are measured in the frequency range from 7.6 GHz to 9.8GHz with the reflection power lower than -20 dB to ensure the antenna operate with its best performance. Fig. 7 shows the normalized radiation patterns of the antenna for three different inflation heights (0, 10.9 mm, and 21.8 mm) at two sample frequencies (8.5 GHz and 9.5 GHz). By rotating the receiving horn antenna by 90°, the radiation patterns of vertical polarization are also measured. Based on the radiation patterns shown in Fig. 7, the axial ratio (AR) of the antenna at different dome heights and operation frequencies is obtained and given in Table 1.

Table 1: axial ratio for different inflation heights and frequencies.

	0 (mm)	10.9 (mm)	21.8 (mm)
8.5 GHz	1.25	1.54	1.32
9.5 GHz	1.58	1.10	1.08

As discussed above, the radiation strength or directivity of the antenna can be improved only in the inflation direction of the dome. Therefore, the radiation patterns of the antenna are measured only from 0° to 180° along the main lobe direction or the inflation direction for meaningful use purpose. The measured radiation patterns show good directivity for all the measured heights at the two sample frequencies along the inflation direction. More importantly, as the inflation height increases, the antenna exhibits stronger directivity along its main lobe direction, thus optimizing its directivity along the inflation direction. Specifically, at 8.5 GHz, by inflating the antenna from 0 mm to 10.9 mm and further to 21.8 mm, the signal strength is improved by 2 dB and further to 3 dB in the horizontal polarization direction. Similarly, at 9.5 GHz, the signal

strength in the inflation direction is also enhanced by 3 dB and 6 dB, for the horizontal and vertical polarization, respectively. It should be pointed out that as the radiation pattern shows a good similarity between the vertical and horizontal polarizations, the antenna provides good circular polarization of an electromagnetic wave.



Figure 7: Measured radiation patterns of both horizontal and vertical polarizations from 0° to 180° for different inflation heights at two sample frequencies: 8.5 GHz (a, b), and 9.5 GHz (c, d).

Furthermore, benefitting from the deformability of the liquid metal and elastomer, the present antenna is able to preserve its electromagnetic properties after being inflated for 500 times to the maximum height of 21.8 mm.

To obtain the benefit of structural tuning, the device needs to operate at temperatures that keep the infused liquid metal in a liquid state. For EGaIn used in this study has a melting point of ~15.5°C, making it liquid at room temperature. It should be pointed out that in case that lower operation temperatures are required, the device requires using other liquid metals or alloys with lower melting points. Also, theoretically, the directivity of the device can be further enhanced by adding a back cavity with a gap of a quarter of wavelength ($\lambda/4$) between a back reflector and the planar antenna. However, it is difficult to maintain the $\lambda/4$ gap during the mechanical deformation of the antenna. We will optimize the device structure and introduce a cavity to the current design in the future. Nevertheless, the demonstrated reconfigurability of the present device allows for continuous tuning the radiation pattern and directivity of the antenna.

CONCLUSIONS

A directivity reconfigurable liquid metal based antenna with wide operating frequency band was developed and demonstrated. The liquid metal based antenna was fabricated by encasing the liquid metal into a highly stretchable elastomer. Benefiting from this fabrication process and structure, the antenna can withstand large deformation with great reliability and repeatability. By pneumatically inflating the antenna with MEMS micro-blower, the antenna can form spherical shapes with different heights. By increasing the inflation heights, the signal strength of present antenna can be improved to different extents from height of 0 mm to 21mm. With an improved directivity in a broad operating frequency range, the antenna can radiate more energy towards the receiver. The potential application of this antenna can be found in systems that require targeting, directivity improving and a wide operating frequency range.

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HIGH-SPEED, LARGE SCAN AREA, DISTORTION-FREE OPERATION OF SINGLE-**CHIP ATOMIC FORCE MICROSCOPES**

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ABSTRACT

We report a 50-fold increase in the scan speed and a 200% improvement in the distortion-free scan range of a single-chip atomic force microscope (sc-AFM). The thermal time constants of 3-DOF actuators have been reduced by a factor of ~10 to increase the bandwidth of quasi-static AFM modes, while also increasing efficiency in dynamic modes. Importantly, the lifetime of the instrument is shown to exceed that of a conventional AFM cantilever by a factor of >100x. These devices have produced the first sc-AFM images of a 22 nm SRAM bank on an Intel Ivy Bridge CPU.

INTRODUCTION

Despite the exquisite resolution that may be obtained with AFMs, the industrial nanometrology enterprise has been reluctant to include them in their suite of inspection tools. The single-chip AFM (sc-AFM) [1] was introduced to overcome several shortcomings of conventional AFMs by replacing bulky piezoelectric scanners with 3 degree-of-freedom electrothermal (ET) MEMS actuators and by replacing laser detection with thermal-piezoresistive resonant sensing. These single-chip instruments are well-suited to high-speed operation and may also be implemented as arrays.

Perhaps the most significant drawback of conventional AFMs, when compared to other scanning microscopes such as confocal or scanning electron microscopes, is that it takes 1-100 minutes to obtain a high-quality AFM image. The first paper on high-speed AFM was published 25 years ago [2]. Several highly customized scanners were then developed to demonstrate video-rate AFM [3,4]. These instruments place severe constraints on the sample volume because the reported scanners cannot accommodate large masses (i.e. wafers). The present work obviates any sample movement because the tip is scanned in 3DOF by MEMS actuators, thus markedly raising the lowest natural frequency of the instrument.

Tip wear is an important concern in AFM productivity. Along with the increased cost of consumables, the tip-exchange process is both time-consuming and tedious. The user must first install a small silicon die into a custom fixture, and then align a laser to reflect from the surface of a small cantilever onto a photodiode. The reported device may be used for over 9000 scans of a sample with no visible tip wear in intermittent contact mode. Furthermore, the tip exchange process does not require any finesse, as the force sensor is integrated on-chip.

DEVICE GEOMETRY

The geometry of the reported instrument is shown in Fig.1. Lateral actuators are arranged in isothermal pairs to enable scanning at a constant temperature [5], thereby suppressing thermal coupling to piezoresistive sensors. A lever arrangement provides sufficient geometric advantage to obtain 15 x 15µm scans. A range of >60µm may be obtained as a single isothermal line profile, and scanning with various aspect ratios has been demonstrated.



Figure 1: False color SEM image of a single-chip AFM with several components highlighted.

A stiffer flexural suspension yields a first lateral mode that is $\sim 2x$ higher than the cantilever's natural frequency (Fig.2); the opposite is true for AFMs reported to date, which suffer from deleterious resonances at high scan speeds.



Figure 2a) First mode of the sc-AFM is a vertical resonance and b) second mode is a lateral resonance.

Optimal placement of heaters within actuators reduces their thermal time constants by an order of magnitude to enable fast scanning (Fig.3). In previous design iterations, the polysilicon heaters in the chevron were located within the shuttle region. Current renditions of the design place the heaters within contoured beams of the chevron. The temperature distribution of the chevron allows for 3x the amount of displacement with similar force output before mechanical failure occurs when thermal budgets are exceeded. Also, while the heat capacity of both chevrons is similar, the thermal resistance from the heater to the active material is much lower in the present design, increasing the bandwidth of the actuator. This allows the actuator to operate at higher frequencies without compromising the scan range.

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Figure 3: Temperature distribution within different chevron actuators. The left actuator shows a heater located within the shuttle of the device whereas the right actuator shows the heaters located within the contoured chevrons.

Similarly, the heater resistor location in the Z-actuator has been optimized such that sensor data can be captured at faster rates. Placing the heater beams directly under a vertical bimorph can help to distribute the heat, allowing for a faster response time (Fig.4).



Figure 4: Transient heat distribution after 1 ms of similar heat flux. The actuator on the left contains heaters that are located directly under the vertical bimorph whereas the right device contains heaters that are located at the base of the vertical bimorph. The quicker temperature generation over the bimorph allows for a faster reaction speed.

IMAGING RESULTS

Buried vias of a 0.35 μ m CMOS process were imaged using the sc-AFM to demonstrate a scan range of roughly 15 x 15 μ m. Each of the 0.5 μ m vias are spaced at a pitch of 1 μ m (Fig.5a). The topography of an air-bearing surface on a hard-disk reader head was imaged (Fig. 5b). The varying rates of material removal in the lapping process result in the observed patterns. Atomic lattice steps on SiC with 1.5 nm height have also been captured to demonstrate the vertical sensitivity of the sc-AFM (Fig.5c). The sc-AFM has also imaged various biological samples including the compound eye of a butterfly, where small striations within the ommatidium serve as an antireflective coating (Fig.5d).



Figure 5a) Buried vias spelling ICSPI and CIRFE, b) Hard drive read head, c) 1.5 nm tall SiC lattice steps, d) Compound eye of butterfly

QUASI-STATIC OPERATION

Vertical actuators with short thermal time constants have enabled the acquisition of tip-sample interaction dynamics at high data rates for the first time with an sc-AFM (Fig.6). Once the tip and sample are engaged, compressive stress in the cantilever may be measured to reveal topography. Upon retraction, the abrupt transition from attractive tip-sample interaction forces to freeoscillation is captured. The harder the contact, the more the cantilever must retract before the tip detaches from the sample. This information may be used to reconstruct images of the topography, adhesion, and dissipation in the sample simultaneously in a single pass.



Figure 6: The free-oscillation of the sc-AFM cantilever is captured with the integrated polysilicon resistors after abruptly detaching from a surface with varying degrees of contact force.

HIGH-SPEED OPERATION

To date, high-speed AFM research has employed contact mode, which causes rapid tip wear. The sc-AFM may be operated at higher speeds than conventional instruments in intermittent contact mode (AM-AFM) as shown in Fig 7a. A square-rooted sinusoidal wave is used to drive the Z-actuator into resonance in order to obtain an image that is enhanced by the Q factor of the device. The strain experienced in the piezoresistive bridge of the sc-AFM is used to adjust the PID such that the tip will be held at a constant height above the sample. Fig 7b shows the line scan of a 100 nm calibration grating, obtained with progressively higher scan rates, to demonstrate that a 50x improvement in scan speed has been achieved. Beyond 30Hz (60 lines/sec), features appear rounded because of the low-pass filter at the output stage of the demodulator. The lateral range of data also diminishes overtime, as this device contains chevron actuators that do not have the optimized heater placement. The higher thermal resistance reduces the chevron bandwidth at higher frequencies of operation.



Figure 7a) Block diagram of the intermittent contact AFM used to perform b) line scans at progressively higher speeds.

The sc-AFM can also operate in contact mode, where the cantilever is brought into contact with the sample. A DC offset is experienced between the sense and dummy piezoresistors on the sc-AFM when contact is made with the sample. As the tip is scanned across the surface, static deflections change the output of the piezoresistive bridge signal, and a PID may be used to adjust the output voltage of the Z-actuator to maintain a constant height above the sample, as shown in Fig 8a. Fig 8b shows the line scan of a 100 nm calibration grating obtained with a progressively higher scan rate. Box-car averaging is used to reduce the noise in the data. Unlike the device used in the intermittent contact, this device contains optimized heater placements in the chevron lateral actuator, allowing for higher frequency of operation. The faster scan rates do not limit the scan range.



Figure 8a) Block diagram of DC coupled contact AFM used to perform b) line scans at progressively higher speeds (50ms, 15ms and 6ms).

Finally the sc-AFM is operated in open-loop while applying an AC signal directly to the piezoresistive bridge, as depicted in Fig. 9a. After the approach, the device scans across the surface with the tip in contact. Fig. 9b shows the line scan of a 100 nm tall DVD sample. Irregular bumps are seen in the line scan, as the DVD contains significant variations in the distribution of peaks and valleys. As the line is scanned at a progressively faster rate, the quality of data does not diminish. Rates beyond 200 Hz (400 lines/sec) are possible using this method, suggesting that video rate AFM is achievable with the sc-AFM system.



Figure 9a) Block diagram of AC-coupled contact AFM used to perform b) line scans at progressively higher speeds (60ms, 30ms, 15ms, 7ms).

LONG-TERM RELIABILITY

In [6], images of a 22nm SRAM bank were reported using Scanning Thermal Microscopy (SThM). The topography was not visible by AFM, and SThM images were severely distorted. The present device obtained images of the same sample, clearly revealing the contact-level structure of the 6-transistor bit cell as seen in Fig. 10a. The tip wear was characterized by imaging the same area over the period of several days. Ghosting was observed in the first 100 images, presumably due to residual polymer coverage of the tip from the fabrication process used to release the devices. Imaging operation removes the polymer and sharpens the image. No observable changes occurred after 100 cycles and lower resolution images were taken after 700 cycles (Fig. 10b-d), in order to accelerate the distance traversed by the tip. After 9000+ cycles, no additional tip wear was observed.



Figure 10: Imagine of 22 nm SRAM cells after a) 1 cycle, b) 250 cycles, c) 500 cycles, d) 700 cycles

Conventional cantilevers are typically replaced after 50 images under the reported operating conditions. The $<1 \mu m$ of lateral drift occurs due to temperature changes in the room over the course of the imaging experiment.

CONCLUSION

The sc-AFM has been used to demonstrate consistent imaging over a 15 x 15 μ m area with a vertical resolution of 1.5 nm, both 200% better than previously reported. Quasi-static operation has been investigated, where characteristics such as adhesion can be probed. High speed line scanning has been performed, showing that up to 400 lines of data can be obtained in one second. Tip wear has been characterized through a series of lifetime tests. Scanning over the same sample area does not degrade the tip, even over the course of 9000 scans.

Near-term goals are to make video rate sc-AFM a reality and to provide high quality, fast and accurate imaging operation with minimal tip wear.

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LITHOGRAPHY FREE SELF-RECONFIGURABLE POST-RELEASE STRESS ENGINEERING FOR FIELD-PROGRAMMABLE MICROSTRESSBOTS

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ABSTRACT

Stress Engineering is a method that induces curvature in initially planar micromachined structures by deposition of a stressor layer. Post-release stress engineering occurs when the stressor layer is deposited and patterned after the release of the underlying microstructures. In this paper we present a post-release stress engineering process which patterns the stressor layer without the use of a photolithography step through a set of in-situ fabricated reconfigurable shadow masks. These shadow masks are made by the same surface micromachining fabrication process as the stress-engineered structures, and their self-reconfiguration enables precise definition of the evaporated stressor layer. The alignment of the masks is automated using surface-micromachined actuators called scratch drives Actuators (SDAs) which move the shadow masks to the desired configuration. We present both theoretical and experimental results showing the viability of the new post-release stress-engineering method. In particular, application of self-reconfigurable shadow masks to developing a suite of field-programmable MicroStressBots (i.e. MicroStressBots where the steering arms can be programmed after most of the micromachining process) is presented.

INTRODUCTION

Post-release stress-engineering using *in-situ* fabricated shadow masks was first introduced in [1] to eliminate stressor (Chromium (Cr)) layer photolithography and inherent possibility of galvanic attack. In this paper we extend the concept of post-release stress engineering to present novel designs of self-actuated reconfigurable shadow masks that automatically pattern the stressor layer, and hence program, the stress engineered MEMS microrobots (MicroStressBots) [2]. The shadow-masks are actuated using built-in tethered scratch drive actuators (SDAs), which control the level of coverage during the stress engineering process, and hence the deflection of the steering arms of the MicroStressBots. We envision a miniature portable PVD chamber that can be used to program a set of initially planar microrobots, allowing for simple in-the-field (i.e. after the fabrication process) programming of MicroStressBots prior to their deployment.

The self-reconfigurable shadow masks allows for programming of MicroStressBots [2] by customizing the coverage of the stressor layer (Cr) on the steering arms. The variations in Crcoverage in turn leads to differences in snap-down and release voltages, and thus different trajectories of motion for the individual robots. Consequently, self-reconfigurable stress engineering can thus enable simultaneous control of multiple MEMS microrobots.

RELATED WORK

The past decade has seen the development of several microscale mobile robotic systems, e.g. electrostatically driven stress-engineered micro robots (MicroStressBots) [2], resonating stepping robots [3], stick-slip magnetic walkers [4], and micro screw-based swimmers[5].

Stress engineering is a crucial part for programing the behavior of independently controlled microrobots [6]. The Stoney's equation first reported in [7] studies the effect of thin metallic films on underlying microstructures. The theory has been modified to include the effect on microcantilevers in [8]. Judy *et.*

978-1-940470-02-3/HH2016/\$25©2016TRF DOI 10.31438/trf.hh2016.67 *al.* [9] has also reported deflection of microstructures due to deposition of thin film layer. Previous work on stress engineering [7, 8] takes into account only pre-release stressor layer deposition. However, post-release stress engineering which removes the photolithography steps and the effects of galvanic attack was first described in Majumdar *et. al.* [1]. This paper extends this work by introducing self-patterning of the evaporated metallic stressor layer by selective shadow masking propelled using in-situ fabricated scratch drive actuators (SDAs).

MICROSTRESSBOTS

Simultaneous independent control of multiple microrobots on the same homogenous operating environment is a challenging task. Donald *et. al.* [10] showed that this is a highly under actuated control problem, and for a planar system can be viewed as a path matching problem which can be solved using Global Control Selective Response (GCSR) [6].

The stress-engineered MEMS microrobot (MicroStressBot for short) consists of an untethered scratch drive actuator (USDA) [11] that provides forward motion and a curved steering-arm actuator that determines whether the robot moves straight or turns. Fig. 1 shows a kinematic view of a MicroStressBot. The steering-arm actuator consists of a long cantilever beam with a circular pad and a dimple to prevent irreversible stiction. Independent parallel control of many such microrobots can be facilitated by the process of stress engineering which enables varying coverage of stressor layer deposition on MicroStressBot [2] arms and hence individually changing the snap-down and release voltages of each microrobot.



Figure 1: Kinematics of MicroStressBots.

POST RELEASE STRESS ENGINEERING

Post release stress engineering requires patterning of an evaporated stressor layer (in this case Chromium) covered by *insitu* shadow masks *after* the HF release. Hence, this process requires taking into account the gradual deflection of the test structures during the course of stressor deposition. The modelling can be performed numerically with the following equations as shown in [1],

$$d(j,i) = \rho(j,i-1) - \rho(j,i-1)\cos(iL/b\rho(j,i-1))$$
(1)

$$\rho(j,i) = \frac{6E_1h_1^2E_2h_2^2(j,i) + (4E_1h_1^3E_2 + 3E_1E_2h_1^3)h_2(j,i) + E_1^2h_1^4}{6h_1h_2^2(j,i)E_1\sigma_{ini} + 6h_1^2E_1h_2(j,i)\sigma_{ini}}$$
(II)

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$$h_{2}(j+1,i) = h_{2}(j,i) + (cr\cos(\theta(j,i))),$$
 (III)

where for each time step *j* and for each segment on the cantilever *i* the deflection d(j,i), radius of curvature $\rho(j,i)$ and Cr thickness h2(j,i) are iteratively calculated.

The MicroStressBots along with the reconfigurable *in-situ* shadow masks are fabricated by surface micromachining in a third party foundry process [12]. It consists of three mechanically independent layers of polycrystalline silicon with two oxide layers in between. As the shadow masks itself is not resistant to bending during Cr deposition, stress-resistant structures can be attached separately which prevents the shadow masks from bending. Different cantilever cross-section designs have been developed to resist bending during blanket stressor evaporation. Fig. 2 illustrates the different radius of curvature for three (a-c) type cross section, while Tab. I shows the design of microstructure (a) has the largest radius of curvature and hence minimum deflection which is the main requirement of a stress-resistant structure.



Figure 2: Cantilevers (a)-(c) with different cross sections.

Table I: Different designs of stress-resistant structures with cross sections and respective radii of curvature



SELF-RECONFIGURABLE SHADOW MASKS

Scratch Drive Actuators (SDAs) have been traditionally used as linear electrostatic actuators [13]. We employ microfabricated SDAs with movable *in-situ* shadow masks to provide reconfigurability of the final stressor layer deposition. Fig. 4 shows a twelve SDA linear actuator used to selectively cover four cantilevers from stressor layer deposition. The shadow masks and the cantilevers are made of surface micromachined polysilicon layers. The support beams are made of stress-resistant structures described in Tab. I to prevent the entire structure from bending during the stressor deposition. After the deposition process, the shadow masks were manually retracted in Fig. 4(iv) and the deflections of the cantilever beams were measured.



Figure 4: Sequence (i)- (iv) showing the shadow masks sliding over a cantilever test structures before stressor layer deposition (i-iii), and after deposition of 75nm Cr(iv).

Two separate runs of 75nm Cr stressor deposition were performed on the same test structures with similar shadow masks but of different lengths of exposed parts. The profilometric data thus obtained and shown in Fig. 5 clearly reveals the change in deflection of the same test structures with varying lengths of coverage due to the reconfigurable shadow masks.



Figure 5: Profilometric data of two test structures (a), (b) in two different runs having different Cr coverage area (deposition thickness: 75nm). The red and blue dotted lines demarcates the exposed and unexposed parts of the test structures.

Self-reconfigurable shadow mask shown in Fig. 6 has been implemented to pattern the steering arms of MicroStressBots. The shadow masks consist of a long stress-resistant beam attached to five SDAs on each side. Symmetry has been followed through the whole structure to provide equal force from each side. The beam itself is connected with the main shadow mask on one end and a retractable spring on the other. Similar to the previous design, both the MicroStressBots [2] which are used as test structures and the shadow mask are made of polycrystalline silicon layers.



Figure 6: Sequence of micrographs (i), (ii) and (iii) showing the motion of shadow masks over the MicroStressBots. Inset shows patterning of steering arms.

Optical micrographs taken before and after the Cr deposition in Fig. 7 reveals minor deflection of the shadow mask that did not affect the stressor evaporation on the steering arm.



Figure 7: (left) Self-reconfigurable shadow mask and MicroStressBots before and (right) after Cr deposition of 75nm.

Similar profilometric analysis on the MicroStressBot [2] arms were performed based on single run of 75nm Cr deposition but with different exposed areas. Fig. 8 shows that the difference in deflection of the two arms resulted from the process due to variable Cr coverage.



Figure 8: Optical profilometry data of the steering arm of MicroStressBots with different areas of Cr deposition coverage

FIELD-PROGRAMMABLE MICROSTRESSBOTS

The reconfigurable shadow masks designed in this work allow lithography-free self-reconfigurable patterning of the stressor layer over the MicroStressBot chassis thus ensuring proper programming of the individual devices. In order to enable fieldprogramming of the MicroStressBots, a small evaporation chamber can be envisioned. The shadow masks will define the area of Cr deposition dependent on the desired programming of the steering arms. This would in turn ensure independent control during multimicrorobot actuation [6].

It is also important to ensure that the backplate of the SDA is protected from stressor deposition. This is important, as blanket chrome deposition on the entire microrobot results in deformed back plate and erratic microrobot actuation. Optical profilometry of two microrobots in Fig. 9 with patterned Cr deposition with reconfigurable shadow masks (bottom) and blank Cr deposition (top).



Figure 9: (top) Backplate of MicroStressBot curved due to blanket Cr deposition. (bottom) Backplate unaffected due to patterned Cr deposition.

The two microrobots shown in Fig. 2 were placed side by side and tested on an operating environment consisting of a set of insulated parallel interdigitated electrodes. Our experiments confirmed that the MicroStressBot that experienced blanket Cr deposition is unable to move compared with the shadowed device.



Figure 10: Difference in motion of two microrobots on the operating environment. (left) Microrobot with patterned Cr deposition moves as expected whereas (right) microrobot with blanket Cr deposition does not move.

The snap-down and release voltages of each microrobot are among other dictated by the area exposed to the stressor (Cr) deposition on steering arm. The self-reconfigurable shadow masks allow us to vary the Cr deposition without the use of lithography, or any processing steps prior to stressor evaporation. The postrelease stress-engineered MicroStressBots were then tested for their two degrees of motion, translation and rotation as given in Fig. 11.



Figure 11: Composite optical micrograph of a MicroStressBot with patterned Cr deposition showing two degrees of motion.

Fig. 12 shows the two control primitives which were used to actuate the robot in Fig. 11. The use of in-situ reconfigurable shadow masks allowed us to program the robot with the respective snap-down and release voltages.



Figure 12: Control primitives for a single MicroStressBot having two degrees of motion

CONCLUSION

We presented a post-release stress-engineering process for MicroStressBots using in-situ fabricated reconfigurable shadow masks, and applied this process to several different structures including the steering-arms of stress-engineered MEMS microrobots. We showed designs of shadow masks that can be reconfigured using Scratch Drive Actuators (SDAs) prior to the deposition of the stressor layer. This enables precise definition of the curvature of steering-arms of the MicroStressBots without the need for an additional photolithography steps, greatly simplifying the fabrication process. The presented process not only avoids potential galvanic attack during final release etch of the MEMS structures but also is highly compatible with die-level post processing of devices fabricated using multi-project wafer processes [12]. Moreover, the presented post-release stressengineering process was applied to define the deflections of steering arm of several MicroStressBots which in turn helps in multi robot control mechanism. The shadow masks can be configured to cover different parts of the steering arms thus fabricating multiple microrobots of varying motion simultaneously. The reconfiguration of the shadow-masks using insitu fabricated actuators allow precise definition of curvature of planar MicroStressBots by the use of a simple small PVD chamber, and is envisioned to be used on-site (in the field) prior to the deployment of the fabricated MicroStressBots. Such capability is envisioned to have big potential to enable parallel control of large numbers of MicroStressBots.

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NEAR-NAVIGATION GRADE QUAD MASS GYROSCOPE WITH Q-FACTOR LIMITED BY THERMO-ELASTIC DAMPING

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ABSTRACT

This paper presents experimental results on the noise performance of an ultra high quality factor (Q-factor) micromachined silicon Quad Mass Gyroscope (QMG). The device was vacuum sealed at below 0.1mTorr in an LCC package with activated getters, demonstrating the Q-factor better than 1.7 million, along the drive and sense modes, at the 1.6 kHz center frequency. A bias stability of 0.09 deg/hr and Angle Random Walk (ARW) of 0.015 deg/rt-hr were demonstrated, without any thermal compensation or post-processing of data. To the best of our knowledge, the result presented in this paper is the highest reported Q-factor for MEMS silicon gyroscopes, now demonstrating a true potential to achieve the navigation-grade performance.

INTRODUCTION

High Q-factor, isotropy of stiffness, and symmetry of damping along the drive and sense modes are the essential attributes of high precision Coriolis Vibratory Gyroscopes (CVGs). Q-factor maximization of the drive- and sense-mode is a key to augment performance of the mode matched or nearly-matched CVGs.

In our previous work, a QMG with as-fabricated frequency mismatch of 0.2 Hz and Q-factor of 1.17 million has been demonstrated with 0.88 deg/hr in-run bias stability and ARW of 0.06 deg/rt-hr, operating in force-rebalance (FRB) mode [1]. A vacuum sealed silicon Disk Resonator Gyroscope (DRG) has been reported with a Q-factor as high as 80,000 at 14 kHz center frequency, reporting in-run bias stability of 0.012 deg/hr and ARW of 0.002 deg/rt-hr with active temperature control and compensation [2]. A Q-factor of up to 1.38 million has been measured on bulk acoustic wave (m=3) degenerate modes at 2.745 MHz center frequency, in an actively controlled vacuum chamber [3]. A Q-factor as high as 2.7 million has been demonstrated on a vacuum sealed single-axis silicon resonator at 570 Hz resonant frequency [4].

In this work, we present the most recent characteristics of a vacuum sealed Quad Mass Gyroscope (QMG) packaged in a ceramic Leadless Chip Carrier (LCC) with getter activation. Device was operated in an open-loop rate mode, demonstrating a Q-factor of better than 1.7 million. Figure 1 demonstrates the ringdown results, showing the Q-factor of 1.7 million and 1.8 million along the X- and Y-axis, respectively. The open-loop rate mode characterization was performed in the lab conditions with phase-locked loop (PLL) and amplitude gain control (AGC) loops, without any additional calibration or compensation loops.

RATE AND RATE-INTEGRATING GYROSCOPES

In the rate operation mode, the scale factor is amplified by the quality factor of the sense mode, thus offering a higher ratesensitivity and a reduced Mechanical-Thermal Noise (MTN). The theoretical MTN angle random walk (ARW) for the gyroscope in operating in the open-loop mode is [5]:

$$ARW_{MTN} \approx \sqrt{\frac{k_B T \omega_y}{A M \omega_x^2 Q_y}} \left[1 + \left(\frac{Q_y(\omega_y^2 - \omega_x^2)}{\omega_x \omega_y}\right)^2 \right]^{-1} \left(\frac{rad}{\sqrt{sec}}\right)$$
(1)

Here, k_B is the Boltzmann's constant, M is the gyro mass, T is

978-1-940470-02-3/HH2016/\$25©2016TRF DOI 10.31438/trf.hh2016.68 the operating temperature measured in Kelvins, ω_x and ω_y are the drive and sense resonance frequencies measured in rad/sec, and Q_y is the sense-mode quality factor. When the device is operated in the angular rate mode, the higher the quality factor of the sense mode and the lower the frequency split between the drive- and the sense-modes, the lower ARW and the higher signal-to-noise ratio of the gyro response.

In the rate-integrating operation mode, or the whole-angle (WA) mode, the Coriolis force transfers energy between two orthogonal degenerate vibratory modes and allows free precession of the sensing element. The resolution of the angle measurement is derived in [6], and is limited by the anisoelasticity ($\Delta\omega$) and anisodamping $\Delta(1/\tau)$. Thus, maximization of the energy dissipation time constant, matching modal frequencies and symmetry of the damping are the defining factors for the angular rate drift in an angle measuring gyroscope. The bias drift can be expressed as

$$\Omega_{drift} \approx \frac{1}{2} \Delta \left(\frac{1}{\tau}\right) + \frac{1}{2} \Delta \omega \tag{2}$$

Therefore, a high performance MEMS gyroscope with an interchangeable rate and rate-integrating operation mode must possess the high Q-factor degenerate modes in stiffness and damping.

VACCUM SEALED QUAD MASS GYROSCOPE

A Quad Mass Gyroscope (QMG) is a device with four symmetrically decoupled "tines" that operates in a dynamically balanced anti-phase degenerate mode [4]. The anti-phase motion of multiple masses assures minimization of the net reaction forces and moments on the anchors, which mitigates the energy loss through the substrate. Mechanical stability and mode ordering are enabled by an outer lever synchronization mechanism and inner secondary beam resonators. Four pairs of beam resonators provide the mass coupling, while widening the frequency separation between desired anti-phase mode and parasitic in-phase modes, shifting the in-phase modes to higher frequencies for common mode rejection of linear accelerations and reduction of the mode conversion losses [7].



Figure 1: Experimental results of ring-down of a vacuum sealed QMG device under test. The exponential fit to the data shows the energy decay time at (1/e) normalized amplitude.

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Figure 2: 8.6 mm footprint Quad Mass Gyroscope (QMG) is die attached and wire bonded. The device is vacuum sealed with an activated getter material in the LCC package.

QMGs were fabricated using Silicon-on-Insulator (SOI) process with a 100 μ m device layer, 5 μ m buried oxide layer, and a 500 μ m handle wafer. After fabrication and dicing, individual sensors were attached to LCC packages using eutectic bonding. Packages were dehydrogenated at 400 °C in a vacuum chamber for 3 hours prior to the die attachment. The dehydrogenation process effectively improved desorption of the trapped gas molecules in the ceramic packages. A well-suited and vacuum compatible eutectic alloy composed of 80% gold and 20% tin (AuSn 80/20) was used for the die attachment.

QMG sensors were sealed in vacuum at <0.1 mTorr using an SST 3150 sealing furnace. The in-house sealing process consists of three main steps: (1) a long (> 24 hours) vacuum bake-out step at 220 °C to effectively eliminate moisture and adsorbed water molecules on surfaces of the package, the lid, and the device, (2) activation of the deposited thin film getter material (SAES PageLid®) to maintain the high vacuum inside the sealed cavity and enable a long-term stability, and (3) thermal reflow of the eutectic AuSn 80/20 solder perform at $350^{\circ}C-370^{\circ}C$ for a defect-free bond between the Kovar lids and the ceramic packages. The Q-factors above 1 million were repeatedly achieved on QMGs using the developed sealing process, [8]. Table 1 summarizes the frequency response characteristics of four recently vacuum sealed devices, with the device #1 being the focus of this study.

Table 1: QMG device parameter extraction after vacuum seal with activated getter, exhibiting high quality factor despite their high frequency split. (Device# 1 studied in this paper).

Device #	Center Frequency (Hz)	Frequency Split (Hz)	Q-factor
1	x-axis: 1,673 y-axis: 1,688	15	1,727,000 1,828,000
2	x-axis: 2,120 y-axis: 2,085	35	1,206,000 1,102,000
3	x-axis: 2,133 y-axis: 2,089	44	1,130,000 1,040,000
4	x-axis: 2,142 y-axis: 2,098	44	1,141,000 1,125,000

ENERGY DISSIPATION MECHANISMS

Energy dissipation in microsystems, due to internal and external friction, defines the performance limit of vibratory systems. For instance, the Q-factor is a parameter of interest in resonators and CVG devices. The total Q-factor is calculated by taking all the losses into consideration. The primary energy dissipation mechanisms are viscous damping, anchor loss, and Thermo-Elastic Damping (TED). The effective quality factor is defined by the dominant energy loss mechanism. For more details on the dissipation mechanisms in a vibratory MEMS resonator. For example, our recent analysis in [4] explores the contributing factors of different dissipation mechanisms in resonators, collectively expressed as

$$\frac{1}{Q_{Total}} = \frac{1}{Q_{Viscous}} + \frac{1}{Q_{Ancho}} + \frac{1}{Q_{TED}} + \frac{1}{Q_{Other}}$$
(3)

Thermal coupling is the local heating/cooling of a material, which undergoes tension and compression. This causes local irreversible heat transfer within flexures, anchored to the structure. TED-limited Q-factor was simulated based on a 2D model of the QMG device, resulting in Q-factors of 3.55×10^6 and 2.42×10^4 for anti-phase and in-phase modes, respectively. As demonstrated in Figure 3, due to lower spring stiffness and frequency (1,703 Hz) in the anti-phase mode, the stress within this vibratory mode is much lower compared to the in-phase mode. This is due to a higher stiffness, and higher frequency (4,048 Hz) in-phase vibratory mode, resulting in higher energy dissipation within the flexure.



Figure 3: Finite element analysis of a Quad Mass Gyroscope (QMG), showing the fundamental TED limit of 3.54 million with a 1.7 kHz center frequency in the anti-phase mode.

TED and anchor losses are the two dominant energy loss mechanisms in our device. The anchor loss is defined by the symmetry and dynamic balance of the structure.

ELECTROSTATIC TUNING

To extract parameters of the device, each mode was excited independently. In order to excite and track a high quality factor resonance frequency, a narrow bandwidth loop filter in the drive and sense loops were designed. For the vibratory system, this is the time, τ , it takes for a settled drive amplitude to drop down under the free vibration to 1/e of the drive amplitude. In our experiment, the X-mode Q-factor was measured at $Q_x = 1,727,000$, which corresponds to $\tau_x = 328$ (s) at $f_x = 1,673$ Hz, and along the Y-mode it was measured at $Q_y = 1,828,000$, which corresponds to $\tau_y = 344$ (s) at $f_y = 1,688$ Hz. Q-factor measurement was repeated over three months period, demonstrating a leak-free sealing, vacuum stability and successful activation of getter, Figure 4.



Figure 4: Stability of Q-factor was measured bi-weekly, resulting in 0.02 ppm variations in the Q-factor during the period of twelve weeks.



Figure 5: Experimental result of electrostatic tuning of X-Y mode frequencies, by applying DC bias to the Y-mode pick-off electrodes.

Fabrication imperfections in QMGs are the primary cause of frequency mismatch between the drive and sense modes. To correct for imperfections, a frequency tuning is necessary. The QMG design consists of four proof masses with differential parallel electrodes surrounding each mass. In total, there are 16 electrodes that operate as forcer or pick-off for X and Y modes. In our experiments, a constant 10 Volt DC bias was applied to the drive forcer electrodes; the pick-off electrodes were used for DC electrostatic tuning. We also applied a DC voltage to the parallel plate electrodes of the mode with higher resonance frequency (Ymode), which decreased the resonance frequency along the Y-axis. The center frequency of both X- and Y- modes were monitored using an off-chip lock-in amplifier. In order to monitor the frequency mismatch under tuned condition (typically <1Hz), the following method was used. Under an open-loop condition with only the PLL locked to the drive resonance frequency with constant oscillation amplitude in the sense direction, the gyro was given an impulse disturbance. As a result of free oscillation between the principal axes of the gyro, the frequency value was detected by the sense pick-off electrodes. The frequency mismatch was extracted and minimized by monitoring the peak in power spectrum domain while actively controlling the applied DC voltage. The frequency mismatch was electrostatically tuned down to 60 mHz using 16.58 Volts DC bias on the Y-mode electrode, with the resolution of 3.5 digits of the power supply, Figure 5. As a result of DC tuning, a drop in Q-factor from 1,828,000 to 1,521,000 was observed along the Y- axis, Figure 6.



Figure 6: Experimental characterization of the Y-mode quality factor under electrostatic tuning, resulting in a drop of the Q-factor, while maintaining the mode-matched condition.

Figure 7 shows the increase in cross-coupling between orthogonal axes of the quad mass gyroscope (X-Y mode pick-off electrodes) as a result of electrostatic tuning. The frequency mismatch was tuned and detuned with respect to the X-mode. Due to the presence of anisoelasticity in a non-ideal gyroscope, the tuning along X-Y axes presumably resulted in an unbalanced reaction force on the anchor, leading to energy dissipation through the substrate. This is a likely explanation for changes in the orientation of the principal axes of elasticity as a result of unidirectional tuning. Therefore, a dedicated set of quadrature electrodes are likely required for the simultaneous and balanced tuning of frequency asymmetry and Quality factor.



Figure 7: Drive and sense mode coupling vs. frequency mismatch, normalized to drive pick-off amplitude (Y-mode) at 15 Hz frequency split under a fixed actuation input.

PERFORMANCE ANALYSIS

The open loop rate mode characterization was performed with two primary loops, including Phase-Locked Loop (PLL) and Amplitude Gain Control (AGC), Figure 8. The scale factor was extracted using a rate table, with incremental step inputs of 5 deg/sec, in the clockwise and the counterclockwise direction, resulting in an open-loop scale factor of 2.1mV/(deg/s), Figure 9.



Figure 8: Overall control structure, AGC and PLL were enabled along the drive axis of the device (X-mode). The DC electrostatic voltage was applied to the Y-mode electrode.

The experiment was conducted in a lab environment without any thermal compensation. After electrostatic tuning, a bias stability of $0.09(^{\circ}/hr)$ and an ARW of $0.015(^{\circ}/\sqrt{hr})$ were measured, Figure 10. The result shows an improvement in signal-to-noise ratio compared to the result demonstrated previously on a QMG with Q-factors of 1.1 and 1.2 million and a frequency mismatch of



 $\Delta f = 35$ Hz [8]. As expected, the scale factor of the sensor improves as the frequency mismatch reduces and the Q-factor increases.

Figure 9: Rate characterization response to clockwise and counter-clockwise rotation with different step input amplitudes of $0,\pm 5,\pm 10,\pm 15,\pm 20$ deg/sec, revealing an open-loop Scale Factor of 2.1mV/(deg/s).



Figure 10: Performance of QMG without self-calibration algorithm ("disabled"). A vacuum sealed QMG with getter $(Q=1.5M, \Delta f=60mHz, tuned)$ revealed $ARW=0.015(^{\circ}/\sqrt{hr})$ and bias = 0.09 (°/hr) in the open-loop operation. Lower frequency mismatch and higher Quality factor are expected to reduce the inrun bias stability.

CONCLUSION

We demonstrated a vacuum sealed silicon Quad Mass Gyroscope (QMG) with the Q-factor on the level of higher than 1.7 million, with a $0.09(^{\circ}/hr)$ bias stability and a $0.015(\sqrt{hr})$ ARW in the open-loop rate operation mode without any compensation or correction loops. The data was recorded in the lab conditions with temperature fluctuation from 23.4°C to 25.6°C with no thermal control or thermal compensation. Electrostatic tuning along the layout-defined X-Y axes resulted in dissipation of vibration energy through the substrate, higher cross-axis coupling, and reduction of the effective Q-factor. Electrostatic tuning along the principal axis of stiffness is a necessary condition to maintain the high Q-factor while matching frequencies of the drive and sense axes. In the current design of QMG, the only electrostatic force that can be applied are along X- or Y- directions, thus an electrode with greater tuning degree of freedom is required to compensate simultaneously for frequency mismatch and alignment of the principal axes of elasticity.

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PIEZOELECTRIC MEMS MICROPHONE NOISE SOURCES

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ABSTRACT

Piezoelectric MEMS microphones have been researched for more than 30 years. Despite many advantages, they have not seen widespread use because the signal-to-noise ratio (SNR) has been poor relative to capacitive MEMS microphones. This work describes piezoelectric MEMS microphones, utilizing aluminum nitride as a piezoelectric material, with a better SNR than many capacitive MEMS microphones on the market today. Further, this work describes and measures the noise sources in these microphones. Piezoelectric MEMS microphones with an SNR of 66 dB and 68 dB are presented.

INTRODUCTION

MEMS microphones are used in ubiquitous devices such as smart phones, tablets, and laptop computers, as well as emerging markets such as hearables and smart home devices. The microphone market is not only large but also growing rapidly. IHS, a market research firm, predicts that the MEMS microphone market will grow to 5.8 billion units per year by 2019 [1]. Many applications for microphones use arrays of microphones to focus on specific sound sources and filter out background noise. For these applications, microphone signal-to-noise ratio (SNR) and microphone matching are of the utmost importance [2, 3].

Today, all MEMS microphones on the market use capacitive sensing. Capacitive MEMS microphones typically consist of a moving diaphragm and a stationary backplate. As sound pressure causes the diaphragm to move, the distance between the diaphragm and backplate changes, causing a change in capacitance. These MEMS microphones also contain an application specific integrated circuit (ASIC). This ASIC contains circuitry to provide either a bias voltage or bias charge between the diaphragm and backplate. When the voltage or charge is held constant, the change in capacitance causes a change in charge or voltage, respectively. The ASIC then contains additional circuitry to generate an output voltage proportional to this change in charge or voltage. Capacitive MEMS microphones have been commercialized for nearly 15 years [4] and have undergone significant improvements in performance.

While capacitive MEMS microphones today are very small and achieve good performance compared to other commercially available microphones, they also have some negative aspects. Although the SNR of capacitive MEMS microphones reaches 66 dB – 67 dB [5, 6, 7], microphone customers still want better SNR. A significant noise source in capacitive MEMS microphones is the acoustic thermal noise caused by squeeze film damping between the diaphragm and backplate. Another limitation of capacitive MEMS microphones is that they are sensitive to dust and water. If a dust particle gets between the diaphragm and backplate, the microphone performance is reduced [8, 9]. Capacitive MEMS microphones can also shift in performance or fail if the diaphragm sticks to the backplate (called stiction). This can happen if the microphone is exposed to water or other liquids or can be caused by solder/flux as the microphone is installed in a device [10]. The diaphragm can also stick to the backplate if they come in contact due to high sound pressure levels. Sensitivity to dust and water are especially important for array applications because any drift in performance will cause the microphones to become unmatched. Therefore, even if a microphone array is built with well-matched

microphones, environmental contaminants can cause the array performance to be lost over time.

Piezoelectric MEMS microphones have been a topic of research since the early 1980s [11]. Piezoelectric MEMS microphones are an attractive alternative to their capacitive counterparts because piezoelectric MEMS microphones do not require the generation of a bias charge or voltage, simplifying the ASIC. Also, these microphones do not have a small capacitive sensing gap. Because piezoelectric MEMS microphones do not have the small sensing gap, one of the main noise sources in capacitive MEMS microphones, squeeze film damping, is not present in piezoelectric MEMS microphones. The absence of a small sensing gap also removes the primary source of performance degradation due to environmental factors such as dust and water. Today, much of the work on piezoelectric MEMS microphones is focused on their use in aeroacoustic applications, where environmental robustness is critical [12].

This work differs from previous works by this author in that this is the first time that an integrated circuit has been used to buffer the signal. Previous works used a discrete JFET in a common source amplifier circuit to buffer the signal [13]. By using an integrated circuit, the circuit noise can be reduced, allowing other noise sources to be measured. This work also demonstrates improved performance compared to that published in previous works.

METHOD

Piezoelectric MEMS microphones contain three parts, the MEMS transducer, the ASIC, and the package. Each of these parts is critical to the overall microphone performance. The design and fabrication of each part and the testing process are summarized in this section.

MEMS Transducer

The MEMS transducers were built at GLOBALFOUNDRIES on 200 mm wafers. An illustration of a cross-section of the MEMS die is shown in Fig. 1. The microphones consist of two layers of aluminum nitride (AlN) interspersed between three metal electrode layers. Because acoustic pressure causes an in-plane stress and the electrodes are oriented on top and bottom of each layer, this device uses the 3-1 mode of operation. During bending, the direction of stress in the top half of the cantilever plate is opposite to that in the bottom half. Therefore, by using two AlN layers and a center electrode, both layers can generate signal and contribute to the microphone output.



Figure 1: Cross-section of MEMS transducer. The MEMS transducer has two AlN layers interspersed between three metal electrode layers. The structure is isolated from the silicon substrate by a silicon dioxide layer.

978-1-940470-02-3/HH2016/\$25©2016TRF DOI 10.31438/trf.hh2016.69 Solid-State Sensors, Actuators and Microsystems Workshop Hilton Head Island, South Carolina, June 5-9, 2016 A top-down image of the MEMS die is shown in Fig. 2. The MEMS transducer consists of four triangular plates, each fixed to the substrate at the base of the triangle and free at the tip. One key design metric is the gap size between the plates. This gap size, in combination with the package geometry, will determine the low-frequency roll-off characteristics of the microphone. This geometry was selected in order to minimize the change in gap size caused by plate bending due to residual film stress variation. In this design, each plate is 490 μ m from the base to the tip. The total die is 1.4 mm X 1.4 mm and it is 400 μ m thick. The capacitance of the MEMS die is about 1.5 pF.



Figure 2: Microscope image of MEMS transducer die. The MEMS transducer consists of four triangular plates, each fixed to the substrate at the base and free to move at the tip.

ASIC

The signal that comes out of the MEMS die is at the mV level but the output impedance is high. Due to this high output impedance, the output is susceptible to electromagnetic interference and cannot drive low impedance loads. Therefore, an ASIC must be placed close to the MEMS die in order to buffer the output signal. This ASIC is built in a 180 nm CMOS process and consists of a single stage to buffer the output signal. The current consumption is 220 μ A and it is designed to contribute minimal noise to the overall noise floor of the microphone.

Package

The MEMS die and ASIC are both placed into a microphone package. The packaged microphone is shown in Fig. 3. This package consists of a substrate and a metal lid. The package serves several purposes. First, the MEMS transducer is sealed to the substrate around the microphone port. Therefore the back cavity of the microphone prevents sound pressure from reaching the opposite side of the transducer and cancelling with the pressure on the front of the diaphragm. Additionally, the metal in the package substrate along with that in the lid provides a shield to electromagnetic interference. The total package size for this study is 3.76 mm X 2.95 mm X 1.10 mm. This package size provides for a back cavity volume of approximately 4.6 mm³. This back cavity volume also adds stiffness to the system, reducing sensitivity. A complete model of the package is necessary to effectively predict microphone performance. Several examples can be found in the literature [12, 13, 14, 15].



Figure 3: Packaged MEMS microphone on a dime for scale. This package is 3.76 mm X 2.95 mm X 1.10 mm.

Testing

The microphones were tested using a plane wave tube system as described in previous work [16]. In this system, a speaker sends a plane wave down a tube of small diameter. The diameter of the tube ensures that only plane waves can travel through the tube at frequencies below about 30 kHz, with higher order modes being evanescent. Several cm from the sound source is a calibrated 1/4" GRAS microphone on one side of the tube and the microphone to be tested directly opposing the calibrated microphone on the other side of the tube. In order to measure frequency response, sound pressure across the frequency range of interest excites both the calibrated microphone and the microphone to be tested and the output of the microphone to be tested is compared to that of the calibrated microphone. This plane wave system is placed in a sound isolation chamber so frequency response and noise can both be measured in succession. The noise was measured at ambient pressure and in vacuum in order to separate sources of acoustic and electrical noise.

RESULTS AND DISCUSSION

The measured microphone frequency response is shown in Fig. 4. The microphone sensitivity at 1 kHz is -42 dB re 1 V/Pa. For this plot, the gain of the electronics has been removed so the sensitivity is that of the MEMS transducer itself. The microphone response is relatively flat from 100 Hz to 8 kHz with a resonance frequency around 12 kHz. This response is suitable for many applications, especially those targeting voice.



Figure 4: Frequency response of a piezoelectric MEMS microphone. This microphone ha a sensitivity of -42 dB re 1 V/Pa.

The microphone noise spectrum at ambient pressure can be seen in Fig. 5. This noise spectrum contains noise due to both electrical noise as well as acoustic noise. In order to separate the two, the microphone is measured in vacuum. Because this microphone has a resonance below 20 kHz, the vacuum measurements of this microphone show a peak in the A-weighting band due to mechanical thermal noise and lack of complete Therefore, the A-weighted noise of this vibration isolation. microphone in vacuum is not purely a measure of electrical noise. For this reason, a microphone with the same capacitance but a higher resonance frequency is used for the vacuum measurement. This vacuum measurement is also shown in Fig. 5. Similar to the plot of sensitivity, the gain of the electronics is removed so all noise is referred to the output of the MEMS transducer (i.e. the input of the ASIC).



Figure 5: Output noise of a piezoelectric MEMS microphone. The A-weighted output noise at ambient pressure is -108 dBV and that in vacuum in -111 dBV.

The A-weighted output noise of the microphone, integrated from 20 Hz to 20 kHz is -108 dBV. The A-weighted output noise in vacuum is -111 dBV. Because noise sources are uncorrelated, two identical noise sources will have a total noise that is 3 dB above the noise of a single source. The 3 dB difference between the electrical noise measurement and the total noise indicates that the electrical noise and the acoustic thermal noise are at approximately equal levels in this microphone. With a sensitivity of -42 dB re 1 V/Pa and a noise floor of -108 dBV, the SNR is 66 dB.

The electrical noise can further be divided into electrical noise from the MEMS transducer and that from the ASIC. While it is difficult to separate these experimentally, the modeled ASIC noise is -117 dB and that of the MEMS transducer is -112 dB. The electrical noise in the MEMS transducer comes from the dissipation factor of the AIN film. When added together, these provide a total noise of -111 dB, which is in agreement with the measurement.

Although this microphone does not have squeeze film damping, the acoustic thermal noise is still significant. This is primarily because the resonance frequency is below 20 kHz. This resonance, therefore, amplifies the noise in the A-weighting band. In order to improve the frequency response and noise floor of the microphone, a filter can be added to the microphone to flatten the frequency response. An analog notch filter has been added to the circuitry, which allows the microphone sensitivity to be unchanged through most of the band but attenuates the resonance peak. The

frequency response of the microphone with and without this filter can be seen in Fig. 6.



Figure 6: Frequency response of a piezoelectric MEMS microphone with and without a filter to attenuate the resonance peak.

This filter not only helps to create a more flat frequency response, but also reduces the impact of resonance on the microphone noise. The output noise with and without the filter can be seen in Fig. 7. With the filter, the A-weighted output noise decreases from -108 dBV to -110 dBV. Because the sensitivity at 1 kHz is unchanged and the noise is reduced by 2 dB, the microphone with this filter has a SNR of 68 dB.



Figure 7: Output noise of a piezoelectric MEMS microphone with and without a filter to attenuate the resonance peak. With the filter, the A-weighted noise floor drops from -108 dBV to -110 dBV.

Table 1 shows performance levels of several piezoelectric MEMS microphones. Because sensitivity and SNR can be improved by building larger microphones, the sensing element area has also been included in this comparison. Much of the earlier work on piezoelectric MEMS microphones used ZnO as the piezoelectric film. More recently, however, AlN has become widely used in FBAR devices, leading to improved physical vapor deposition (PVD) techniques for AlN. The piezoelectric MEMS microphone development over the past ten years has benefitted from these film improvements [17].

Table 1: Performance overview of piezoelectric MEMS microphones.

Author	Matl.	Sensing Element	Sensitivity (mV/Pa)	SNR (A- weighted,
		(mm ²)		ub)
Royer et al. 1983 [11]	ZnO	7.1	0.25	28
Franz et al. 1988 [18]	AlN	0.72	0.025	24
Kim et al. 1991 [19]	ZnO	9.2	1.0	43
Ried et al. 1993 [20]	ZnO	6.3	0.92	37
Kressman et al. 1996 [21]	PVDF	1.0	0.21	39
Fazzio et al. 2007 [22]	AlN	0.38	N/R	34
Littrell 2009 [13]	AlN	0.62	1.82	57
Williams 2011 [12]	AlN	0.54	0.039	19
This work	AlN	0.96	7.9	66, 68*

* With frequency response equalization circuit

CONSLUSIONS

Piezoelectric MEMS microphones have been a topic of research for more than 30 years. Over that time, improvements to piezoelectric MEMS microphone design, piezoelectric materials, and piezoelectric material film deposition have enabled significant improvements in performance. Much of the recent work in piezoelectric MEMS microphones has focused on their use in aeroacoustic applications because of their environmental robustness and ability to handle high sound pressure levels [12]. However, piezoelectric MEMS microphones are also capable of achieving high SNR and are, therefore, appropriate for a wide range of consumer and industrial applications as well.

This work demonstrates the highest sensitivity and highest SNR piezoelectric MEMS microphones reported in the literature. Not only is the SNR better than other piezoelectric MEMS microphones reported, but this SNR is on par with or better than capacitive MEMS microphones on the market today. This SNR along with the improved environmental robustness of piezoelectric MEMS microphones makes them especially well suited to array applications where environmental contaminants could cause other microphones to become unmatched over time.

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OPTO-MECHANO-FLUIDIC MEMS FOR EXTREMELY HIGH-THROUGHPUT PHOTONIC SENSING OF FLOWING MICROPARTICLES

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ABSTRACT

MEMS microfluidic resonators allow the high-speed label-free mechanical (density, size, deformability) measurement of nanoparticles using kHz-MHz mechanical modes. Further increasing the sampling rate is hard since it requires increasing of mechanical resonance frequency and improving detection sensitivity which is not easy to achieve with electrostatic or piezoelectric transduction methods. To address these challenges, here we present a new label-free technique for sensing rapid free-flowing particles using opto-mechano-fluidic resonator (OMFR). Unlike other optomechanical sensors till date, the OMFR confines the analytes within a simple microchannel, eliminating the dependence on particle diffusion and random binding, and therefore improves the throughput to a potential 10,000 particles/s rate with nearly 100% detection efficiency.

INTRODUCTION

Photonic resonators enable label-free nanoparticle sensing (refractive index, scattering) in fluids near the ultimate limits of detection, by reducing mode volume or by increasing the light-particle interaction time [1,2]. However, photonic measurement of mechanical properties (stiffness moduli, mass) of flowing particles remains challenging since these properties do not fundamentally couple to optical fields. State-of-the-art MEMS microfluidic resonant sensors [3, 4] are already capable of sensing the mechanical properties (density, size, deformability) of flowing nanoparticles using kHz-MHz mechanical modes [5], but further increase of speed and sampling-rate can only be obtained by increasing the mechanical resonance frequency and improving detection sensitivity. We propose that the solution lies in optomechanics, using which GHz-frequency mechanical (phonon) modes can be easily coupled to photons without being burdened by the parasitic feedthroughs that are intrinsic to electrostatic and piezoelectric detection methods [6-8]. Such high-frequency modes overcome the fundamental Nyquist limitations and can enable mechanical measurements with extremely high temporal resolution and sensitivity. Till date, most optomechanical sensors that operate in fluids are simply submerged, and thus rely on random diffusion and statistical measurements of large numbers of particles, resulting in low throughput. Recently, a new, hollow opto-mechano-fluidic resonator (OMFR) platform has emerged. The OMFRs can be internally pressure tuned [9], and they support frequencies ranging from 10 MHz - 12 GHz [8]. Since OMFR confines sample fluid within a simple microchannel optomechanical device in a similar way of the suspended microchannel resonator [3], it reduces the viscous and acoustic radiation loss, enabling optomechanical sensing of liquid density [10] and viscosity [11]. Here we present, the first OMFR sensor capable of measuring mechanical properties flowing (density, compressibility) of individual freely microparticles in a carrier fluid. We eliminate the reliance on random diffusion of the particles by flowing the sample fluid inside the resonator, therefore improving the throughput to a potential 10,000 particles/s rate. Moreover, the phonon mode spans across both the shell and the fluid, enabling the interaction with analytes in the entire fluid volume (Fig. 1b), thus making a nearly 100% detection efficiency possible.

METHODS

We microfabricate OMFRs through linear laser-drawing of fused-silica capillary preforms (Polymicro Technologies TSP-700850) [12]. The diameter of the device can be locally varied by controlling the laser power during fabrication, forming a microbottle resonator geometry (Fig. 1a,b) of typical diameter 40-60 µm. At the widest region, photonic and phononic modes are co-confined (Fig. 1b,c). A continuous wave laser monitors the fluctuations of the photonic whispering gallery modes (WGMs) of the OMFR (Fig. 1d). Thermal-mechanical fluctuations of the phonon modes (around 30-50 MHz) affect this photonic mode, allowing the phonon mode spectrum to be monitored with extremely high sensitivity in real-time (Fig. 1d) with a real-time spectrum analyzer. We infuse fluid samples with dissolved particles through one end of the OMFR using a syringe pump. The OMFR is vertically oriented and after the pumping is stopped, the particles with a density larger than that of the carrier fluid continue to flow because of gravity. Phonon mode frequency shifts and linewidth perturbations caused by the particles are monitored (Fig. 1d inset), with two digital cameras confirming the particle transits (Fig. 1d). Particle location within the OMFR can be triangulated using these cameras.



Figure 1: (a) Experimental setup. (b) Particles flowing through the core of an opto-mechano-fluidic resonator (OMFR; inset SEM) perturb the frequency and dissipation of (c) the local fluid-shell

978-1-940470-02-3/HH2016/\$25©2016TRF DOI 10.31438/trf.hh2016.70 Solid-State Sensors, Actuators and Microsystems Workshop Hilton Head Island, South Carolina, June 5-9, 2016 hybrid phonon modes (typically 30-50 MHz). (d) The phonon mode fluctuations are detected with extremely high sensitivity using a simple optomechanical test setup (inset shows example fluctuation of phonon spectrum).

RESULTS AND DISCUSSION

We experimentally demonstrate microparticle detections with the OMFR using three different analyte particles: 3 um yeast cells, 6 um melamine resin particles, and 11 um polystyrene particles, (Fig. 2). Real-time spectrograms are collected over long durations from which the center frequency of the phonon mode can be tracked (Fig. 2a), using Lorentzian curve fitting to each of the spectrogram. From the cases presented (Fig. 2a-c) with all three types of particles, we observe that particle transits generally shift phonon frequency to higher values. Less frequently, particle transits shift the phonon frequency lower. We note that the frequency shift is entirely caused by the interaction between phonon and particles since the optical mode is not perturbed during a particle transits evidenced by the unaffected optical transmission during particle transits. Also, these frequency shift pattern generated by heavier-than-water particles doesn't follow the well known mass loading mechanism used by mechanical oscillators. In order to determine the nature of the interaction between phonon and analytes, we first did a multiphysical finite-element model to compute the phonon eigenmodes of the OMFR. The cross-section of the first-order mode shape is visualized in Fig. 1c. It indicates that the observed 30 MHz mode is a breathing mode with the OMFR wall rapidly expanding and contracting. This wall motion creates a pressure field in fluid, with a pressure maximum at the center of the OMFR and a pressure node near the wall. Since the pressure mode is azimuthally symmetric, we can plot the induced frequency shift as a function of the particle radial position shown in Fig. 3a. We notice immediately that the frequency perturbation follows the mode shape of the standing wave. The highest sensitivity happens at the center of the OMFR, while a sensing null locates near the inner OMFR wall.

If we view the OMFR as an acoustic cavity, the interaction of the particle inside the cavity and the pressure mode causes an acoustic potential acting on the particle. In turn, the resonance frequency shifts due to the perturbation [13]. This interaction varies with both the properties of the fluid and particle (size, density, compressibility) [14], and the phonon mode shape [15]. If we assume the small particle only brings perturbation of the pressure field in fluid, p, such that we can approximate the perturbed field, p', as p, our model describing the frequency modification of the phonon mode, f', is presented as following:

$$f' = f_o \sqrt{\left(1 - \frac{\rho_s - \rho_l}{\rho_s}B\right) / \left(1 + \frac{\kappa_s - \kappa_l}{\kappa_l}A\right)},$$
(1)
where $A = \frac{\int_{V_s} p^2 dv}{\int_{V_c} p^2 dv}$ and $B = \frac{\int_{V_s} |\vec{\nabla}p|^2 dv}{\kappa_l^2 \int_{V_c} |p|^2 dv}.$

Here, we use subscript *l* standing for the fluid inside OMFR and the subscript *s* for the sample particle. ρ and κ represents density and compressibility, respectively. The compressibility of the particle, κ_s , is given by $1/\rho_s(c_P^2 - \frac{4}{3}c_s^2)$, with c_P and c_S representing the P-wave and S-wave velocities of the solid-phase particle material, respectively [14]. Whereas $\kappa_l = 1/\rho_l c_l^2$, where c_l is the speed of sound in the fluid. $k_l = 2\pi f_o/c_l$ is the unperturbed wavenumber for phonons in the fluid fraction. The Integration volume are V_c , which is the entire cavity volume within the OMFR, and V_s , which is the sample particle volume. Apparently, the *A* term is proportional to the acoustic potential energy over the particle volume, and the *B*

term is proportional to the acoustic kinetic energy over the particle volume.



Figure 2. (a) Real-time spectrogram output from the photodetector measures the phonon mode spectrum. Example trace is measured for 6 μ m melamine resin particles flowing through the OMFR. After performing center-frequency fitting, the phonon mode frequency trace is extracted. (b) Similar phonon mode frequency trace is collected for 3 μ m yeast cells. (c) During the particle transits of 11 μ m polystyrene particles, phonon dissipation is observed to increase, due to the particle material viscoelasticity. (d) A spectrogram example showing the transits of three consecutive 6 μ m particles is measured simultaneous by two adjacent phonon modes.

This model indicates that the frequency shift is dependent on density- and compressibility-contrast of the particle relative to the carrier fluid, and to the particle size and position. In the cases presented, the particles are heavier and generally more rigid than the ambient fluid. Thus, when the particle is at the acoustic pressure maximum (OMFR center, Fig. 1c), the potential energy of the phonon field is reduced (A is large) while kinetic energy change is negligible (B is small), resulting in a frequency increase seen experimentally (Fig. 3a). Since both A and B can be computed numerically through simulation, we can predict the phonon mode frequency shift as in Fig. 3b, which agrees well with the experimental data. Using Eqn. 1, we can experimentally measure the density and compressibility of the particle material given we know the phonon mode shape and other properties of the particle. We can even determine the density and compressibility separately since A and B vanish at different location.



Figure 3. (a) Phonon frequency shift is measured with respect to radial position for both 11 μ m and 6 μ m particles transiting through the same OMFR. (b) Simulation results obtained from our model (Eqn. 1) agree well with the experimental data.

Since the radial phonon modes extend across the entire OMFR cross-section, this system can capture measurements on essentially every particle flowing through the device. We experimentally estimate the throughput of the OMFR to exceed 10,000 particles/s. Our experiments also indicate that the phonon mode dissipation (linewidth) increases during particle transits (Fig. 2c), indicating sensitivity to viscoelasticity of the particle material [11]. Finally, our experiments show that the OMFR is capable of sensing spatially distinct phonon modes (Fig. 2d), which is potentially useful for resolving the dynamic distribution of analytes [5].

CONCLUSION

This work presents a new phonon-mediated optical technique for performing high-throughput mechanical measurements on free-flowinng particles in fluid. This resonant measurement technique doesn't depend on random diffusion and spatially decouples the optical mode from the sensing region. While a breathing phonon mode is used to prove the concept here, many more phonon modes spanning from 10 MHz to 12 GHz regimes can be used in a single OMFR structure [Bahl2013], which potentially allows the perfect capture and inertial imaging of every particle that flows through the device. More broadly, the principle we demonstrate here enables optomechanical investigations of defect states in non-solid phase materials.

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OPTOMECHANICAL TRANSDUCER-BASED SOFT AND HIGH FREQUENCY NANOSCALE CANTILEVER FOR ATOMIC FORCE MICROSCOPY

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ABSTRACT

We introduce an optomechanical transducer-based nanoscale cantilever probe for atomic force microscopy (AFM). The high optical quality factor of the microdisk resonator enables detection of the nanoscale cantilever motion with high sensitivity. The low stiffness (≈ 1 N/m) and high frequency (above ≈ 4 MHz) nanoscale cantilever provides both a wide bandwidth for fast motion detection and a high force sensitivity. We demonstrate the capabilities of the device in AFM for fast scanning (nominal 30 µm × 30 µm, 39.06 Hz line rate, 2.93 mm/s tip speed) with a fast settling time (< 2 µs). Furthermore, the detection of photo-thermal induced resonance (PTIR) signals at a 50 nm thick polymer film is also demonstrated.

INTRODUCTION

Fast-scanning AFM with sensitive force measurement is essential for investigating short time-constant motion or dynamics at small scales, and the demand for this capability is growing rapidly in various fields of nanoscale science and technology [1-3]. The key to realizing the desired performance is the use of a cantilever with a high mechanical resonance frequency (ω) , in order to provide a wide detection bandwidth, and a low stiffness (k), in order to provide high force sensitivity. These two goals can be realized by reducing the cantilever size to the nanoscale. However, if the cantilever size is reduced in this manner, optical transduction is inefficient for the typical beam bounce far-field or interferometric detection methods, used in conventional AFM systems. This occurs because the light cannot be focused only on the nanoscale cantilever due to the optical diffraction limit. For a given minimum size and mass (m), the stiffness increases with larger ω , since $k = m\omega^2$, resulting in low sensitivity.

Here we introduce a fully-integrated silicon microdisk cavity optomechanical transducer-based nanoscale cantilever to make a fast-scanning, sensitive AFM. An integrated near-field detection scheme [4] allows reduction of the mechanical probe cross-section to the 100 nm scale. A remarkable advantage of our cavity optomechanical transducer is its highly sensitive mechanical motion measurement, which is enabled by the strong interaction between photons that are confined inside the cavity and the cantilever motion. Optical shot noise, rather than thermal noise, fundamentally limits the motion readout precision, and optical power does not, in principle, have to be dissipated in the transducer. Readout precision is sufficient for the probe to operate at its fundamental thermo-mechanical limit. Among various types of optomechanical transducers such as suspended membranes [5], planar photonic crystal cavities [6], and microdisk resonators [7,8], we choose to utilize a microdisk type transducer for geometrical reasons: the cantilever can be simply incorporated around the rim of the microdisk, leading to an extended cantilever-disk interaction region, greatly enhancing the optomechanical coupling gom. This layout geometry facilitates the engineering of ω and k through geometry variations, and enables practical experiments in AFM systems. Furthermore, the integrated chip (optomechanical transducer and waveguide) is connected by optical fiber, which eliminates the need for laser alignment and provides a stable output signal.

We use a custom adapter to incorporate our transducer chip in

978-1-940470-02-3/HH2016/\$25©2016TRF DOI 10.31438/trf.hh2016.71 place of a conventional AFM cantilever chip into a commercial AFM and PTIR system, and demonstrate improved performance of both instruments.

NANOFABRICATION OF THE DEVICE

Figure 1 shows the nanofabrication process of the device. The devices are fabricated in a 260 nm thick silicon layer on top of the 1 μ m buried oxide (BOX) of a silicon-on-insulator (SOI) chip. Typically, multiple 5 mm × 5 mm individual die are fabricated on a single 3 cm × 3 cm chip. In this work electron (E)-beam and



Figure 1: Nanofabrication of the optomechanical device. (a) E-beam lithography. (b) Oxide & nitride depositions. (c) Photolithography and nitride dry etch (d) Oxide and KOH etching. (e) Cantilever releasing with wet HF etching, critical point drying, mechanical and FIB under-cut below the cantilever, and gluing the cleaved optical fiber. Inset shows sidewall profile from the Si etch, scale bar 500 nm.

multiple contact optical lithography steps are combined for defining device layers. This work is focused on device demonstration, and uses a few steps that are not compatible with batch fabrication. A much more sophisticated process aimed at batch fabrication of a wide class of similar devices is reported in a separate paper at this conference.

In the first step, E-beam lithography is used to define all features in the SOI Si, with critical features being the narrow gaps and the small cantilever width, followed by inductively-coupled plasma reactive ion etching for patterning the Si layer with a C₄F₈ passivation-based SF₆ etching technique [9] as shown in Fig. 1(a). Note that we check the etched vertical profile with a test silicon chip which has 1 μ m line pattern before device etching, as depicted in the inset of Fig. 1(a). We realize vertical and very smooth sidewall profiles, which are required for controlling optical mode shapes and achieving the high optical quality factor for high sensitivity and selectivity for in-plane mechanical motion.

Next step is the low-pressure chemical vapor deposition (LPCVD) of SiO₂ and nitride as shown in Fig. 1(b). The oxide is used as optical cladding and sacrificial layer, and nitride as a mask layer for the subsequent Hydrofluoric acid (HF) release step. Nitride windows near the microdisk/cantilever and the fiber couplers are opened by photolithography and reactive ion etching, as shown in Fig. 1(c). At this point, the chip is annealed for 1 hour at 1000 °C to densify and improve the film quality of the oxide and nitride. Following this, we lithographically pattern and dry etch all layers stopping on the Si substrate to define patterns for fiber v-grooves and for chip singulation. Potassium hydroxide (KOH) etching defines 75 μ m deep v-grooves. During this step, the SOI silicon is sufficiently protected by the BOX and top oxide layers as shown in Fig. 1(d).

We mechanically cleave to singulate 5 mm \times 5mm die, followed by mechanical polishing of the Si die corner at 45° from the backside and focused ion beam (FIB) undercut of the transducer area to expose the cantilever probe at the chip corner (Fig. 2) and to sharpen the AFM probe tip. This mechanical processing is needed for unimpeded sample approach with the in-plane fabricated probe in AFM experiments (in the future may be accomplished by an appropriate combination of scalable back and front bulk micromachining techniques). The devices are released by wet etching in 49 % by mass HF solution in water, followed by rinsing and supercritical point drying. Figure 2(a) shows side view of the die corner after mechanical milling for gross under-cut of the Si substrate under the cantilever with 45° milling angle using diamond grit sandpaper. Figure 2(b) shows SEM side view image of a finished, FIB-milled and HF-released device.



Figure 2: (a) Mechanical milling result. Si substrate under the cantilever is firstly polished by milling machine with diamond sand paper at 45° angle. (b) FIB milling is precisely performed near the cantilever area.



Figure 3: (a) Optical microscope images of the fabricated optomechanical device. (b) Installation of the optical fiber glued device into a commercial AFM system.

Figure 3(a) shows a top down optical microscope view of the optomechanical device and the v-groove terminations with Si waveguide optical couplers. The released die is attached with epoxy to a custom 3D-printed metal adapter and then bare, cleaved, single mode telecom optical fibers are actively aligned in the v-grooves to the on-chip inverse-taper optical couplers using a 1550 nm wavelength laser source. The fibers are glued into the v-grooves and to the adapters using ultra-violet (UV) curable epoxy. The fiber facet and the suspended Si inverse taper structure at the end of the v-grooves typically remain epoxy-free.

We install this optical fiber glued device into a commercial AFM system. We only use the chip cantilever chip holder and the sample scanner of the AFM, while our optomechanical signal is fed back into the instrument in place of its normal far-field beam-bounce optical detection system as shown in Fig. 3(b). The active corner on the die with the nanoscale sharpened probe is oriented down toward the sample stage of the AFM.

DEVICE DESIGN AND CHARACTERIZATION

Figure 4(a) shows an optical micrograph of the fabricated integrated silicon cavity optomechanical transducer with a basic schematic of the AFM measurement. The nominal fabricated disk diameter is 10 μ m, while the cantilever nominal width and length are 120 nm and 24 μ m, respectively; the gap between cantilever and microdisk is \approx 200 nm, and the tip radius after FIB sharpening is \approx 15 nm, which is comparable to the tips of commercial AFM cantilevers.

Figure 4(b) shows the working principle for a cavity optomechanical transducer-based near-field detection of cantilever motion. The mechanically-stationary microdisk is a high quality factor whispering gallery optical cavity that can be sensed through an evanescently coupled Si waveguide. Coupling into a cavity optical resonance mode results in a spectrally-narrow dip in waveguide transmission, Fig 4b. Optical quality factors of the 10 μ m diameter microdisk resonator typically lie between 15 000 to 100 000 depending on the surface cleanliness of the microdisk rim, the roughness of the etched sidewall, and the geometry of the waveguide-microdisk coupling area.

The cantilever is too narrow to support a guided optical mode and to guide any light away from the optical cavity. Thus it does not affect the cavity optical loss. Instead, moving the cantilever into (out of) the evanescent fields of the cavity lowers (increases) the cavity optical resonance frequency, shifting the transmission dip. The shift of the spectrum can be translated into an amplitude modulation of the transmitted light by tuning the excitation laser to a fixed wavelength on the shoulder of the resonance, Fig. 4(b). Therefore, the photodetector voltage becomes linearly proportional to the cantilever position. Figures 5(a) and 7(b) shows that the motion readout noise is low enough to clearly resolve the thermal fluctuations of the mechanical probe itself, both when free and in contact with the sample. Therefore, the measurement performance is determined by the probe's thermal mechanical noise.

The non-contact noise spectrum for the cantilever used in the AFM experiment is shown in Fig. 5(a), measured with \approx 3 mW of input optical power. The mechanical stiffness is estimated by finite element calculation to be \approx 1 N/m and cantilever mass is in the sub-picogram range. The observed resonance frequency of \approx 4 MHz agrees well with the calculation. These parameters together with the measured quality factor Q \approx 20, are used to calibrate the sensitivity using the equipartition theorem. This gives the readout noise background in Fig. 5(a) of \approx 7 fm/Hz^{1/2} and the thermodynamic



Figure 4: Optomechanical transducer-based nanoscale cantilever. (a) Device and detection schematic. (b) Working principle.



Figure 5: (a) Measured mechanical resonance frequency (4 MHz). (b) Simulation results of corresponding mechanical resonance.

Langevin force noise density of $\approx 6 \text{ fN/Hz}^{1/2}$ acting on the cantilever, in air. Note that we can, in principle, widely vary the cantilever resonance frequency (200 kHz to 110 MHz) and stiffness (0.01 N/m to 290 N/m) by varying the design of e-beam lithography pattern [10].

This motion readout signal can be used by the AFM system as the cantilever "deflection" signal. Following conventional contact mode protocol, the AFM closes a feedback loop by adjusting the height of the probe relative to the sample to maintain deflection near a fixed setpoint value, adjusting for the changes in the sample topography as the probe is scanned.

EXPERIEMTNAL RESULTS

We performed fast scanning AFM and a PTIR measurement on a single location with the optomechanical transducer-based nanoscale cantilever.

Fast scanning

Figure 6(a) shows the contact-mode fast-scanning AFM image of a 3 μ m period grating in ambient conditions. A wide area of 30 μ m × 30 μ m is imaged a high resolution (512 × 512 pixels) using a line scan rate of 39.06 Hz in the fast direction, which corresponds to a cantilever linear speed of 2.93 mm/s (total scan time: 13.5 s). Note that these times and frequencies properly account for the additional scanner turn-around time between each line. Even at this speed, the cantilever followed the sharp 25 nm high steps of the surface topography of the grating as shown in the Topo + Deflection images



Figure 6: AFM measurement of a test grating. (a) AFM image of the 25 nm tall grating lines. (b)-(d) a single line scan. Experimental uncertainty is smaller than the data marker size.



Figure 7: PTIR measurement. (a) Optomechanical probe is used instead of a conventional AFM cantilever and its beam-bounce readout in PTIR nanoscale chemically-sensitive imaging system. (b) Cantilever probe is limited by fundamental thermodynamic mechanical noise which is lower than thermal noise of conventional cantilevers in air. Noise power is relative to 1 mW. (c) Signal from a 50 nm thick PMMA film.

and line profiles in Fig. 6(b). The cantilever deflection changes very quickly at the grating steps, followed by the slower compensation by the AFM z feedback ("topography" channel). At the steps, the cantilever climbs up the wall (Fig. 6(c)) and descends the valley (Fig. 6(d)) with the response that is within about 20 % of the final value achieved within the 2 μ s single time step duration. Based on the standard deviation among 10 adjacent points on a flat portion of the sample, taken at 2 μ s per point, the noise per point is estimated as 20 pm one standard deviation.

PTIR measurement

We have integrated our optomechanical probe into a commercial photo-thermal induced resonance (PTIR) system [11]. PTIR combines the nanoscale resolution of AFM and optical spectroscopy and enables measuring local infrared absorption spectra and mapping chemical composition with nanoscale resolution [12,13]. The system operates by illuminating a thin-film sample with a short pulse from a tunable infrared laser. If the sample at a given location absorbs the specific infrared wavelength of the pulse, the optical energy is locally converted into heat and results in surface motion due to thermal expansion. An AFM cantilever in contact with the sample at such location experiences a mechanical "kick" which is detected. Typically, the short thermo-mechanical kick from the sample excites one or more mechanical resonant modes of the cantilever, and amplitude of the cantilever vibration ringdown provides a measure of local infrared absorption.

The exquisitely high-precision motion readout and low thermal noise of our nanoscale sensor compared to larger cantilevers in air will enable fast PTIR measurements of extremely thin samples. The low readout noise means fewer ringdown traces have to be averaged to achieve good signal-to-noise ratio at each point, and therefore faster acquisition of images and absorption spectra will be possible. By substituting our nanoscale cantilever and optical readout instead of a conventional microscale cantilever and beam-bounce readout (Fig. 7(a)), and leveraging the smaller thermomechanical and readout noises of our system (Fig. 7b), in preliminary experiments we were able to easily measure the PTIR ringdown signals from a test film of poly (methyl methacrylate) (PMMA) only about 50 nm thick (Fig. 7c) with 1024 averages.

CONCLUSION

We have integrated an optomechanical transducer-based nanoscale cantilever into AFM and PTIR systems. We demonstrate fully-functional contact mode AFM imaging with the probe, highlighting fast scanning and low noise. We further integrate the probe in a photo-thermal induced resonance system, and detect PTIR signal from a thin sample.

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PIEZOELECTRIC PRESSURE ENERGY HARVESTERS USING CIRCULAR DIAPHRAGMS WITH CONCENTRIC RING-BOSS STRUCTURES

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ABSTRACT

This paper describes the design, fabrication and testing results aluminum nitride piezoelectric micromachined circular of diaphragm energy harvesters with concentric ring-boss structures to convert pulsed pressure to electrical power with high efficiency. Compared to the state-of-the-art, the concentric-ring-boss harvesters (CRBH) produce multiple strained regions for: (1) more effective electrode areas; (2) high charge extraction rates; and (3) high power outputs. Finite element modeling is used to evaluate the effectiveness of the proposed design by comparing stress and stretching energy of various designs. The conventional single-boss and concentric ring-boss harvesters have been fabricated and tested under a mean pressure of 1.5 kPa with 1 kHz frequency. Experimental results show the ring-boss and the conventional single-boss harvesters can generate power of 1.3 and 0.82 μ W, respectively. As such, the CRBH is 1.6X higher in energy conversion efficiency and power density as compared to single-boss design of similar dimensions.

INTRODUCTION

Battery replacements have become a practical problem for long-term sustainable implantable systems such as pacemakers, deep brain stimulators, and telemetry systems for epilepsy and chronic pain treatments. In general, patients have to undergo a surgical procedure for battery replacement in every two or three years (depending on personal usage), which is not only a medical risk but also an economic burden. Therefore, it could be desirable to develop an implantable power source which can generate electrical power within the body continuously. Recent advances in device designs of microelectronics and low power circuit have reduced the power requirements for the implantable devices considerably, making energy harvesting within human body even more attracting as a viable solution.

Recent efforts to harvest energy within human body include a vibration based energy harvester utilizing eardrum vibrations [1]; a glucose fuel cell to convert glucose content of cerebrospinal fluid to electricity [2]; and a flexible ZnO-based piezoelectric sheet to convert heart and lung deflections to electricity [3]. We aim to make use of pressure fluctuations of body fluids as a power source; thus, in this study we propose a novel circular diaphragm energy harvester design to enhance the power output. Micro-scale pressure energy harvesters generally utilize a circular diaphragm to convert incoming pressure waves to mechanical energy, and the stored mechanical energy is then converted to electrical energy using the piezoelectric effect. Various researchers have proposed pressure harvesters for different applications. Lai et al. proposed a SiC/AlN harvester having electrode covering majority of the surface for high temperature environments and operation frequencies of 1-2 kHz [4]. This harvester has a single-boss structure to increase the stress on the diaphragm and decrease the system natural frequency. Horowitz et al. have proposed a harvester design utilizing the Helmholtzresonance phenomena to convert acoustic waves between 1-4 kHz, and a power density of 0.34 μ W/cm² ^u under 149 dB has been reported [5]. Kimura et al. developed a circular diaphragm harvester for audible sound and demonstrated improved power density when operating in the third resonance mode $[\hat{6}]$.



Figure 1: a) The proposed harvester with a concentric ring-boss structure (a single ring in this example); b) the conventional circular single-boss harvester; c) dimensions of the structure shown on axisymmetric cross-sectional view; d) simulation results of stress on the top surface versus radius plot for various r_0 and w combinations under an applied pressure of 1 kPa.

CONCENTRIC RING-BOSSED HARVESTER (CRBH)

In this study, our aim is to develop a harvester which is capable of converting pressure fluctuations in fluid medium with increased power density and higher efficiency. The majority of the designs for pressure energy harvesting utilize either a single-boss structure or multiple electrodes. The boss structure can reduce the natural frequency of the whole system and increase the stress around the boss and membrane interface regions. However, the area on top of the boss itself becomes a zero-strain region without the possibility to generate extra charges. On the other hand, the placement of multiple electrodes around the membrane can cover larger stressed regions to increase harvested energy. However, the electrodes need to be placed at regions that have large strain under the applied pressure to be effective. However, as the high strain areas generated under the fundamental vibration mode are fully covered by electrodes, the second or higher vibration modes will be required for larger areas with high strain regions and the structural frequency will increase considerably. The concentric ring-boss design together with multiple electrodes can increase the high stress regions on diaphragm while operating under the fundamental vibration mode. Figure 1 illustrates the design of CRBH with a circular diaphragm and a concentric ring and boss underneath.

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Table 1: Structural material properties used for simulations [10].

	AlN	Silicon
Density (kg/m ³)	3300	2329
Young's modulus (GPa)	330	170
Poisson's ratio	0.24	0.28

The concentric ring-boss structures provide several high stress regions on top of the diaphragm in operation under the first mode. For example, the single ring structure as shown in Figure 1a produces two high stress regions as compared to the single high stress region from the single-boss structure. Depending on the application and operating frequency, it is also possible to increase the number of the ring structures to increase the number of strained regions on the diaphragm. Figure 1b shows critical dimensions of the CRBH, such as overall radius of the diaphragm, R_1 , outer radius of the ring, R_2 ; width between the ring and boss, w; and the distance from the center of the diaphragm to the midpoint between the edge of the boss and the inner radius of the ring, r_0 . In order to show the effect of the proposed design, Comsol finite element program was used to simulate stress generated on the harvester diaphragm upon an applied pressure. Figure 1d shows the stress generated on the CRBH for different combinations of r_0 and w. Compared to the conventional design shown in Figure 1c, the proposed design has larger stress concentration regions while preserving the amplitude of the maximum stress. By utilizing a piezoelectric layer as a diaphragm, the enhanced stress regions can provide additional charge extractions for higher power outputs.

In order to show the effect of this design, we investigated mechanical energy of the diaphragm upon applied pressure [7, 8]. The work done on the diaphragm when a pressure is applied consists of stretching (due to extension of the middle plane of the diaphragm) and bending (due to transverse deflection) components. Because stretching energy, V_s , is orders of magnitude higher than the bending energy, V_b ; we focus on V_s generated on the diaphragm which is calculated as:

$$V_{s} = \pi D_{s} \int_{0}^{R_{1}} \left(\varepsilon_{r}^{2} + \varepsilon_{\theta}^{2} + 2\nu_{p}\varepsilon_{r}\varepsilon_{\theta} \right) r dr$$
(1)

where \mathcal{E}_r and \mathcal{E}_{θ} are radial and circumferential strains, respectively; v_p is the Poisson's ratio; D_s is the stretching stiffness; and r is the radial coordinate. Figure 2 shows the simulated stretching energy versus r_o for the boss-ring structure of different w and single-boss harvester. These harvesters have 1.25 mm as R_1 , and the outer radius of the ring structure, R_2 , is 1.1 mm.



Figure 2: Strain energy by Comsol simulation of the CRBH $(R_1=1250 \ \mu m \ and \ R_2=1100 \ \mu m)$ and single-boss harvester $(R_1=1250 \ \mu m \ and \ boss \ radius \ of 1100 \ \mu m)$ versus r_0 .



Figure 3: Optimization results of the electrode width, L, using Comsol finite element program using same configuration as in Figure 2 with r_0 and w of 600 µm and 200 µm, respectively.

According to the simulation results, the proposed design is able to generate 1.75x higher stretching energy with r_o of 600 μ m and wof 300 μ m as compared with the conventional single-boss structure. As such, the piezoelectric material is able to generate more energy per unit time compared to the conventional design.

Another critical element that should be considered is the radial electrode width [9]. An optimization study was as shown in Figure 3 to find electrode widths for maximum power output. Key aluminum nitride piezoelectric material properties used during simulations are as follows:

	28.6	-8.99	-3.83	0	0	0]
$s_E =$	0	31.2	-9.65	0	0	0	
	0	0	30.4	0	0	0	10^{-12} [m^2 / M^2]
	0	0 0	0	8.05	0	0	$\begin{bmatrix} 10 & [m / N] \end{bmatrix}$
	0	0	0	0	8.05	0	
	0	0	0	0	0	8.05	j
	-						-
	0	0	0	0	-3.84	0	
<i>d</i> =	0	0	0	-3.84	0	0	$10^{-12}[C/N]$
	-1.73	-2.78	5.49	0	0	0	j
	F a a 4		٦				
$\varepsilon_r =$	9.21	0 0					
	0 9	9.21 0					
	0	0 10.	3				

The simulated single-ring CRBH design has the same configuration as in Figure 2 with r_o and w of 600 μ m and 200 μ m, respectively. It can be seen that the electrode radial width, L, can influence the maximum power output and maximum power output is obtained for $L=60 \ \mu$ m which corresponds to an area coverage of 36% along the width of the harvester. Furthermore, the widths included a 5 μ m "non-active" portion on top of boss or ring regions to account for possible misalignments during the electrode fabrication process.

FABRICATION

Figure 3a shows CMOS-compatible fabrication flow of the proposed harvester. A stack of AlN/Mo/AlN/Mo layers were deposited on a <100> Si wafer. The first 1 μ m-thick AlN layer serves as structural (passive) layer as well as the stop layer during final backside deep reactive ion etching process, while the second 1 μ m-thick AlN layer serves as the active piezoelectric layer. Mo layers of 100 nm in thickness serve as bottom and top electrodes for



Figure 4: a) Fabrication flow of the proposed harvester, b) image of the fabricated CRBH and single-boss harvesters showing front and backside of the chips on a \$1-cent US coin.

piezoelectric layer. The first Mo metal layer was patterned to form the top electrode by a chlorine-based reactive ion etching process, then the active AlN layer was patterned to allow contacts from the bottom electrode. Finally, diaphragms were released after backside deep reactive ion etching (DRIE) process.

Figure 3b shows the fabricated chips which have both the proposed CRBH and the single-bossed harvesters on \$1-cent US coin. All of the fabricated harvesters have a diaphragm radius, R_I , of 1250 μ m. In order to employ a fair comparison between the two designs, the single-bossed harvester has a boss radius of 1100 μ m, while the CRBH's all have outer ring radius, R_2 , of 1100 μ m with different combinations of r_0 , w, and L.

CHARACTERIZATION

In order to characterize the proposed CRBH and compare its performance with respect to the conventional single-boss harvester, a test setup mimicking a pulsed-pressure environment was built. A commercial rotating fan having equally spacing slits was utilized and connected to a rotational speed controller circuit. Figure 5 depicts the setup. A pipe with static airflow is mounted on top of the fan and the harvesters and frequency of pressure pulsations was controlled by adjusting the rotation speed of the fan. A pressure sensor is placed next to the harvester to measure the frequency and the amplitude of the pulsations. In order to give quantitative expressions for the amplitude of the pressure fluctuations, it was presented as rms values in the experimental results. Table 2 gives the dimensions of the tested harvesters. Since there are several electrodes on the harvester, various types of electrical connections



Figure 5: Test setup used and electrical connections of the harvester for characterization of the harvesters.



Figure 6: Measured pressure pulsation waveform around the harvesters at 1 kHz, rms pressure of 0.95 kPa and maximum pressure of 2.34 kPa.

can be made for the characterizations. The first type employed here was to verify the polarity of the generated electric fields of the oppositely stressed regions (i.e. compressive and tensile) which can be done by dividing the electrodes into groups and coupling them with the ground electrode [11].

Figure 6 shows the measured pressure pulsation waveform around the harvesters at 1 kHz with the rms pressure of 0.95 kPa and maximum pressure of 2.34 kPa. Figure 7 gives the voltage readings of the harvesters to verify the oppositely stressed regions to be in opposite polarity when excited. Due to the relatively high natural frequency of the harvester compared to the excitation frequency, oscillations with frequency of approximately 22 kHz are also visible in the voltage-time plot.

Table 2: Dimensions of the tested CRBH and single-bossed harvester (SBH) (all dimensions are in μ m's).

	R_1	R_2	r_o	w	t _{piezo}	<i>t</i> _{str}
CRBH	1250	1100	675	150	1	1
SBH	1250	1100	-	-	1	1

Another and probably more practical method is illustrated in the Figure 5, where resistive load is connected to ends of oppositely stressed regions and bottom electrode is connected to a common ground to simplify the routing and to increase the output voltage. Figure 8 shows the characterization results and comparison between the voltage and power outputs of the proposed CRBH and the singleboss structure with similar dimensions. It should be noted that due to the $1M\Omega$ internal resistance of the oscilloscope, it was not



Figure 7: Output voltage of the CRBH under periodic pressure pulsations of 1kHz. Ch-1 and Ch-2 are voltage readings between conversely stressed regions (i.e. tensile and compressive) and ground electrode.



Figure 8: Characterization results and comparison of the CRBH and the single-bossed harvester, a) power-load resistance plot, b) voltage-load resistance plot.

possible to go beyond $0.82M\Omega$ during experiments. Figure 8a shows power output from the harvesters when excited with rms pressure of 1.5 kPa at frequency of 1 kHz. Because of the lower internal resistance of the CRBH, the optimal resistance of the tested harvester was observed during experiments. On the other hand, for the single-boss harvester, due to the oscilloscope's internal resistance limitation, it was not possible to go beyond the optimal point. However, it can be seen that power plot of the single-boss harvester was already saturated around 0.8 M Ω , thus does not affect its maximum power output considerably. Figure 8b is the voltage output for when various load resistances were connected to the harvesters.

The proposed CRBH design was able to generate $1.3 \,\mu\text{W}$, while the single-bossed harvester generated $0.82 \,\mu\text{W}$ under similar conditions. Conversion efficiency for pressure energy harvesters can be calculated using $\Pi = P_{out}/(IA)$, where P_{out} is the output power, and I is intensity of incoming pressure waves, and A is the surface area [12]. The conversion efficiencies for the characterized CRBH and single-bossed harvester are calculated as 0.0051% and 0.0032%, respectively. Therefore, it is evident that the proposed CRBH is able to generate higher power output that the single-boss harvester under same conditions.

CONCLUSION

Concentric ring-boss harvesters have been designed, fabricated, and tested. The CRBH is similar to the conventional single-boss harvesters in terms of fabrication but has the advantages of higher power output and better energy conversion efficiency. Furthermore, the number of ring structures can be increased to further increase the energy conversion efficiency, which might bring fabrication challenges especially during backside deep-reactive-ionetching process. Another advantage of the CRBH is that it allows for various electrical routing possibilities such as series or parallel connections of electrodes. By using serially connected electrodes, from the CRBH with diaphragm radius of 1.2 mm, maximum power of 1.3 μ W (1.6X increase as compared to the single-boss harvester) was generated under the rms pressure of 1.5 kPa at 1 kHz, which translates as a power density of 26.5 μ W/cm². It is believed that such kind of harvester having higher conversion efficiency would be critical, especially under low frequency excitations, such as human body. In future studies we will be exploring the usage and effectiveness of the proposed device in low frequency environments.

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STABILIZATION OF CORIOLIS VIBRATORY GYROSCOPES BY FREQUENCY LOCKING TO ULTRA STABLE CLOCKS

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ABSTRACT

This work demonstrates experimentally, for the first time, stabilization of a MEMS Coriolis Vibratory Gyroscope (CVG) drive loop by frequency-locking to an ultra-stable clock acting as a frequency reference. A theoretical model is presented to explain the experimental results and predict ultimately achievable rate bias instability performance based on a variety of high stability reference oscillators. A 600 μ m diameter, 40 μ m thick silicon disk resonator gyroscope (DRG) operated in its n = 3 wineglass mode is employed as the proof of concept test device and the stabilized drive loop is implemented using a Zurich Instruments HF2LI digital lock-in amplifier.

INTRODUCTION

MEMS Coriolis Vibratory Gyroscopes (CVGs) have favorable cost, size, weight, and power compared with conventional gyroscope technologies, but suffer from relatively poor stability [1]. To date, microfabrication and environmental sensitivity hurdles have limited the adoption of MEMS gyroscopes in high-precision applications. A significant effort has been expended by the MEMS research community on novel fabrication and interrogation methods for MEMS CVG resonators, seeking mainly to increase the stability of these devices in the gyroscope application through improvements in symmetry, mitigation of energy losses to maximize mechanical quality factor (Q), and elimination of conductive over-coatings which damage the pristine nature of the as-fabricated structures [2-7]. While a great amount of progress has been made, and many novel technologies proposed and developed at the device level, these approaches all rely on the CVG resonator itself as the source of stability in the gyroscope readout system, in addition to its function as the rotation sensitive component.

We propose an alternative approach to improving stability of MEMS CVGs, in which the source of system stability comes from a separate, ultra-stable frequency reference, wherein the CVG resonator only serves as the rotation sensitive component. HRL has recently demonstrated that significant improvement is possible in the stability of a silicon disk resonator gyroscope (DRG) by controlling the temperature of the DRG to a few milli-Kelvin, which ultimately can be performed on a micro-ovenization platform with integrated heaters and resistance temperature detectors (RTDs) [8]. The present work builds on the frequency-locked ovenization scheme in [8] by moving the frequency locking concept directly into the gyroscope control loops and introducing an ultra-stable frequency reference.

CONCEPT AND THEORY

HRL's new control scheme treats the MEMS CVG resonator as a gearbox on a highly-stable clock, which could be a commercial chip-scale atomic clock (CSAC), a conventional ovenized quartz crystal oscillator (OCXO), or an emerging high-performance MEMS oscillator. Figure 1a shows the typical or measured clock stability in terms of Allan Deviation (ADEV) of fractional frequency of a few potential reference clocks. Based on the seminal Lynch model [9], HRL has developed a theoretical model which relates the gyro bias instability ADEV ($B(\tau) = \sigma_{\Omega}(\tau)$) to the clock ADEV ($\sigma_y(\tau)$) which is assumed to represent the contribution of each individual mode to residual frequency mismatch (Δf) between the mode-matched and stabilized CVG control loops, where k is the CVG angular gain and Q is the quality factor of the CVG modes:

$$B(\tau) = \frac{90}{k} \frac{\sqrt{2}f\sigma_y(\tau)}{Q} [^{\circ}/s]; \sqrt{2}f\sigma_y(\tau) \approx \Delta f.$$
(1)



Figure 1: (a) Measured and typical Allan Deviation (ADEV) of fractional frequency of potential reference clocks shows that a pairing of an OCXO and a CSAC will provide the best stability over a wide range of integration times. (b) Reference clock stability converted to equivalent ultimate gyro stability showing that an 8 mm silicon DRG will exceed its state-of-the-art intrinsic performance [8] when locked to either an OCXO or a CSAC.

The above model, when applied to measured 8 mm diameter, 250 μ m thick silicon DRG drive loop frequency stability (purple trace in Figure 1b) with the addition of thermomechanical noise using the well-known gyro angle-random-walk (ARW) formula [10], closely matches (within an order of magnitude) measured rate ADEV of a silicon DRG precisely controlled down to 3 mK (green trace in Figure 1b) [8]. The extra experimental noise beyond the model is expected to arise due to additional bias instability resulting

978-1-940470-02-3/HH2016/\$25©2016TRF DOI 10.31438/trf.hh2016.73 from demodulator errors. This result affirms the model is capable of predicting the ultimate CVG noise when all parameters are controlled to high stability. Using the predictive capability of HRL's new model, stabilizing the DRG to a CSAC (red trace in Figure 1b) or an OCXO (blue trace in Figure 1b) would give orders of magnitude improvement in ARW and bias instability beyond state-of-the-art MEMS gyroscopes.

Figures 2 and 3 present the original frequency and rate stability data for the 8 mm DRG from [8] which was used in the analysis presented in Figure 1b to derive the purple and green ADEV curves.



Figure 2: 8 mm DRG frequency versus RTD temperature variations over a 14 hour run showing a total variation in the frequency and DRG temperature of 2.6 mHz and 5.8 mK, respectively [8].



Figure 3: 8 mm DRG rate output versus RTD temperature variations over a 14 hour run [8].

EXPERIMENTAL DEMONSTRATION

CVG Device

The demonstration vehicle for proving the concept of frequency locking the control loops of a CVG to an ultra-stable clock was chosen to be a 600 μ m diameter, 40 μ m thick variant of the silicon DRG which was available in large quantities thanks to the Stanford epi-seal MPW run funded by DARPA. This resonator was designed at HRL based on the existing 8-fold symmetric DRG,

with appropriate modifications to fit into the Stanford process. Also, the electrodes were designed to be paired periphery electrodes in contrast to the internal electrodes of the standard DRG [10]. An SEM of this structure is shown in Figure 4.



Figure 4: SEM of HRL's 600 µm diameter, 40 µm thick silicon Disk Resonator Gyroscope (DRG) fabricated courtesy of DARPA and the Kenny group at Stanford. SEM courtesy of Yushi Yang at Stanford.

The overall die size including electrodes, pad and routing metal, and vias was 2 mm by 2 mm as shown in in Figure 5. Also presented in Figure 5 is the pin assignment and wirebonding configuration. Since the device was fabricated on a (100) silicon wafer, it is apparent that the n = 3 mode is the appropriate mode of choice for gyroscope operation due to the expected low frequency split. The traditional n = 2 modes of the DRG are expected to have a large frequency split on a (100) wafer, limiting their usefulness for gyroscope application. The epi-sealed die was attached to a 24 pin CERDIP package for easy interfacing of the device.



Figure 5: Wirebonding configuration and pin assignment of 2 mm by 2 mm epi-sealed silicon DRG die mounted in 24 pin CERDIP package.

The measured resonances are shown in Figure 6 along with finite element predictions of the modal frequencies and shapes. The measured frequencies and frequency splits agree well with their finite element predicted values. The n = 2 modes are situated in the range 240 to 260 kHz, and exhibit the expected large frequency split of 12 kHz. The n = 3 modes measure around 311 kHz and exhibit a frequency split of only 270 Hz. Tuning experiments have shown that up to 500 Hz of tuning range is available on these devices with only 5 V of DC tuning, which is sufficient to mode match the DRG.



Figure 6: Measured and simulated n = 2 and n = 3 resonances of the 600 μ m diameter silicon DRG. The n = 2 modes measure 242.34 kHz and 254.84 kHz, respectively for a 12.5 kHz frequency split. The n=3 modes measure 311.48 kHz and 311.75 kHz, for a frequency split of only 270 Hz.



Figure 7: Zurich Instruments HF2LI Digital Lock-In Amplifier used to demonstrate frequency locking of a gyroscope control loop to an ultra-stable frequency source. The 600 μ m diameter silicon DRG is interfaced to the HF2LI through a PCB breakout board which contains the 24 pin CERDIP socket and direct pinout to SMA connectors.

Test Setup and Experimental Results

To demonstrate experimentally that a MEMS CVG can be locked to a stable frequency source and inherit its stability, HRL has locked the drive loop of the 600 μ m silicon DRG to an OCXO and improved the loop frequency stability > 10 X using a benchtop system centered on a Zurich Instruments HF2LI digital lock-in amplifier that includes a 10 MHz high-stability OCXO reference (Figure 7). The HF2LI was fitted with the Ultra-High-Stability (UHS) option, which provides short term stability < 50 parts per trillion, long term aging of < 0.4 ppm/year, and a phase noise of -140 dBc/Hz @ 1 kHz offset from carrier.

The block diagram of this digital implementation of frequencylocking is shown in Figure 8a. The DRG is operated in its n = 3wineglass mode which is sensed using a transimpedance amplifier (TIA) fed into the PLL block to adjust the local oscillator (f_{drive}) to track the natural resonance of the DRG. The measured ADEV shows the intrinsic frequency stability of the unstabilized DRG is 2 ppb at approximately 60 s (Figure 8b top line). A second oscillator is used to provide a reference frequency $(f_{set-point})$ in the tuning range. The output signal of the DRG is demodulated at $f_{\text{set-point}}$, providing a phase difference which is routed to the input of a PID controller with a phase set-point equal to the natural resonance of the DRG. The PID applies a control voltage to the frequency tuning port (T) of the DRG, tuning its resonance to $f_{\text{set-point}}$. In this case, the tuning port consisted of applying an equal DC voltage on both the clockwise and counterclockwise quadrature tuning electrodes due to the n = 3wirebonding and electrode configuration. Future devices are expected to have independent tuning ports for each independent wineglass mode. The ADEV of this frequency-locked DRG is plotted in Figure 8b (bottom line) and does not show a minimum for all times measured (up to 1,000 s). At 60 s, the stability is better than 0.1 ppb, 20 X better than without locking. Beyond 60 s, the control scheme improves the stability to 100 X over the uncontrolled DRG. With this first experimental demonstration of frequency-locking of a CVG to an ultra-stable clock, we have shown that orders of magnitude of stability of a silicon MEMS gyroscope can be gained.

DISCUSSION

Equation (1) is valid when the CVG is a symmetric structure operated in a mode-matched condition. The frequency split in Equation (1) results from the inability to perfectly control the frequency in each control loop (drive mode and sense mode) of the gyroscope. The limit of frequency split control is determined by the fractional frequency stability of each control loop. Since the frequency noise in each control loop is assumed to be uncorrelated, the factor of $\sqrt{2}$ accounts for drift in the frequency of each mode relative to the other in the total frequency split.

In the original Lynch model [9], there is a factor of $\sin(2\theta_{\tau})$ which should be considered in Equation (1), where θ_{τ} represents the angle between the transducer axes and the principal modal damping axes. However, in a highly symmetric structure, such as the DRG, the damping is distributed around the circumferential direction of the structure and it is not clear that there is a preferred set of principal damping axes. Therefore, given the above and the inability to predict a value of θ_{τ} for a given DRG device in practice, we took the worst case situation by letting $\sin(2\theta_{\tau}) = 1$ in our model. It remains to be seen if some DRGs have smaller sensitivity to this source of bias drift than others, at which point a conclusion can be made as to whether a set of preferred damping axes exist in the DRG structure.

The novel control loop in Figure 8a should be implemented around both the CVG drive mode and sense mode. This experiment is in progress to show the ultimate goal of improving the output rate stability of the DRG. Also, further tuning of the loop parameters is needed for optimal performance. However, this concept of using a high stability frequency source to stabilize the control loop of a sensor applies to other types of resonant MEMS sensors beyond CVGs. For example, resonant magnetometers, accelerometers, or gravimetric sensors could all improve their ultimate sensitivity by stabilizing the sensitive device to a higher intrinsic stability frequency reference.



Figure 8: First demonstration of frequency-locking CVG control scheme showing enhanced frequency stability (> 100 X @ 1000 s) of HRL's silicon DRG when locked to an OCXO frequency reference. (a) Block diagram of control scheme used to stabilize HRL's 600 μ m Stanford-fabricated epi-seal encapsulated silicon DRG. The light gray box with dashed outline indicates the traditional MEMS CVG control loop, while the outlined beige box indicates the new control loop added to stabilize the control loop frequency. Operational parameters are: $V_{bias} = 25 V$, $V_D =$ 20 m V_{ao} and $f_{n=3} = 301.8$ kHz (b) Measured frequency stability shows orders of magnitude improvement with stabilized control loop operation (PID-on).

CONCLUSION

The demonstrated frequency-locking concept significantly reduces microfabrication and environmental sensitivity hurdles that have limited the adoption of MEMS gyroscopes in high-precision applications. We anticipate that this technology will enable MEMS sensors to progress significantly beyond their current levels of performance.

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THIN FLEXIBLE AND STRETCHABLE TACTILE SENSOR BASED ON A DEFORMABLE MICROWAVE TRANSMISSION LINE

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ABSTRACT

This paper presents a MEMS tactile pressure sensor based on distributed microwave sensing. Rather than using arrays of capacitive or resistive pressure sensors as is currently the state of the art, the sensor reported here determines the degree and location of applied pressure by measuring reflections on a thin embedded microwave transmission line. This approach allows for a rugged wide-area sensor that is easily and inexpensively fabricated and which requires only a single two-conductor connection to external electronics. The sensor is both flexible and stretchable, a rare combination of features among other tactile sensors.

INTRODUCTION

Current state of the art in tactile sensors

Over the past decade there have been numerous publications on tactile sensors and skins aimed at replicating the human sense of touch in applications such as robotics, healthcare, and prosthetics [1][2][3]. A variety of sensing approaches are used [1], with the dominant ones being piezoresistive [4] and capacitive [5][6]. The trend has been toward increasingly complex, thin, and sophisticated skins. While impressive resolutions, sensitivities, and degrees of integration have been achieved, mechanical durability and inexpensive manufacturing, properties essential for widespread use of tactile skins, have not advanced proportionately.

All current tactile sensing skins use arrays of discrete sensors. Forming X-Y arrays of devices, which is the standard technique in microelectronics, has the advantages of density and performance. However, in the unique application of tactile skins, it has serious drawbacks as well. Arrays, by definition, require a minimum of $2\sqrt{N}$ wires for N sensors if each sensor requires only two wires and can be addressed by row and column. While not a serious problem in rigid electronics, in flexible and stretchable devices making the interconnections and reliably connecting rows and columns to external electronics present serious challenges. Furthermore, arrays are intrinsically fragile as damaging a single device can disable an entire row and/or column of sensors.

Other methods have been proposed to address the interconnect challenge, most notably [7], which demonstrates the use of flexible embedded silicon "nanomembranes" containing traditional silicon electronics that could perform decoding or even wireless data transfer functions. However, the need still exists for simple inexpensive sensing skins that do not require advanced electronics, which would be expensive except at a huge scale.

Microwave sensing concept

The sensing approach presented here addresses the arrayinterconnect challenge by eliminating the array. Conceptually, the technology works by applying a high frequency pulse to a transmission line with an impedance discontinuity due to a depression of the dielectric caused by applied pressure. This discontinuity causes a portion of the pulse to be reflected back to the source. Through time domain reflectometry (TDR), the roundtrip time-of-flight of the pulse and its amplitude can be used to determine the location and magnitude of the depression and hence the applied pressure; see Figure 1. Thus, the transmission line becomes a two-terminal distributed sensor that can be snaked over a two-dimensional area.



Figure 1: Concept for microwave transmission line sensing. In this conceptual drawing the red pulse from the generator is partially reflected off of the impedance discontinuity in the line caused by pressure (black arrow).

TDR is a standard technique for finding faults in cables, for example. While it works, TDR presents a few problems. First, if more than one discontinuity is present, the pulse can re-reflect between them causing "ghosts" in the response, which cannot be easily distinguished from genuine responses. Furthermore, a practical distributed pressure sensor will be exposed to continuous gradients of pressure, not discrete points, breaking the TDR analogy completely. For these reasons, the sensor is not excited here with a single pulse. Rather, its complex impedance is measured with a vector network analyzer (VNA) at 201 frequencies from about 30 MHz to about 6 GHz. This data is then processed as described below to reconstruct the applied pressure.

THEORY

Reconstructing the deformation of the transmission line from measurements of its terminal impedance first requires understanding how the resulting line depressions perturb waves propagating along the line. To gain this understanding, the traditional Telegrapher's Equations are modified to be make the inductance per unit length L(x) and the capacitance per unit length C(x) functions of the position x along the line. Thus,

$$\frac{\partial V(x,t)}{\partial x} = -L(x)\frac{\partial I(x,t)}{\partial t} \tag{1}$$

$$\frac{\partial I(x,t)}{\partial x} = -C(x)\frac{\partial V(x,t)}{\partial t}$$
(2)

where t is time, V(x, t) is voltage and I(x, t) is current.

To solve (1) and (2), several assumptions are made. First, assume the perturbation in line impedance is small, that is, the line is compressed less than about 10-20% of its thickness. Second, assume the line to be a lossless and properly terminated ideal parallel plate transmission line having length l and plate gap spacing g(x). With these assumptions, perturbation analysis yields

$$V(x,t) = V^* e^{j\omega(t-\frac{x}{c})} + \frac{V^*}{2G_0} e^{j\omega(t-\frac{x}{c})} \int_x \frac{dg(x')}{dx} e^{-2j\omega\frac{x'}{c}} dx' \quad (3)$$

where G_0 is the nominal conductor spacing with the line at rest, g(x) is the perturbed conductor spacing as a function of position along the line, and V^* is the amplitude of the wave launched at the terminals at x = 0 with phase velocity c and angular frequency ω . In (3), the first term is a forward-traveling wave, and corresponds

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to the 0th-order term in the perturbation series. The second term is a backward-traveling wave that reflects off line depressions, and corresponds to the 1st-order term in the perturbation series. Thus, (1) and (2) are solved to first order in a perturbation parameter that scales $g(x)/G_0 - 1$. For $S_{11}(\omega)$ defined as the ratio of the backward-traveling wave to the forward-traveling wave at x = 0, (3) can be rearranged to yield

$$S_{11}(\omega) = \frac{1}{2G_0} \int_0^l \frac{dg(x')}{dx} e^{-2j\omega \frac{x'}{c}} dx'$$
(4)

where l is the length of the line and all other variables have been previously defined. $S_{11}(\omega)$ will be measured experimentally.

It is now desired to recover g(x) from $S_{11}(\omega)$. To do so, observe that (4) is essentially a Fourier transform that can be inverted. Inversion is carried out numerically using a sampled measurement set. This results in

$$\frac{dg}{dx} = \frac{4N^*G_0f_0}{c} \operatorname{IFFT}[S_{11}(f)]\frac{1}{\alpha}$$
(5)

where $f = 2\pi\omega$ is circular frequency, f_0 is the frequency sample step size, and c is the phase velocity in the transmission line which is slower than the speed of light by the velocity factor of the line. N^* is the number of data points after the $S_{11}(f)$ measurement set has been made Hermitian so as to have a purely real inverse transform. This is accomplished by combining it with its complex conjugate and adding a DC reflection coefficient, which is assumed to be zero. The resulting measurement set has a length of $N^* = 2N + 1$ where N was the number of measured frequency points. The parameter α is defined as $G/Z \cdot dZ/dG$ where Z is the impedance of the line and G is the gap. For an ideal parallel plate transmission line $\alpha = 1$ but due to the effects of fringing fields α is less than 1 for a real transmission line. Using the equations for the impedance of a real strip line transmission line given in [9] and the dimensions of the actual line, a value of $\alpha = 0.6$ is derived.

Integrating (5) yields a reconstruction of the deformation of the transmission line gap as a function of position. Pressure is then calculated using the Young's modulus of the dielectric.

The distance between position data points $\Delta X = c/(2N^*f_0)$ so the resolution of the sensor is limited by the shortest propagating wavelength. Thus, the resolution can be improved by either increasing the excitation frequency or decreasing the propagation speed. The frequency is limited to about 6 GHz by loss and reflection due to manufacturing tolerances, but the wave speed can be decreased by increasing the dielectric constant of the PDMS dielectric. This is achieved, as in [8], by adding a high- κ ceramic material, CCTO, to the PDMS.

FABRICATION

Much attention was paid during design to ensure that the sensors could be manufactured with simple molding techniques that can be easily scaled to arbitrarily large areas, as opposed to with lithographic techniques, which are generally limited by wafer sizes. Care was also taken to ensure insure that the completed sensors remained stretchable and flexible.

First, Polydimethylsiloxane (PDMS, Sylgard® 184) is mixed with 10% linker and optionally CCTO powder in a centrifugal mixer (Kurabo Mazerustar KK-250S). Next a piece of stretchable silver cloth, Statex Shieldex® MedTex[™] P-130 is cut to be slightly larger than the finished line, soaked in pure PDMS and



Figure 2: Fabrication diagram for transmission line pressure sensor. a) The silver cloth for the ground plane is soaked in PDMS, mounted in the mold and cured. b) The mold is filled with PDMS (optionally doped with CCTO), the cover is attached, and the PDMS is cured. c) The silver cloth transmission line soaked in PDMS and protected with Kapton® tape is installed and cured. d) The line is removed from the mold, the tape is removed, the excess silver cloth ground plane is trimmed and the SMA connectors are installed with conductive silver epoxy.

clamped in the mold. Before soaking, two small pieces of Kapton® tape are used to protect the back of this cloth from the PDMS where the SMA connectors will attach. The mold is then placed in a vacuum chamber until all air is removed. It is then cured in an oven at 120 °C for 20 minutes. With the mold thus prepared, it is filled with PDMS (optionally doped with CCTO powder) to form the dielectric. Again it is degassed; the cover is bolted onto it, and it is again cured. Next, another piece of silver cloth is protected with Kapton® and then precisely cut into a 4.1mm-wide strip, calculated to achieve a resting impedance of 50 Ω . After being soaked in pure PDMS, this strip is adhered to the top of the dielectric, tape side up. The lid is then replaced on the mold and it is cured one final time. Finally, the mold is disassembled, the excess silver cloth ground plane is cut off, and SMA connectors are attached to either end with MG Chemicals MG-8331 conductive silver epoxy. See the fabrication diagram in Figure 2 and the finished sensor attached to a glove in Figure 3.



Figure 3: Completed sensor mounted on a finger. The deformation caused by pressing on the red plastic rod is easily detected.



Figure 4: Device used for applying precise depression to the transmission line.

Testing Procedure

For testing, one end of the line end is connected to Port 1 of an HP 8410C vector network analyzer (VNA) with a flexible phase stable test port extension cable; the other end is terminated by a 50 Ω microwave terminator. To apply a known depression to the line, the apparatus shown in Figure 4 is used. It consists of a micrometer head with a non-rotating spindle that presses a 15 mm wide piece of acrylic onto the line surface. Because the line is not shielded, care must be taken to avoid conductive or magnetic materials in close proximity with the sensor top. This is achieved by protecting the micrometer spindle with acetal.

The network analyzer is connected to a computer running a real-time version of the algorithm described above that allows the effects of depressing the line to be seen immediately. The software can also perform base-line subtraction on the collected data. This is necessary due to imperfections in line fabrication that result in permanent impedance discontinuities that would otherwise appear in the data. The baseline is subtracted after the line is inserted into the test apparatus and after the apparatus has been set to just begin depressing the line. This has the benefit of also subtracting off any effect due to the proximity of the spindle to the line.

To measure the position at which pressure was applied, the line was marked with a ruler and pressure was applied at the marked points. Note that zero position refers to the calibration plane of the vector network analyzer which is about 9 mm from the beginning of the active portion of the sensor.

RESULTS AND DISCUSSION

Position and Accuracy

The sensor is very accurate in position, as position accuracy is affected only by the velocity factor of the line and the frequency accuracy of the VNA. Figure 5 shows the response of the line to depressions made at 40 mm, 100 mm and 160 mm beyond the calibration plane of the VNA. In each case the response is accurate to within the spacing of one data point (7.3 mm for pure PDMS at 6 GHz). In this reconstruction a velocity factor of 0.585 was used, which matches the value of 0.584 found by directly measuring the propagation delay of the line with the VNA. Thus, the reconstruction appears to be quite accurate with regard to position.



Figure 5: Plot showing depression response of transmission line sensor for three different pressure locations. The uncertainty trace represents 1 standard deviation.

Depression Accuracy

As seen in Figure 5, the depths of the depressions applied 40 mm from the start of the line are reconstructed to within 30% of their actual values. The discrepancy is believed to result from inaccuracy in the α parameter, which was calculated assuming a lossless transmission line. Indeed, experiments with a low-loss line made with copper foil conductors show agreement with theory at 40 mm to within 6%. The response drops off more at positions farther away from the start of the line. This is primarily due to loss in the transmission line; from end to end it has a total resistance of 15 Ω due to the resistance of the silver cloth. In future devices this will be addressed with improved reconstruction algorithms and/or more highly conductive stretchable materials.

A second effect seen in the data is that the depression settles to a value less than zero for positions along the line beyond the deformed region. This effect has been demonstrated through experiment and simulation to be due primarily to the resistance of the cloth decreasing locally where pressure is applied.

A third source of error is observed for narrowly deflected regions, narrower than a wavelength, for which the response is dramatically decreased. Experiments with simulated data that is otherwise free from error show that the response to depressions having a 10-mm and 4-mm width drops to 80% and 33% of normal, respectively. As a corollary, no conclusions can be drawn from features in a deformation plot that are smaller than about 15 mm due to the limited resolution.

Finally, note that the experimental uncertainty in Figure 5 increases with position along the line due to cumulative error in the integral used to reconstruct the depression. See Equation (4).

Resolution and effect of added CCTO and Frequency

Separate from the position accuracy discussed above, the sensor resolution must also be considered. The resolution is defined here as the distance between two discrete depressions at which they can no longer be distinguished from a single depression. To measure resolution, the sensor was depressed at two points with a constant force (from a weight) and these points were gradually brought together until the response from the sensor showed just one depression. At this point the distance between the points was measured with a ruler and recorded as the resolution.

The resolution (and the distance between individual position data points) of the sensor is limited by the size of the shortest



Figure 6: Plot showing resolution measurement. The measured resolution is 12 mm since that is the spacing at which the peaks converge. Curves have been staggered by 50 μ m for clarity.

wavelength used to excite it. Wavelength can be decreased by increasing the frequency, but the usefulness of increasing frequency is limited by the high frequency loss in the line. Wavelength can also be decreased by decreasing the velocity factor of the line which, per [8] can be achieved by adding high- κ ceramic particles, such as CCTO to the PDMS. Table 1 lists the resolutions and velocity factors for sensors made with five different concentrations of CCTO. From the velocity factor, the wavelength at 6 GHz was also computed. From this data two conclusions can be reached. First, the resolution of the sensor can indeed be increased by adding CCTO to the PDMS. The effect is modest though, at only about 20%, a result consistent with that reported in [8]. Second, the minimum discernable resolution is consistently approximately half a wavelength and tracks with velocity factor demonstrating that the resolution limitations of the sensor are due to the wavelength of the propagating wave.

Table 1: Resolution as a function of CCTO concent	ration
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ССТО	Resolution	Velocity	Resolution /
Concentration (%)	(mm)	Factor	Wavelength
0.00	12±1	0.584±0.003	$0.411 \pm .036$
6.31	12±1	0.528±0.003	$0.455 \pm .041$
10.2	12±1	0.476±0.003	$0.505 \pm .046$
15.4	11±1	0.456±0.003	$0.482 \pm .047$
20.3	9±1	0.403 ± 0.003	$0.447 \pm .053$

Determination of applied force from depression

Due to the geometric complexity of deforming a line with an arbitrarily shaped object, the relationship between deformation and applied pressure is complex and beyond the scope of this paper. However, assuming the general shape of the line to be an elastic sheet, and that this geometry results in the PDMS behaving as a locally ideal spring, and further assuming that the Young's modulus of the sensor is equal to that of pure PDMS, ~500 kPa, (the material becomes harder with added CCTO), the 10 kPa pressure sensitivity of human skin [2] would result in a 37 μ m depression, which could easily be detected by the sensor. Thus, the device has a sensitivity approaching that of human skin.

Other Considerations

No attempt was made to measure the time response of the

sensor because the slow VNA sweep rate dominates. However, it should be limited only by the mechanical response of the PDMS.

Due to aliasing, impedance measurement at sampled frequencies results in a limited maximum length of line that can be measured. However, for the \sim 30 MHz step size used here this length is nearly 3 m. This effect is therefore not of practical concern.

At present, the major limiting factor to the practical application of this technology is the size and cost of the VNA required to measure the impedance data. However, portable VNAs exist; the Agilent FieldFox for example weighs only 3 kg and measures only 292 x 188 x 72 mm. With the highly sophisticated RF electronics in cell phones it seems likely that wrist- or pocket-sized custom electronics could be developed for this application.

SUMMARY AND CONCLUSIONS

While still in an early stage of development, the distributed microwave pressure sensor for tactile skins shows much promise. The resulting sensors are very simple to manufacture, flexible, stretchable, and quite durable. The sensors achieve a depression accuracy of 30% and a position accuracy of 7.3 mm for the pure PDMS sensor. The resolution and position accuracy can be increased by adding CCTO to the PDMS. The overall pressure sensitivity of the device approaches that of human skin.

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THERMALLY ISOLATED MICROMACHINED QUARTZ RESONATOR ARRAY FOR DIFFERENTIAL MICRO-CALORIMETRIC MEASUREMENTS

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INTRODUCTION

We have reported on the concept and performance of a calorimetric biosensor using a micromachined Y-cut quartz resonator as sensitive thermometer and a physically decoupled but closely located microfluidic reaction chamber[1]. Using layer by layer urease enzyme immobilization and combining with microfluidic pumping of the analyte we were able to demonstrate continuous measurements of urea with resolution of $\sim 2 \text{ mM}$ [2]. Micromachined bulk acoustic wave resonators with frequencies in few hundred MHz allow for temperature resolution of tens of microKelvin and better SNR as compared to thin film thermistors and thermocouples [1]. Proximity coupling of the thermal energy via conduction through thin film of air and radiation from the reaction chamber to the resonator thermometer, allows for complete separation of the biology and the physical sensor/electronics. In this paper we report on over 18 times improvement of this calorimetric sensor design achieved through improved thermal isolation of the resonators and differential sensor configuration to remove common mode temperature artifacts.

RESULTS AND DISCUSSION

8-resonator arrays of monolithic, micromachined quartz resonators of thickness 10 µm (167 MHz) were fabricated using RIE etching of quartz [3] as schematically shown in Fig. 1. The inverted mesa design of the resonators resulted in large thermal coupling and cross-talk between the resonators. Focused ion beam etching was used to create a thermally isolated resonator pixel (Fig. 1). The thermally isolated resonator was used as the reference sensor which provides a sensitive reference for background temperature changes as well as cancels out any spurious noise. For these measurements a 1 mm diameter Kapton® tubing with wall thickness of 25 µm was used. A nichrome wire of 4 Ω resistance was wound and placed upstream in the tube and the temperature of the water downstream from the heater was measured by inserting a thermocouple into the tube. Two such Kapton[®] tubes (channels) are placed above the resonator chip (Fig. 2). Although only channel 1 was exposed to heated water (6.05 mW for 20 s), in the case where the resonator is not thermally isolated, heat is conducted through the quartz substrate and results in the temperature of both resonators to rise and results in poor compensation in the differential mode (Fig. 3(a)). For the thermally isolated resonator chip, when only channel 1 was exposed to heat (450 µW, 20 s), excellent compensation in the differential mode is achieved (Fig. 3(b)). Similarly, when a common mode thermal signal (3.0 mW, 20 s) is applied simultaneously to both channels, excellent compensation is achieved in the thermally isolated sensor chip (Fig. 3(c)). In this case the reference sensor effectively cancels both thermal drift as well as the large ~175 Hz common mode response. Allan deviation noise characteristics of similar

frequency Y-cut and AT-cut resonators are compared in Fig. 4. The noise in Y-cut resonator is 10 times larger mainly due to radiatively and conductively induced thermal response. Fig. 5 shows the thermal sensitivity of various Y-cut resonators, showing 2.25 times higher sensitivity for the 165 MHz resonator in comparison to a 68 MHz resonator. Furthermore, the 167 MHz thinner and thermally isolated resonator with IR reflective gold electrode has 1.76 times lower noise than 68 MHz Y-cut resonator thereby improving the overall SNR by 4.73 times. Combining the improved differential measurement achieved by the thermally isolated sensor with the improved SNR of the 167 MHz resonator, an improved resolution of 110 uW from the previous limit of 2 mW measured for a non-isolated 90 MHz resonator is realized. The improved resonator design is expected to enable quantification of urea down to 275 µM where previously only 5 mM was possible.







Figure 2: Schematic of the dual channel calorimetric sensing system.

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Figure 3: Differential measurement based compensation: (a) Resonator array (without thermal isolation). Heated solution in channel 1 and channel 2 acts as a reference. (b) Resonator array with thermally isolated resonator under channel 2 acts as a reference. Heated solution in channel 1 (c) Common mode thermal signal compensation using the thermally isolated resonator array.



Figure 4: Allan Deviation of Y-cut resonators at 68 MHz (red), a 70 MHz AT cut quartz resonator (blue), a 165 MHz resonator (black) and an isolated 165 MHz resonator (green).



Figure 5: The sensitivity of a non-isolated 68 MHz resonator (red) and 167 MHz resonator (black) are compared with that of a thermally isolated 165 MHz resonator (blue).

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MICROFLUIDIC LABEL-FREE IMMUNOCHIP FOR EARLY DIAGNOSTICS OF BREAST CANCER USING FUNCTIONALIZED POROUS GRAPHENE

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ABSTRACT

We demonstrate a label-free microfluidic immunosensor for early diagnostics of breast cancer (epidermal growth factor receptor or ErbB2) with a detection limit at the level of 1.0 fM. This device utilizes a uniquely structured immunoelectrode made of porous graphene foam (GF) modified by titanium dioxide nanofibers (nTiO₂) at its surface. The antibody of ErbB2 acts as a detection probe immobilized on the GF-nTiO₂ electrode *via* EDC-NHS chemistry. This immunosensor provides a high sensitivity of 43.7 k Ω/μ M in a wide detection range, good stability, and fast response (3 min) for detection of ErbB2 antigen.

INTRODUCTION

The ability to detect breast cancer biomarkers at an early stage is urgently required for clinical diagnostics and treatment for patient survival. Human epidermal growth factor receptor (EGFR, ErbB2, or HER2) gene encodes a 185-kDa transmembrane glycoprotein and a receptor tyrosine kinase with intrinsic tyrosine kinase activity. Overexpression of ErbB2 (~33%) is linked to increasing breast cancer metastasis [1]. X-ray mammography, magnetic resonance imaging, computed tomography, and ultrasound imaging are commonly used methods of detecting cancerous tumors. Nevertheless, these tools rely heavily on imaging to determine the tumor status, providing insufficient information on the onset of cancer or monitoring of early stage cancerous cells.

Recently, microfluidics-based immunosensors, in conjunction with sensitive nanomaterials and nanostructures, have shown a great potential to revolutionize early detection of cancerous cells and therapy practices, offering cost-effectiveness, high sensitivity and selectivity, and portability. In particular, the two dimensional material, graphene, has been utilized to realize immunosensors capable of quantifying breast cancer biomarkers with a detection limit of 1.0 pM [2].

Here we report a microfluidic electrochemical immunosensor with a uniquely structured bioelectrode made of porous graphene foam (GF) modified by titanium dioxide nanofibers (nTiO₂). Anti-ErbB2 antibody is conjugated at the surface of GF-nTiO₂ electrode to detect breast cancer biomarker ErbB2 via antigen-antibody interactions. The electrochemical impedance spectroscopy (EIS) is employed to monitor changes in interfacial properties of the bioelectrode (e.g., charge transfer resistance) induced by specific protein adsorption on the electrode surface.

DEVICE FABRICATION

Fig. 1a depicts the structure of the proposed microfluidic immunosensor. The device has a T-shaped, GF-nTiO₂-based working electrode functionalized with anti-ErbB2 and attached to the upper side of a microfluidic channel through a pre-punched hole. A gold (Au) counter electrode and an Ag/AgCl reference electrode are formed on a glass slide (Fig. 1b). The immunoelectrode is placed above and in parallel with the counter electrode with a gap distance of about 500 μ m. Biological functionalization of anti-ErbB2 on the surface of the GF-nTiO₂ composite is shown in Fig. 1c. The skeletons of porous GF is covered with electrospun nTiO₂. -COOH groups are generated at

the composite electrode *via* oxygen plasma treatment. This facilitates binding -NH₂ groups of anti-ErbB2 with amide bond (CO-NH) through amidation reaction using EDC-NHS covalent chemistry. The non-specific sites of anti-ErbB2/GF-nTiO₂ are blocked by treating bovine serum albumin (BSA) molecules.



Figure 1: (a) Schematic (cross-sectional view) for a microfluidic electrochemical immunosensor for detection of breast cancer biomarker using a porous GF modified by TiO_2 nanofibers. (b) Photo of the microfabricated immunosensor. (c) Pictorial representation of the proposed porous immunoelectrode, where anti-ErbB2 are attached to $nTiO_2$ enabled GF. The SEM images shows the hierarchical structure of GF covered by $nTiO_2$.

RESULTS AND DISCUSSION

Fig. 2 shows the Nyquist plots obtained by EIS for different electrodes, including GF, anti-ErbB2/GF, GF-nTiO2, and anti-ErbB2/GF-nTiO2 before and after being modified with BSA molecules. Measurements are conducted in presence of PBS buffer (pH: 7.4) containing 5 mM [Fe(CN)₆]^{3-/4-} as a redox probe. The charge transfer resistance (Rct) values of these electrodes are found based on the diameter of semicircle of the Nyquist plots. The GF electrode with antibody shows a higher R_{ct} (45.3 k Ω) compared to that without antibody (29.8 k Ω). The modification of GF electrode with nTiO₂ increases R_{ct} to 87.7 k Ω compared to the GF electrode. It should be noted that the larger surface area of the GF provides an easy access of nTiO₂ suspension into its interior scaffolds, resulting in an enhanced loading capacity of anti-ErbB2 and thus a high readout signal. After immobilized with anti-ErbB2 and BSA, the immunoelectrode has an increased R_{ct} of 152.6 k Ω , due to the insulating nature of the BSA proteins that hinder redox conversion. Moreover, the anti-ErbB2 and BSA molecules immobilized on the GF-nTiO₂ electrode lead to a higher surface coverage (80.7%) compared to the bare GF-nTiO₂ electrode (65.6%). This may help enhance sensitivity and detection limit of the sensor.

The EIS technique is utilized for detection of ErbB2 by varying its concentration from 1 fM to 0.1 μ M. Fig. 3 shows that the value of R_{et} increases with increasing concentration of ErbB2.

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Electrodes	Techniques	Biomarkers	Sensitivity (µM ⁻¹)	Range (µM)	Ref.
ZnO nanofibers	Impedance	ErbB2	7.76 kΩ	1×10 ⁻⁹ -0.5	[1]
Graphene oxide-SiO ₂ nanoparticles	Conductance	HER2	NA	1×10 ⁻⁶ -1.0	[2]
Carbon nanotubes	Cyclic voltammetry	Anti-breast cancer agent	8-925 nA	40–160	[3]
ZnO nanowires	Differential pulse voltammetry	BRCA1	6.36 μΑ	10–100	[4]
GF-nTiO ₂	Impedance	ErbB2	43.7 kΩ	1×10 ⁻⁹ -0.1	This work

Table 1: Comparison for sensing performances of our sensor with other electrochemical sensors reported in literature

Essentially, the interaction between specific binding sites of anti-ErbB2 and ErbB2 on the sensor surface results in the formation of immunocomplex that obstructs transfer of electrons generated from redox conversion. The sensor demonstrates a high sensitivity of 43.7 k Ω/μ M with a low detection limit of 1.0 fM for the breast cancer biomarker. Also, the association constant of the immunoeletrode is as high as 4.46 kM⁻¹s⁻¹, indicating a high affinity towards ErbB2 antigen.



Figure 2: EIS spectra for five electrodes, including GF, anti-ErbB2/GF, GF-nTiO2, and anti-ErbB2/GF-nTiO2 before and after modified with BSA molecules. Inset shows an equivalent circuit model for the Nyquist plot to obtain the value of R_{ct} .



Figure 3: (a) EIS spectra of the sensor for detecting breast cancer biomarker (ErbB2) as the concentration of ErB2 is varied from 1.0 fM to 0.1 μ M. (b) Calibration plot for charge transfer resistance (R_{ct}) as a function of concentration of ErbB2.

Fig. 4a demonstrates the selectivity of the sensor in presence of human IgG, cholesterol, and BSA molecules as is evident by its low relative standard deviation (RSD: $\pm 2.0\%$). Fig. 4b demonstrates the stability of the sensor (RSD: $\pm 1.0\%$) within 35 days. A minor deviation of $\pm 4.5\%$ in impedance changes is observed for four identical sensors, suggesting reasonable reproducibility. The response time for this sensor is about 180 sec (inset of Fig. 4b).

Table 1 compares the immunosensing performance of the sensor with its comparison with other electrochemical sensors

reported in literature [1-4]. Due to different mechanisms and different biomarkers, point-to-point comparisons are difficult. However, with the same antibody ErbB2, our device shows a sensitivity (43.7 k Ω/μ M) about six times of magnitude higher than that using ZnO nanofibers-based immunoelectrode (7.76 k Ω/μ M).



Figure 4: (a) EIS curves to investigate for interference studies of the fabricated immunosensor using different interferents (BSA, IgG, and cholesterol) in presence of 100 nM, ErbB2, inset showing the R_{ct} vs interferents. (b) Response obtained for stability test of the sensor over 35 days. Inset shows the plot of dynamic change in impedance as time.

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TOWARDS PATHOGEN DETECTION VIA OPTICAL INTERROGATION OF MAGNETIC MICRODISCS

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ABSTRACT

We present progress in the development of a micro/nanobased scheme for portable, rapid, low-cost detection of foodborne bacterial pathogens, such as *Escherichia coli*, *Listeria monocytogenes*, or *Salmonella*. The system uses magnetic microdiscs that are functionalized for selective binding to target pathogens. The presence of a target pathogen is evaluated by optically monitoring the rotation of the microdiscs when subjected to a rotating magnetic field. This article summarizes the following advancements: (1) wafer-level microfabrication of 600 million gold-coated microdiscs per substrate; (2) functionalization of the microdiscs with DNA aptamers and subsequent binding to *E. coli* O157:H7; and (3) magnetic manipulation of the bound bacteria.

INTRODUCTION

Outbreaks of foodborne pathogens are becoming increasingly frequent and represent a growing threat to public health [1]. The existing approaches for pathogen screening rely on highly skilled personnel, expensive laboratory equipment, and long time frames (e.g. 8–24 hr for DNA amplification and PCR detection) [2].

The proposed system involves optical measurements of the rotational dynamics of suspended magnetic microdiscs functionalized with pathogen-binding aptamers. The magnetic microdiscs (1.5 µm in diameter) have low-aspect-ratio and are fabricated using a soft ferromagnetic material: 70 nm permalloy $(Ni_{80}Fe_{20})$ with a 5 nm surface coating of gold (Au). These microdiscs exhibit a closed magnetic spin arrangement (spin vortex) with zero magnetic stray field. This behavior is critical to avoid disc agglomeration when in free suspension [3] (i.e. fluidic samples). With very high surface area for functionalization and volumes 10,000x larger than commonly used superparamagnetic nanoparticles, these micro-sized microdiscs are well suited for tagging, trapping, actuating, or interrogating bacterial pathogens, which have comparable sizes to bacterial pathogens in the range of $0.3 - 1 \mu m$ [4]. These magnetic microdiscs tend to align their in-plane magnetic easy-axis to an external rotating magnetic field, $\overline{B_{ext}}$, hence they can be actuated synchronously to act as an "optical shutter" [5] (Fig. 1).

The experimental setup proposed in this work takes advantage of the synchronous behavior between the rotating microdiscs and the rotating magnetic field ("optical shuttering"). Here, a combination of magnetic actuation and optical detection is performed using two orthogonal magnetic coils, a laser, and a photodiode to control and monitor the light transmission modulation, or light intensity, of the laser (Fig. 1). A phase-lag exists between the rotating magnetic field and the disc orientation, which depends on the magnetic field amplitude, rotation frequency, fluid viscosity, and disc hydrodynamic diameter (Fig. 2).

Previous work has shown how this apparatus is sensitive to its environment and can be used to measure fluid viscosity [5]. Therefore, this approach of monitoring the light transmission modulation will be adapted for pathogen detection applications by characterizing the light intensity sensed by the photodiode for different control and target bacteria concentrations when exposed to the biofunctionalized suspended microdiscs. For pathogenic detection, the goal is to alter the hydrodynamic diameter via specific binding with target bacteria, which will dramatically change the rotational dynamics.



Figure 1: "Optical shutter" behavior of microdiscs actuated by the rotating magnetic field (left) and proposed pathogen detector apparatus (right). (Reproduced from [5] with permission of AIP Publishing)



Figure 2: Torques acting on the microdiscs (top) and light intensity measurement example (bottom). (Reproduced from [5] with permission of AIP Publishing)

RESULTS

Microfabrication process

Dense arrays of magnetic microdiscs are microfabricated on 100 mm Si substrates, yielding 600 million microdiscs (0.86 mg) per wafer using standard microfabrication techniques, such as photolithography, thin metal deposition, and lift-off. Here, a hexagonal array of circular polymer pillars (1.5 μ m in diameter; 3.0 μ m minimum center-to-center spacing) is patterned using a positive photoresist in an area of ~20 cm² of the wafer. In order to obtain higher resolution and re-entrant sidewalls for the photoresist pillars, an image reversal recipe is utilized before sputtering. Next, a metal stack of Au (5 nm)/Ni₈₀Fe₂₀ (70 nm)/Au (5 nm) is sputtered. Finally, the magnetic microdiscs are lifted off from the substrate using ultrasonic agitation with acetone and triple-washed in DI water (Fig.3).

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Figure 3: (left) Microfabrication process: (a) photoresist pillars, (b) metal deposition, (c) lift-off; and (right) SEM image of dense hexagonal array of microdiscs on photoresist pillars.

Disc functionalization

After the disc microfabrication, these are functionalized for selective binding to a biological target (Fig.4). Here, we use Eschericia coli (E. coli) O157:H7, a common food-borne bacterial pathogen, as a demonstration pathogen. The gold surfaces of the microdiscs are functionalized with a DNA aptamer (21 mer), which functions as a capture probe. The aptamer discriminates E. coli O157:H7 from other gram-negative bacteria by forming a covalent bond with cell surface O-antigen structures (binding with O-antigen occurs at the 5' end of the aptamer). The DNA contains a thiol group and a six-carbon spacer at the 3' terminal end, which ensures adequate mobility for bacteria binding as demonstrated in [6]. The aptamers are in a dithiotriotol HEPES buffer at 20 nM concentration and pH of 7.1. Here, 10 µL of the DNA aptamer solution is added to an 8 mL solution of discs (12.5 million discs per mL). The aptamers adsorb to the discs via gold-thiol binding. Bacteria binding and magnetic manipulation

After functionalization, the microdiscs are exposed to GFPexpressing *E. coli* O157:H7. The bacteria concentration was only 1 CFU/mL, which is at the lower limit of detectability for other biosensors. Here, 10 μ L of the bacteria suspension was added to the 8 mL solution of functionalized discs. In this experiment, the concentration of magnetic microdiscs was much higher than *E. coli* in order to maximize capture of target cells. After 10 min incubation, the discs were concentrated using an external magnet (Fig. 5), and 1 μ L was pipetted and then deposited on a coverslip. Figure 6 shows representative images of the *E. coli* successfully bound to the aptamer-functionalized discs. Here the dark areas are clusters of discs (magnetically agglomerated in this case), and the bright green spots are the GFP *E. coli*.

OUTLOOK

With the ability to synthesize large batches of biofunctionalized magnetic microdiscs, ongoing efforts are focused on optimizing the concentration of discs and also detection of bacteria via the optical interrogation of magnetic microdiscs under rotation.



Figure 4: Schematic of binding process: (a) gold coated magnetic microdisc, (b) biofunctionalized microdisc with DNA aptamer capture probe, and (c) bound cell (E. coli).



Figure 5: Microdiscs trapping and concentration using permanent magnet before fluorescent observation under microscope.



Figure 6: Fluorescent/optical image overlay of magnetically concentrated magnetic microdiscs (dark areas) bound to GFP E. coli O157:H7 (bright fluorescent green spots).

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ULTRA LOW COST BLOOD DIAGNOSTIC CHIP FOR POINT-OF-CARE CLINICAL DETECTION

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ABSTRACT

The current requirement for expensive laboratory equipment and highly trained personnel highlight the critical need for simple, low cost technology capable of point-of-care (POC) clinical detection, especially in resource-limited regions. The primary goal of this work is to develop an innovative, ultra-low cost, plasticbased blood diagnostic platform for POC detection that require small volumes (< 3 μ L) of whole blood. The novelty of our approach lies in the finger-powered asymmetric capillary force driven by the spray-coated superhydrophilic plastic plate that is integrated with adhesive tape. We simply quantified the biosensing capability of the blood diagnostic chip using a finger-powered, capillary-driven colorimetric assay.

INTRODUCTION

Blood tests are integral in global health applications (such as infectious diseases) due to the massive amount of critical information it contains that relates to how the whole body is functioning [1]. The need for only a small volume of blood in blood tests is particularly useful for POC detection from pediatric/neonatal patients in resource-limited and geographically isolated regions, including developing countries. Typical methods and most of the reported lab-on-a-chip (LOC) devices [2, 3] have several restrictions that include the need for an external power source, bulkiness, expense, and on-chip platform that lack sufficient integration. Therefore, it is very desirable to develop a simple, low cost technology for POC detection.



Figure 1: Schematic overview of the fabricated ultra-low cost blood diagnostic chip for point-of-care clinical detection.

In contrast to the expensive and labor intensive diagnostic approaches currently being used, our work is unconventional. Anyone can easily fabricate the plastic-based molecular diagnostic chip utilizing just four items: a coated polyethylene terephthalate (PET) plastic plate, regular adhesive tape, tweezers, and scissors (Figure 1). Utilizing a coated plastic plate and a straightforward fabrication procedure, we developed a simple, yet ultra-low cost blood diagnostic chip for the POC detection.



Figure 2: (A) Generation of asymmetric capillary force for blood plasma separation using a layer-by-layer nanoassembly spraying method; (B) difference of the static contact angle between hydrophobic adhesive tape and superhydrophilic PET.

WORKING PRINCIPLE AND RESULTS

At the center of this technology is a spray-coated superhydrophilic [4] PET plastic plate, shown in Figure 2. PET plates are designed for lateral transportation of whole blood by capillary pumping, asymmetric capillary-based blood plasma separation [5], and capillary-driven lateral flow colorimetric assay [6]. Assembled surface-modified PET plates, with adhesive tape, facilitate controlled blood plasma separation within seconds by finger-powered asymmetric capillary force (Figure 2A). The notable difference between the two selected surfaces for asymmetric capillary force is highlighted in Figure 2B.

The scientific principle underlying this proposed concept relates to the finger-powered, capillary-driven lateral flow colorimetric assay on the spray-modified nanoporous PET surface

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(Figure 3). The tetrabromophenol blue (TBPB) solution shown is simply injected onto the nanoporous surface. Once the reaction begins, the phenol in TBPB deprotonates, and the color of the dye shifts to blue which indicates the presence of a spiked protein, bovine serum albumin (BSA), in the separated blood plasma. The coated nanoporous PET surface offers improved binding capabilities and enhanced transduction due to the high surface-tovolume ratio.

The intensity of the color change was quantified by a calibration curve in terms of the protein concentrations (Figure 4B). The regression equation is: *Intensity* = 6.6112ln(x) + 62.789 ($R^2 = 0.9998$), where x is the concentration of protein (in mg/mL). The assay was calibrated with blood plasma samples separated in a device with known protein concentrations. The limit of detection was 25μ g/mL, which is better compared to typical methods as well as paper-based microfluidic device [7, 8].



Figure 3: Finger-powered, capillary-driven colorimetric assay in the blood diagnostic chip. (A) injection of chemicals onto the surface; (B) loading whole blood; (C) blood plasma separation; (D) colorimetric assay.

CONCLUSION

We have developed a novel, ultra-low cost, plastic-based POC blood diagnostic chip, and have further demonstrated its sensing capability using a finger-powered, capillary-driven colorimetric assay. This plastic-based, sample-to-answer, blood diagnostic chip can be mass-produced at low cost and is readily disposable by incineration. By utilizing minute quantities of whole blood and reagents, the developed diagnostic chip also offers great potential for reducing costs while also facilitating rapid turnaround times, compared with conventional testing. Successful implementation of this device will significantly improve the timely and effective POC detection and management of patients, especially pediatric/ neonatal patients in resource-limited and geographically isolated countries.



Figure 4: Procedure for quantification of protein concentration in human whole blood samples using the ultra-low cost blood diagnostic chip. The limit of detection was $25 \mu g/mL$.

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A BIO-INSPIRED REFERENCE ELECTRODE: REGULATING THE RESPIRATION OF MICRO-ORGANISMS

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ABSTRACT

Inspired by the possibility of controlling the respiration of micro-organisms, we present a bio-inspired reference electrode, demonstrating a stable potential for extended period of time. The bio-inspired reference electrode, made of platinum, sits in a microfluidic chamber for growing specific micro-organisms, Geobacter sulfurreducens, which is an exoelectrogen capable of transferring electrons outside its outer membrane to complete its respiration process. Open circuiting the reference electrode forces exoelectrogens on the electrode unable to transfer electrons to the electrode to complete the respiration process. This will set the reference potential to be the open circuit potential of the anolyte, sodium acetate, which is \sim -0.5 V versus the Ag/AgCl in 3M NaCl. The bio-inspired reference electrode maintained its potential stability of ± 1.07 mV/day for over a week. The bio-inspired reference electrode was integrated in a MEMS microbial fuel cell (MFC) to characterize its electrochemical characteristics, including polarization, voltammetry, and electrochemical impedance spectroscopy.

INTRODUCTION

Bio-inspired devices and systems have been an active area of research, including bio-inspired digital cameras [1], bio-inspired artificial lungs [2], bio-inspired camouflage and display for soft machines [3] and bio-inspired supercapacitors [4], which reported not only novel technological advancements but also significant performance enhancement.

MEMS and lab-on-a-chip have been on active research through successful miniaturization of sensors, actuators, and systems [5, 6]. Reference electrodes, which offer maintaining a stable potential, have been widely used in electrochemistry, such as fuel cells and electrochemical sensors [7, 8]. As conventional references, i.e., Ag/AgCl, are too bulky to be integrated in MEMS and lab-on-a-chip setups, researchers have explored miniaturizing reference electrodes in the past. Despite the extensive studies, a microfabricated reference electrode in MEMS lab-on-a-chip has been a long sought challenge to date, primarily due to the poor long-term stability. For instance, Polk et al. electroplated Ag/AgCl thin films to be a reference electrode, yet marked rather poor stability, ±2.88 mV/day for merely 1.4 hours [9]. Another attempt deposited silver chloride nanosheets on silver wire, however, again the reported stability, ±3.2 mV/day for 15 hours, is far from practical use, demanding substantial improvement [10]. Reference electrodes need to provide a stable potential for an extended period, i.e., several days, when being implemented in electrochemical applications such as microbial electrochemical techniques, environmental monitoring, etc..

The aforementioned microfabricated reference electrodes, unfortunately, fall shorts for the demand. The bulky conventional reference electrode not only forces the overall devices / systems to occupy a large footprint, but also often cannot be used in MEMS and lab-on-a-chip. This directly motivates our work: studying a miniaturized reference electrode capable of maintaining its potential for an extended period, over a week.



Figure 1. (a) Optical image of the Pt reference electrode on a glass slide fabricated by one step lift off; (b) Cross-sectional view of (a) across A-A'; (c) Schematic of implementing the reference electrode in a microfluidic chamber (insets: scanning electron microscopy (SEM) image of the Pt reference electrode with Geobacter biofilm formed on top, scale bar: 1 μ m).

MATERIALS AND METHODS

Reference electrode and cathode fabrication

The bio-inspired reference electrode was fabricated by patterning a thin layer of Ti/Pt (20nm/100nm) by lift-off to form an electrode. First, six through holes were drilled on a glass slide (46 mm \times 26 mm \times 1 mm, VWR): one as inlet, one as outlet and the rest four as screw holes. Then the glass slide was cleaned by piranha solution (concentrated H₂SO₄ and 30% H₂O₂, 3:1 volume ratio). Subsequently a layer of photoresist (AZ 4330, 4 µm thick) was patterned on the glass slide, and a layer of Ti/Pt (20/100 nm) was deposited using an electron beam evaporator. The reference electrode was patterned by lifting Ti/Pt off in acetone. The optical microscopy and cross-sectional images of the reference electrode is shown in Figure 1 (a, b). The cathode in this

978-1-940470-02-3/HH2016/\$25©2016TRF DOI 10.31438/trf.hh2016.79 work was made of gold. A layer of Cr/Au (20/200 nm) was deposited using a magnetron sputter on a glass slide (46 mm \times 26 mm \times 1 mm, VWR).

Device assembly

The reference electrode, cathode, proton exchange membrane (PEM, Nafion 117), silicone gaskets (250 μ m thick, Fuel Store Inc.), nanoports (10-32 Coned assembly, IDEX Health & Science), bolts and nuts were assembled to build the MEMS device to evaluate the bio-inspired reference electrode for the electrochemical characterization. Nanoports were glued to the inlet and outlet of reference electrode and cathode on two glass slides. Then, two rectangular silicone gaskets were patterned with a hole of 1 cm \times 1 cm at the center, in order to define the anode and cathode chamber volume of 25 μ L. Finally, a rectangular PEM was cut, and PEM and silicone gaskets were sandwiched between the two glass slides having the reference electrode and cathode to complete the device.

Geobacter sulfurreducens enriched biofilm formation

Geobacter sulfurreducens enriched bacterial community, originally from anaerobic-digestion sludge, was used as inoculum to form a biofilm for the bio-inspired reference electrode. The inoculum was obtained from an acetate-fed microbial electrolysis cell (MEC). The anolyte is 25-mM sodium acetate in 100-mM Phosphate Buffer Solution (PBS) with 1,600 mg NaCl, 380 mg NH₄Cl, 5 mg EDTA, 30 mg MgSO4·7H2O, 5 mg MnSO4·H2O, 10 mg NaCl, 1 mg Co(NO₃)₂, 1 mg CaCl₂, 0.001 mg ZnSO₄ \cdot 7H₂O, 0.001 mg ZnSO₄·7H₂O, 0.1 mg CuSO₄·5H₂O, 0.1 mg AlK(SO₄)₂, 0.1 mg H₃BO₃, 0.1 mg Na₂MoO₄·2H₂O, 0.1 mg Na₂SeO₃, 0.1 mg Na₂WO₄·2H₂O, 0.2 mg NiCl₂·6H₂O, and 1 mg FeSO₄·7H₂O (per liter of deionized water) (pH 7.8 \pm 0.2). For start-up process, inoculum and anolyte were mixed with 1:1 volume ratio. The catholyte is 50-mM potassium ferricyanide in 100-mM PBS (pH 7.4). Anolyte and catholyte were supplied to the MFC through a syringe pump (PHD 2000, Harvard Apparatus).

Stability and Electrochemical characterization

The stability of the reference electrode was evaluated with a commercially available Ag/AgCl in 3M NaCl reference electrode (BASI). A data acquisition system (DAQ/68, National Instrument) recorded the voltage profiles. Polarization curves were obtained by recording voltage across a series of resistors connected between the anode and cathode, ranging from 148 Ω to 1 M Ω . The DAQ recorded anode and cathode potentials independently. A potentiostat (Gamry Instruments) was used to perform electrochemical impedance spectroscopy measurements, by sweeping the frequency between 100 mHz and 100 kHz with a 30 mV_{pp} ac voltage excitation. Gamry Analyst software fit the data from the measurements.

RESULTS AND DISCUSSION Operation principle

Figure 1(c) shows the bio-inspired reference electrode enclosed in a 25 μ L microfluidic chamber. Layers of *Geobacter sulfurreducens* enriched biofilm form on the anode in the 25 μ L reactor. The *Geobacter sulfurreducens* is an exoelectrogen capable of transferring electrons outside its outer membrane to complete its respiration process. In our application it serves as catalyst to break down the acetate in the anolyte to generate electrons, protons, and carbon dioxide, as depicted in Equation 1 [11].

$$\frac{1}{8}CH_{3}COOH + \frac{1}{4}H_{2}O \xrightarrow{\text{Geobacter?}} e^{-} + \frac{1}{4}CO_{2} + H^{+}$$

$$E_{\text{anode onen-circuit}} \approx -0.5V(\text{vs. Ag/ AgCl}) \qquad (1)$$



Figure 2. The operation principle of the bio-inspired reference electrode. Layers of Geobacter sulfurreducens-enriched biofilm form on the electrode, which serves as catalyst to break down acetate and sets the electrochemical potential for the reaction. The biochemical reaction sets the electrode potential to be very stable ~ -0.5 V versus a commercially available reference electrode, Ag/AgCl in 3M NaCl.



Figure 3. Verification of the operation principle of the bio-inspired reference electrode. When Geobacter inoculum is present, the reference electrode potential is almost constant at approximately -0.5V versus Ag/AgCl in 3M NaCl (for both Pt and carbon electrodes), while when no Geobacter inoculum exists the potential varies greatly.

This is an electrochemical reaction and has an electrochemical potential of \sim -0.5V versus Ag/AgCl in 3M NaCl, as illustrated in equation 1 and Figure 2 [12, 13]. Open circuiting the reference electrode forces exoelectrogens on

the electrode unable to transfer electrons to the electrode to complete the respiration process. Consequently the open circuit will set the reference potential to be the open circuit potential of the anolyte, sodium acetate, which is ~ -0.5 V versus the Ag/AgCl in 3M NaCl. Thus, the *Geobacter* enriched biofilm grown electrode may serve as the stable reference.

The bio-inspired reference electrode was evaluated by comparing the potential of the reference electrode with a group of controls, as depicted in Figure 3. When Geobacter sulfurreducens-enriched biofilm forms on the reference electrode, either Pt or carbon, stable potential establishes at approximately -0.5V versus Ag/AgCl in 3M NaCl. In contrast, when no biofilm exists on the anode, the potential of the electrode fluctuates significantly, with a stability of mere ± 42 mV/day. This verifies the Geobacter sulfurreducens-enriched biofilm is critical for the operation of the bio-inspired reference electrode. Furthermore, we a strong acidic solution to used bleach the Geobacter-covered electrode, showing poor potential stability of ± 37 mV/day, suggesting the micro-organisms deliver key features of stable potential over the time.

Long-term stability



Figure 4. Long-term stability of the bio-inspired reference electrode potential (vs. Ag/AgCl in 3M NaCl) for > 1 week. The electrode maintained a stable potential of -0.465 V with a stability of ± 1.07 mV/day; insets: a comparison of stability and period of the bio-inspired reference electrode with prior art on microfabricated reference electrodes, showing the bio-inspired reference electrode provides substantially more stable potential and longer stability period over others.

The long-term stability of the bio-inspired reference electrode was evaluated as well. As illustrated in Figure 4, the bio-inspired reference electrode presents a very stable potential of -0.465 V with a stability of ± 1.07 mV/day for over a week. The stability of the reference electrode is 2.7 and 3.0 fold higher respectively, while the stability period is 116 and 11 fold longer, respectively, over prior work [9, 10]. Both the stability itself and the stability period support the excellent performance of the bio-inspired reference electrode.

Electrochemical characterization

The bio-inspired reference electrode was integrated in a MEMS microbial fuel cell (MFC) to explore the usefulness. A thin film Cr/Au electrode served as anode. Figure 5(a)

shows the polarization curve of the MEMS MFC. The polarization curve includes the voltage across the load as well as anode and cathode potentials, with respect to the reference potential. The curve shows three sections: the activation region, the linear region and the concentration region. The integrated bio-inspired reference electrode allows the independent measurement of the anode and cathode potentials, which has been considered as a significant challenge in the previously reported MEMS MFCs [14-16]. As the current increases (the external load decreases), the cathode potential shows little change, reducing from 0.74V to 0.64V, while the anode potential changes significantly, increasing from -0.06V to 0.62V. The significant anode potential change becomes vivid, from 0.23V to 0.62V, in the concentration region where the current remains almost unchanged. This agrees well with prior studies that the current generation capability of the anode limits the operation of MFCs [4]. The linear fit of the linear region of the polarization curve vields an internal resistance of 1,640 \pm 148 Ω . Figure 5(b) shows the quasi-voltammetry curve. The voltammetry curve follows a typical sigmoid shape and the mid-potential for the quasi-voltammetry curve is 0.08V versus the bio-inspired reference electrode, which is also in good agreement with prior arts [4, 17].



Figure 5. Electrochemical characterization of a MEMS Microbial Fuel Cell (MFC) using the bio-inspired reference electrode, integrated inside the anode chamber: (a) polarization curve, which includes the potentials across the load as well as anode and cathode potentials, with respect to the reference potential; the polarization curve shows three sections, the activation region, the linear region and the concentration region; (b) quasi-voltammetry curve; both curves are well in agreement with prior arts. The mid-potential for the quasi-voltammetry curve is 0.08V versus the bio-inspired reference electrode, which is also in good agreement with prior

arts.



Figure 6. Electrochemical impedance spectroscopy characterization of the MEMS MFC, integrated with the bio-inspired reference electrode. The total internal resistance, 1,891 Ω agrees with the internal resistance by polarization curve.

Figure 6 shows electrochemical impedance spectroscopy characterization of the MEMS MFC, integrated with the bio-inspired reference electrode. The total ohmic resistance of the MEMS MFC was measured to be 1,891 Ω , agreeing well with the internal resistance obtained by the linear fitting of the polarization curve.

CONCLUSION

In summary, inspired by the possibility of controlling the respiration of micro-organisms, this study presents a novel reference electrode having ± 1.07 mV/day stability over one week. This work explores the operation principle of the bio-inspired reference electrode using Geobacter sulfurreducens enriched biofilm on the reference electrode. The long-term stability of the bio-inspired reference electrode was evaluated using a series of controls as well. The long-term stability benefits from the Geobacter sulfurreducens enriched biofilm on the reference electrode, which establishes a stable electrochemical potential of ~ -0.5 V versus the Ag/AgCl in 3M NaCl. The bio-inspired reference electrode was integrated in a MEMS MFC shows electrochemical standard measurements, including polarization, voltammetry and electrochemical impedance spectroscopy. Those data agree well with prior arts which use a bulky Ag/AgCl reference electrode, demonstrating the bio-inspired electrode indeed serves as an alternative stable reference.

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A HEXAGONAL MULTIPOINT PH AND TEMPERATURE SENSING NEEDLE FOR 3D PIG HEART MUSCLE PH MONITORING WITH TEMPERATURE COMPENSATION

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ABSTRACT

The paper presents a hexagonal silicon microneedle with an outer diameter of 0.7mm same size as the medical needle of 22G, where an Au/Cu-based resistive temperature probe is back-to-back, flip-chip boned with a (IrOx)/AgCl-based pH probe using SU-8 as the bonding layer. The microneedle containing two sets of temperature and pH sensors can exhibit a linear sensitivity of $0.0298\Omega/^{\circ}$ C and -58mV/pH respectively and sustain up to ~3.8Nt compressive load before buckling failure that is mechanically robust enough to penetrate myocardium. Owing to the reduction of process complexity by separating probe fabrications, the new needle can have at least 10% overall improvement in comparison with the prior arts in terms of the probe size and sensitivity and 220% improvement in terms of buckling strength. In addition, a 3D pH measurement of a pig heart has been performed with temperature compensation in this work.

INTRODUCTION

Small form factor is the key feature in the next generation biomedical instruments which facilitate the new exploratory in the signal detection down to the tissue or even cell level [1-3]. For instance, Ivorra et al. developed a minimally invasive needle shaped probe to measure the electrical impedance of living tissues of rat kidneys for the study of the extracellular medium and found the appearance of 65% impedance change while the kidney was kept in an ischemia period of 45 minutes [1]. Chan et al. demonstrated a polycrystalline diamond neural probe equipped with both electrical and electrochemical recording abilities can efficiently detect Norepinephrine in vitro [2]. Zheng et al. employed a silicon -nanowire field-effect transistor array for multiplexed real-time monitoring of protein markers and telomerase activity with high sensitivity and selectivity activity in clinical samples, which disclosed the potential application of the platform for diagnosis and treatment of cancer and other complex diseases [3].

On the other hand, recent researches in 3-D measurement has paved a way to help us understand the biosignal distribution and thus the development of appropriate therapies in a biological system. Nanoscale devices, such as nanowire or nanotube FET probes, 3-D macroporous nanoelectronic scaffolds etc. have been successfully developed for intracellular electrical recording of action potentials from single live cells to tissues as well as 3-D neural activity mapping for the fundamental biophysical studies and medical monitoring and intervention [4]. The establishment of multicellular 3D aggregation in vitro model based on the recorded electrical potential and impedance using the combination of the bio-hybrid sensors like integrated electrode arrays with a self-assembling peptide nanofiber scaffold for mimicking the cellular microenvironment in a living organism has become a critical research task for determining the effectiveness of drugs and therapies [5,6].

Prior studies have shown the heart sarcolemma failure can be detected via the change of the pH value of heart muscle, resulting from the massive influx of calcium into the muscle cells during hypoxia [7]. Cardiovascular surgeon could have a lower surgery risk as long as there is a minimally invasive probe can be inserted in the right or left ventricular anterior wall for fixed-point temperature and pH sensing. Previously, we successfully developed a silicon probe integrated with multiple Au/Cu-based resistance temperature detectors (RTDs) and three (IrOx)/AgCl-based pH meters for heart muscle monitoring [8,9]. The integrated sensing probe could detect the pH variance at different sites to show the real-time status of muscle like decomposition and related germs breeding and \sim 3°C difference between the outer and inner parts of the pig heart, which is an important factor in heart surgery.

Nevertheless, bulky needle size and handling difficulty resulting from tens of interconnects and asymmetrical needle shape, respectively, still prevent the probe from the translation of clinical advances into practice. This paper will demonstrate a new probe design with simpler fabrication processes by bonding two micromachined trapezoidal probes to form a robust hexagon-shaped needle where the space for sensor feedthroughs and process-sensitive materials can be effectively reduced and protected for better sensing performance.

PROBE DESIGN AND FABRICATION

In the prior probe design, a total of six sensors, three for each temperature and pH sensor respectively, are distributed in a silicon shank expected to detect the signals at three different depth of myocardium as a result of a 1.1mm wide probe where sixteen electrical interconnects are distributed. For realizing minimal wound that can recover without being sutured and having a good surgical field that requires the probe to be placed in the back site of heart limited by chest cavity during open cheat surgery, it is necessary to resize the probe. Fig. 1 shows the new cardiac sensing probe whose shank width and length have been reduced from 1.1 to 0.7mm and 8 to 6mm, respectively, in comparison with that of the prior one. The newest needle comprises of two silicon probes back-to-back flip-bonded to form a hexagonal type needle where pH and thermal sensors are placed at two sides of the probe shank, respectively, which can also effect resolve the indispensable space resulting from the interconnects.



Fig. 1. A micrograph of two different cardiac sensing probes: prior design (left [9]) and the new design (right).

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Temperature and pH Sensors

The temperature sensor is designed with a serpentine-shaped resistance temperature detector (RTD) made of electroplated Cu with Au same as the prior work because of high temperature coefficient, TCR~4.29x10-3/°C, good linear dependence of resistivity from -100 to 260°C, and compatible with CMOS backend process. Besides, Au surface on the electrical interconnects can exhibit good biocompatibility and be easily applied with any flexible electrical feedthrough via solder bonding or metal-metal thermal-compressive bonding for in-situ monitoring heart muscle. In the temperature sensing probe, there are only two sensing sensors spaced with 5mm apart for myocardium temperature monitoring at different depth. As for the pH probe, 600mm x 200mm silver-silver chloride (Ag/AgCl) and Iridium oxide (IrO_x) pads are used for pH reference and sensing electrode, respectively. Nernstian equation listed as follows show the pH sensitivity will be -59mV/pH at 25°C if the sensing film electrodes are with space charges:

$$E = E^0 - 2.303 \frac{RT}{F} \text{pH} = E^0 - 0.05916 \text{pH}$$
(1)

where *F* is Faraday's constant with a value about 96500 C/mole, *R* is the value of gas constant 8.314 J/mole K, and E^0 is the standard potential with a value of 577mV for AgCl. The pH value is measured by open circuit method.

Microneedle Fabrication

Fig. 2 shows the process flow for the cardiac sensing microneedle fabrication using two 250um thick Si(100) substrates. one for temperature probe and the other for pH probe. Firstly, thermal oxide (6000Å) and LPCVD nitride laver (7000Å) are deposited and photo-patterned using PR AZ4620 and reactive ion etcher on the silicon substrates as electrical insulation and mask layer for KOH silicon anisotropic etching (Fig. 1(a)), Secondly, sputtering and photo -patterning a layer of Ti/Cu/50nm/100nm on the front side of the substrate as a seed layer for electroplating 1µm thick Cu patterned as sensor parts and covered with electroless-plated Ni(0.1µm)/Au (0.4µm) (Fig. 1(b) or Fig. 1(d)). The Au layer can be used for the prevention of copper oxidation. So far, both temperature and pH probes have been proceeded in common. The temperature probe will be realized after the seed layer removal by Cu etchant and buffer HF solution and then KOH etching (Fig. 1(c)).

For the pH probe fabrication, the aforementioned substrate is selectively electroplated with 1.5 µm thick Ag pads, which will be chlorinated with 50mM FeCl3 solution for 20 second and soaked in a 3M KCL solution to stabilize the potential of AgCl for the realization of the on-probe Ag/AgCl reference electrode fabrication (Fig. 2(e)). The substrate is then covered with a layer of Cu to protect the as-fabricated interconnects and pads for probe patterning by KOH etching (Fig. 2(f)). After the removal of the Cu layer, the IrOx-based working electrode is fabricated on the top of the Cu/Ni/Au pads area using the cyclic voltammetry electroplating method where the electroplating solution contains 0.75 mg of IrCl4 dissolved in a solution of 50mL of D. I. water, 0.5mL 30% H₂O₂, and 250 mg Na₂C₂O₄ and will be adjusted to have a pH value of 10.5 by adding 1M K₂CO₃ and stabilized for 2 days. The electrochemical method is conducted using a three electrode cell system, commercial Ag/AgCl as a reference electrode, platinum foil as a counter electrode, and Au pad of the pH probe as a working electrode. Adapt cyclic voltammetry method from 0V to 0.6V with respect to the AgCl electrode at the rate 20 mV/s for 280 cycles to electroplate the IrO_x film followed by bathing the substrate in a pH 7 buffer solution for 15 hrs. for potential stabilization of the IrOx.



Fig. 2. Process flow for the cardiac sensing probe fabrication.

Once two sensor probes are fabricated separately, the probes will be back-to-back flip-chip bonded using SU-8 at 150°C for 3min. to form the hexagonal multipoint pH and temperature sensing needle.

RESULTS AND DISCUSSION

Figure 3 shows the as-fabricated hexagonal microneedle comprising of two sets of RTDs and pH meters (Fig. 1(a) and (b)). Figure 1(c) and (d) show close views on the shank and tip, the RTD, and the pH meter of the microneedle. Fig. 4 shows the characterization results from one pair of the RTDs and pH meters on the hexagonal microneedle whose sensitivities are $0.0298\Omega/^{\circ}C$ and -58mV/pH i.e. ~10% performance improvement than the prior art mainly due to process simplicity. Besides, excellent linear dependence of the resistivity on temperature and the open circuit potential (OCP) and pH values respectively have been accomplished with higher than 99% correlation. The measured titration cycle (Fig. 4(b)) shows the pH sensor can exhibit excellent reversibility.



Fig. 3. As-fabricated hexagon shaped multipoint pH and temperature sensing probe, (a) RTD side, (b) pH meter sides, (c) probe tip and shank, (d) enlarged view on the resistive temperature sensor (top) and enlarged view on the pH meter (down).



Fig. 4. (a) Resistance vs. temperature from one of RTDs, TCR of the Cu/Ni/Au (1 μ m/0.1 μ m/0.4 μ m) ~4.79x10⁻³/°C with linearity of 99.1% (b) The measured titration cycle, and (c) The OCPs from one of pH meters measured by dipping the probe in pH 2, 4, 6, 7, 8 and 10 buffer solutions, the meter sensitivity is -58 mV/pH with linearity of 99.5%.

Fig. 5 shows the measured maximum compressive strength of the microneedle, i.e. before buckling failure, about 2.2X improvement, i.e. from 1.7 to 3.8Nt, in comparison with that of a single trapezoidal probe. Because the needle is broken as two parts and the failure interface does not happen in the SU-8 bonding layer, the maximum load to cause the probe to be in a state of unstable equilibrium, i.e. failure, can be estimated by the equation as follows:

$$F = \frac{\pi^2 EI}{\left(KL\right)^2} \tag{2}$$

where F: maximum load, E: modulus of elasticity, I = area moment of inertia of the cross section of the probe, L = unsupported length of column, and K: column effective length factor. Since the maximum load is proportional to the area moment of inertia of the cross section, the 2.2X improvement is lower than the theoretical calculation. It could be attributed to uneven load applied on the probe during the test. Further investigation is required to understand the failure mechanism.

Fig. 6 shows the multipoint temperature measurement in the pig heart kept at 5°C in a refrigerator for 2 hours and then tested @24°C. With a good temperature match between the RTDs and the commercial sensors, ~3°C difference detected at two points, 0.5 and 1 cm underneath the pig heart surface respectively, indicates that temperature compensation is required for the pH detection in thick muscle. In addition, according to Nernstian relation (Eq. (1)), the pH value is correlated with environmental temperature. Fig. 7 shows the OPV measurement under different temperature where the pH values are calibrated using a commercial pH probe. The slope of the regression lines increases with increasing temperature. Thus, temperature compensation is required for following pH monitoring of pig heart muscle owing to the temperature difference within the pig muscle. Fig. 8 shows a real-time 3D pH measurement in a pig heart. A pH profile at four different positions (A, B, C and D) with different time, t = 0, 2, and 4 hours shows that the pH variance and difference at the same depth and in the inner and outer sides of the heart will become smaller after with temperature compensation. The 3D profile also shows the status of heart muscle will be similar at the positions with the same depth and could be different with muscle depth. Meanwhile, it is found that the detected pH values are larger at the positions close to the heart surface, which is consistent with the prior result. The higher pH value can be attributed to the decomposition of glycogen accompanied by the generation of amino acid and peptide at a higher temperature.

The hexagonal microneedle can be also accomplished using double-side KOH silicon etching instead of back-to-back flip-chip bonding process. Double side etching process can make the sensing needle with better structure rigidity due to the lack of bonding interface in between two probes. However, process compatibility and manufacture yield should be taken into consideration. A passivation layer is required to protect the working and sensing electrodes of the pH probe in the KOH etching and can be patterned without causing any detrimental effects on the IrOx electrode after the KOH etching.



Fig. 5. Maximum compressive strength characterization (Inset). There are total 12 samples tested (6 for each case).



Fig. 6. A multipoint temperature measurement in a pig heart using commercial temperature sensors and the as-fabricated needles: (a) the measurement setup and (b) the temperature difference of $\sim 3 \,^{\circ}$ C detected at two points, which are located 0.5cm and 1 cm. respectively, underneath the pig heart surface.



Fig. 7. The OPV measurement under different temperature.



Fig. 8. Multipoint pH measurement in a pig heart: the pH profile at four different positions (A, B, C and D): the upper/lower parts are without/with temperature compensation, respectively.

CONCLUSION

In conclusion, we have successfully demonstrated a hexagonal silicon microneedle with the size of 22G. The microneedle comprises of two sets of temperature and pH sensors which can provide the functionality of multipoint sensing and sustain up to \sim 3.8Nt compressive load before buckling failure that is mechanically robust enough to penetrate myocardium. Owing to the reduction of process complexity by separating probe fabrications, the new microneedle can exhibit better electrical and mechanical characteristics in comparison with the prior work. In

addition, a 3D pH measurement of a pig heart has been performed with temperature compensation, which shows smaller pH variance and difference at the same depth and in the inner and outer sides of the heart. We think the microneedle can facilitate correct multi-point pH detection potential for enhancing the successful rate of heart transplant surgery.

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A WIRELESS PASSIVE STIMULATOR OF CARDIOMYOCYTES

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ABSTRACT

Electrical stimulation to promote cardiac cell function, is well established; yet current methods are all limited to wired stimulation systems. To overcome this limitation, herein we report a wireless passive stimulator to induce contraction in cardiomyocytes with a desired frequency. The wireless stimulator contains passive electrical components, on-board antenna, and one pMOS, and is activated by external RF carrier/pulses. The performance of the stimulator is evaluated on neonatal rat cardiomyocytes, side by side by wired stimulation. The results show that synchronous cell beating behavior via wireless stimulation concurs well with those upon wired counterparts, suggesting that the wireless passive stimulator holds significant potential in heart electrical stimulation and regeneration.

INTRODUCTION

Applying electrical pulse stimulation on the cardiac cells, to mimic the bio-potential signal generated in native heart, has been proven to significantly improve the key properties of the engineered tissues such as cell alignment, protein expression, and contractility [1]. Currently, the electrical stimulation is mainly executed in vitro when cardiac tissue is exposed to electrical signal source, such as a function generator. Applying electrical stimulation in vivo under an implanted environment holds a substantial challenge mainly due to the wiring setup, imposing severe limitations on regeneration and repair of damaged heart. Despite numerous attempts on wireless implantable stimulator [2-4], few have been specifically designed to stimulate cardiac cells and produces comparable results to a wired counterpart. Here we report a wireless passive cardiac cells stimulator, which features battery-free, little power consumption, and nearly zero heat generation. The stimulator successfully delivered stimulation pulses large enough to induce synchronized beating behavior of cardiomyocytes, suggesting a promising application on heart repair and regeneration.

Figure 1(a, b) shows the structure of the passive stimulator prototype, which consists of a plastic substrate, an on-board antenna, and multi-stage charging/discharging circuits for generating electrical pulse stimulation. The antenna is designed and simulated in HFSS to optimize its directivity and radiation efficiency. Frequency of the RF signal is chosen to be 2.4 GHz based on previous published work [5]. Output of the antenna is fed to the circuits shown in figure 1(c). The principle of the circuits is to use charge pump to increase voltage level and store charges on output capacitor C_4 . The pMOS T_1 is used to control charging/discharging of C_4 to generate output pulse.

One unique characteristic of the stimulator which distinguishes it from previously reported wireless

stimulators [3-4], is the onboard antenna to establish RF communication between the stimulator and external system, compared to utilizing inductive coupling. The inductive coupling imposes a challenge of requiring large cross-sectional orthogonal planes between the external primary and internal secondary coils, which not only causes inefficient power transfer in implantable environments but also may induce unwanted current on the tissue [2-3]. The designed wireless stimulator consumes little power, thus facilitates the RF coupling. When the stimulator is at rest, T_1 turns "off". The equivalent circuits in this state consist only of diodes and capacitors. Charges are stored at the output capacitor, C₄, and ideally no power consumption exists during this process. When T₁ turns "on", the charges accumulated on C₄ begin to discharge, generating a pulse signal across the load. Again, due to the very short pulse duration (msec), the stimulator consumes low power during this process. As a result, total power consumption is extremely low which allows RF coupling to drive the stimulator. The low power consumption also ensures the safety of the stimulator as an implant, via little heat generation in situ.

Figure 1(b) shows the photograph of a fabricated wireless stimulator on Acrylic. The antenna and routing of circuits were formed by a thin gold film (Cr/Au; 20/100 nm). Discrete electronic components were mounted using conductive silver epoxy (Electron Microscopy Science). The preliminary proof-of-concept stimulator sizes $50 \times 60 \text{ mm}^2$. Once we verify the functionality of the stimulator, we will miniaturize it by decreasing the footprint of antenna and implementing circuit integration techniques.



Figure 1: (a). Schematic of the wireless passive stimulator; (b) Photograph of a fabricated stimulator on acrylic sheet; (c) Circuit diagram of the stimulator.

Figure 2 illustrates the external system for RF modulating and transmitting. We use an arbitrary wave generator (Agilent 33250A), to produce a reverse pulse to

AM modulate the 2.4Ghz RF carrier, which is generated by RF signal generator (Agilent E4432B). The modulated signal is amplified and radiated through an external antenna (A10194, Antenova). The stimulator receives and demodulates the modulated signal to generate output pulses where frequency and pulse width are determined by the modulated input.



Figure 2: Schematic of external RF modulation.

EXPERIMENT SETUP AND RESULTS

We conducted electrical stimulation in a custom made bioreactor chamber (figure 3(a)) which was built based on previously established protocols [6]. Briefly, two parallel carbon electrodes (5 mm length) were placed on a plastic petri dish (6 mm diameter) with 1 cm spacing. The carbon electrodes were fixed using silicon adhesive. Platinum wires were attached to each of the carbon electrodes at the opposite ends and were fully covered by silicon adhesive. After assembling, we filled the bioreactor with Dulbecco's phosphate buffered saline (DPBS) and used it as the load of the wireless stimulator. The applied electrical signal was a 1 Hz, monophasic square-wave pulse with 2 ms pulse duration. This parameter was also chosen based on protocol in order to mimic those existed in nature [1].

The electrical property of human tissue is known to be capacitive rather than purely resistive [7]. For RF powered implanted wireless devices, the capacitance imposes loading on the output signal. The loading effect needs to be characterized, especially when the wireless devices often have finite power delivery capability. Therefore, before using bioreactor, we characterized the output of the wireless stimulator with various loading capacitors as shown in figure 3(b). As the loading capacitors become larger, amplitudes begin to drop due to output power limitation. However, even when the loading capacitance is as high as several microfarads, the amplitude can rise over 5 V, which is sufficient for stimulating cardiac cells.

Using bioreactor as the load, we characterized the output amplitude of the wireless stimulator with both radiated RF power and wireless transmission distance. Figure 3(c) displays the measured pulse amplitude changes as a function of RF power at various distances. It can be seen that amplitude drops quickly as RF power decreases or the distance increases. The radiated RF power was limited to 30 dbm (1 W) according to FCC regulations [8]. As a result, the wireless stimulator was required to be placed

within 5 mm away from the external antenna to produce sufficient voltage output for cardiac cells.



Figure 3: (a). Photograph of bioreactor which the wireless passive stimulator was evaluated. (b). Amplitude of the electrical pulse output as a function of loading capacitance. (c). Amplitude of the electrical pulse output to the bioreactor as a function of RF power at different RF transmission distances.

We obtained ventricular cardiomyocytes from 2-day old neonatal rats based on previously established protocols approved by Institution of Animal Care at Arizona State University [9]. Briefly, heart was surgically cut from the thorax and was sliced into pieces and placed inside trypsin solution prepared in Hank's balanced salt solution for 14-16 hours. Then, heart pieces were neutralized and subjected to collagenase treatment to release the cell populations. Gelatin methacrylate (GelMA) hydrogel was synthesized as following [9]: first, type A gelatin (Sigma-Adrich) (10% w/v) was fully dissolved in DPBS at 50°C. Next, methacrylate (MA) (8% v/v) was added dropwise to the gelatin solution and stirred for 3 hours at 50°C. The solution was diluted five times to stop methacrylation reaction. Then, the solution was placed in dialysis tubes (12-14 KDa molecular weight cutoff), which was placed in deionized water at 45°C under stirring for 7 days to eliminate unreacted MA and salt. The dialyzed solution was filtered using a 0.2 µm membrane and then freeze-dried for 7 days to obtain GelMA foam. Gold nanorods (GNRs) were also synthesized via a seed-mediated silver-assisted growth method [10]. Briefly, gold nanoparticles (~4 nm diameter) were synthesized and used as seeds. Next, the seeds were grown anisotropically in growth solutions containing silver nitrate (AgNO₃) and ascorbic acid. Finally, gold nanorods were separated using high-speed centrifuge at 14000 rpm for 12 min. GelMA-GNR hybrid hydrogel constructs (150 µm thick) were fabricated by photo-crosslinking of GelMA-GNR prepolymers solution using UV light (25 sec exposure time). Isolated cardiomyocytes (750,000 cells/sample) were then seeded on GelMA-GNR hybrid hydrogels and fed by cardiac culture media. The media was exchanged with fresh one every other day. Cardiac tissue constructs were utilized

for external stimulation on day 8 of culture.

Cardiac tissue constructs (figure 4(a)) were placed between the two carbon electrodes of the bioreactor, and the bioreactor was placed under microscope (Observer Z1, Zeiss) to monitor cell contraction activity. Once synchronous contractility was observed, a video was captured. At the same time, electrical signal output from stimulator was recorded using data acquisition card (NI6216, National Instrument) and NI signal Express. Electrical current output signal was obtained by measuring the voltage drop across a 1 Ω resistor that was connected in series with the bioreactor. After real time cell contraction recording, we utilized a custom-written MATLAB code to process the videos and generate representative cell beating signals. We conducted both wired and wireless stimulations, and an arbitrary waveform generator (BK Precision 4052) was used as control to apply wired electrical stimulation.



Figure 4: (a) Representative phase-contrast image of cardiac cell; (b) Cardiac cells beating activity at 0.5, 1, 2Hz frequencies under wired and wireless stimulations.

Figure 4(b) shows the representative cell beating signals obtained at 0.5, 1, and 2Hz frequencies for wired and wireless stimulations. Little discrepancy exists between cell beating curves under wired and wireless stimulation, suggesting the robust functionalities of the wireless stimulator. This is the first attempt, as far as the authors are aware, to stimulate synchronous cardiac cell contractions using a wireless stimulator.

To further evaluate the performance of the wireless stimulator, we recorded the output voltage and current waveform from wired/wireless stimulators (figure 5) during cells contractions. Relatively high current (40-60 mA) was needed to successfully stimulate the cardiac cells. The high current driving capability is beyond the output capability of previously reported wireless stimulators [2-4]. The wireless stimulator shows a rapid drop of current and voltage profiles. This phenomenon is caused by the voltage discharging behavior of the onboard capacitor. The output voltage and current profiles of wired stimulator (function generator) showed much less change, as expected, because the function generator has significantly larger driving capabilities than the wireless stimulator.



Figure 5: Electrical voltage and current output applied to cardiac cell recorded in (a) wired stimulator and (b) wireless stimulator.

The wired stimulator is a commercialized function generator whose output resistance is fixed at 50 Ω in order to match 50 Ω transmission line cable. On the other hand, wireless stimulator has an output resistance as high as 10 $k\Omega$. The difference between output resistances directly impacts the discharging speed of any charges stored in bioreactor. As mentioned in published study [1], bioreactor is highly capacitive at DC due to the electrolytes inside. For the wireless stimulator, the combination of large capacitance of bioreactor and high resistance of stimulator resulted in a long discharging time, leading to the non-zero low level waveform shown in figure 5(a). Although the output waveform from wireless stimulator demonstrates inconsistency with its wired counterparts, it is sufficient to induce well controlled cells contractions, achieving the goal of electrical stimulation.



Figure 6: Comparison of voltage excitation thresholds of wireless and wired system 0.5, 1, and 2 Hz frequencies.

Figure 6 illustrates the measured voltage excitation thresholds for wired and wireless stimulators. Excitation threshold is defined as the minimum voltage required for cardiac cells to start beating synchronously. As the frequency becomes higher, the excitation threshold increases, suggesting it become difficult to induce synchronized contraction of the cardiomyocytes. This result corresponds well with the prior studies [1]. At 0.5 and 1 Hz, no significant difference was observed within the measured average excitation thresholds for wired and wireless stimulation (around 3 V). Yet at 2 Hz, the excitation threshold for wireless stimulation became 5V, which is slightly larger than that of wired stimulation (3.5V). This increase might be due to the decrease of pulse width which is shown in figure 5.

Here we reported a wireless passive stimulator to control of the synchronized beating behavior of the GelMA-GNR hybrid hydrogel tissue constructs. The fabricated stimulator is a preliminary proof-of-concept of the prototype, and the footprint and operating distance need to be improved in the future.

The onboard antenna currently occupies large area. This is because of the performance of antenna is a strong function of its electrical length. As the antenna becomes smaller, many of its important characteristics decrease, such as radiation resistance, efficiency, and directivity. Therefore, designing a small size antenna with acceptable performance requires many trade-offs. We believe it is possible to drive the presented wireless stimulator with an antenna that is small enough to be injected. This is due to the fact that very little power is consumed by the stimulator itself, mitigating the design requirement for antenna power output capability.

In this work, all measurements were performed in the air at a very close wireless transmission distance. In the real implanted setting, EM wave keeps attenuated as they propagate within the body tissues. This makes it even more difficult for stimulator to generate sufficient voltage signal *in vivo*. These all demand the stimulator needs to be improved in practical settings. Several mitigations exist, including optimized multi-stage charging/discharging circuits, directivity enhancement of the antenna, and minimization of RF loss in the plastic substrate. Evaluating the wireless stimulator in a tissue emulating phantom also conveys more realistic performance evaluation, which remains as our future study.

The wireless passive stimulator may find potential applications in cardiac stimulation and regenerative therapy [11], through integration/injection of engineered tissues/cells. Currently, the success of this therapy remains hindered by low survival rate and unsynchronized contractions of the transplanted cells [11]. We believe that by injecting a wireless passive stimulator, it would be possible to synchronize cardiac contraction, thus improving the effect of cardiac regenerative therapies.

CONCLUSION

In this work, we report a wireless passive stimulator for inducing of contractions of engineered cardiac tissues. The cardiac cells beating signals recorded under wired/wireless stimulation show little discrepancy. Recorded output electrical signals from the stimulator demonstrate output current amplitude larger than 60 mA, which is significantly higher than those of previously reported stimulators. Cell excitation thresholds for wired/wireless stimulation were measured and compared, which revealed proper agreement with recorded electrical signals. Further optimization and miniaturization of the wireless passive stimulator hold great potential in cardiac regenerative therapy.

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DEEP BRAIN TARGETING STRATEGY FOR BARE PARYLENE NEURAL PROBE ARRAYS

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ABSTRACT

We present a Parylene neural microprobe array and the first demonstration of deep brain (> 5 mm) implantation of bare polymer probes, enabled by a novel micropackaging technique. Targeting brain sites below the surface of the cortex with soft polymer probes typically requires bulky overcoats or insertion shuttles, which greatly increase probe dimensions and tissue damage, negating advantages of the flexible polymers. We achieved deep brain implantation of bare polymer probes by temporarily shortening their effective length using a biodegradable brace, thereby achieving the required transient increase in stiffness (Figure 1). This surgical approach solves a key technical obstacle preventing the widespread adoption of polymer microprobes. Here, we present mechanical, histological, and electrochemical evidence to support this approach.



Figure 1: (a) Buckling force threshold of bare probes is lower than that required for insertion, resulting in deformation. (b) PEG brace reduces the effective length of probes, increasing the buckling force threshold and enabling successful implantation. Buckling force F is modeled by the equation above in which k is the column effective length factor, E is the Young's modulus, I is the moment of inertia, and L is the probe's length. (c) Exposed probes are inserted to brain, PEG brace is dissolved in saline, and probes inserted to desired depth (5.5 mm).

INTRODUCTION

Soft polymers offer an appealing alternative to silicon, glass, or metal materials for the creation of neural probes. Rigid probes suffer inevitable signal degradation over time as chronic tissue irritation leads to an immune cascade that eventually may wall off of the implant [1, 2]. Rigid probes have successfully achieved high-quality recordings on the scale of a few months, but this falls short of long-term goals desired to produce effective, life-long brain machine interfaces [3, 4]. Whereas metals and silicon have Young's moduli on the order of hundreds of GPa, the stiffness of brain tissue is orders of magnitudes softer, at around 10^{-6} GPa. This disparity between the stiffness of brain tissue and implantable neural probes can lead to tissue damage as micro-motion of the brain causes chronic tissue irritation. It is speculated that the use

of soft probes might mitigate this damage and attenuate the immune response and the glial cell sheath that impact achievable signal-to-noise ratios over time [5]. In one study, soft Parylene probes were shown to induce only a 12-17% neuronal loss around the implantation site compared to rigid silicon probes which incurred 40% neuronal loss at 4 weeks post-implantation [6].

The reduced stiffness of polymers, however, presents a technical challenge for surgical insertion into brain tissue. Polymer probes must be temporarily stiffened in order to penetrate brain tissue and for accurate surgical placement, typically via bulky biodegradable overcoats or insertion shuttles [1, 7, 8] which can increase probe cross-section by 1.2-3,900x's thereby significantly adding to acute tissue injury [1, 9]. The use of coatings or stiffeners can lead to other difficulties as well. Coatings may dull probe tips [9], cover electrode recording sites during surgery [7], or cause the probes to curl as the coating dries [10]. Retraction of a stiffener post-insertion may lead to the movement of a probe away from its target site, interfering with proper probe placement [8]. The need for temporary stiffening of probes during implantation has limited development of polymer probes to designs with short shanks (typically 1-2 mm), as shorter probes have larger buckling force thresholds, and has constrained recording sites to superficial cortical structures [1, 11, 12]. Our strategy overcomes these issues, and opens the door for the large scale acquisition of neural recordings from deep brain structures such as the cornu ammonis (CA1, CA3) and dentate gyrus (DG) in the hippocampus.

METHODS

Fabrication and design of Parylene neural probe array

Parylene microprobe arrays, consisting of a Parylene-metal-Parylene sandwich, were fabricated using standard micromachining techniques. A 10 µm layer of chemical vapor deposited (CVD) Parvlene forms the base of each probe. Platinum (Pt) electrode recording sites, traces, and contact pads are lithographically patterned on top of the base layer using sputter deposition at a thickness of 2,000 Å, followed by lift-off. The metal layer is then encapsulated by a 10 µm thick vapor deposited Parylene insulation layer. Electrodes and contact pads are exposed by deep reactive ion etching (DRIE) and the array outline is cut out from the substrate by means of DRIE as well. Devices are removed from the silicon carrier wafer by wetting them with water and gently lifting them with tweezers.

The microprobe array comprises eight probes with 250 μ m center-to-center spacing (5.5 mm long, 20 μ m thick, and 110-150 μ m wide shanks; Figure 2), each with eight platinum (Pt) electrodes (30 μ m exposed diameter). The electrodes are connected to contact pads by 5 μ m wide traces, with 5 μ m spacing. The array is designed to span a 2,000 μ m anterior-posterior distance along a rat's hippocampus, with electrode groups positioned for simultaneous recording from two of the CA1, DG, or CA3 regions. Sham arrays lacking Pt electrode recording sites, traces, and contact pads were used for mechanical, *in vitro*, *in vivo*, and histological testing.

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Figure 2: (a) Schematic showing dimensions of neural probe. Top group of four electrodes targets CA1 and the bottom group targets CA3 in the hippocampus (b) SEM of array (c) close-up of probe tips (d) micrograph close-up of fully fabricated array with Pt electrodes and traces.

Micropackaging of probes for insertion

Prior to surgical insertion, the array is partially encapsulated in a biodegradable polyethylene glycol (PEG) brace to halve the effective length of the probes and increase probe resistance to buckling (Figure 3). The PEG brace is created by sandwiching the probe array in between a two-part polydimethylsiloxane (PDMS) mold, which is then heated in a 50° C oven for 90 seconds. Molten PEG is injected into the cavity of the mold, and the mold is cooled for five minutes before the PEG brace encapsulated array is released from the PDMS mold.

The buckling force threshold of an individual probe can be mechanically modelled as a beam, and is directly proportional to the width (w) and thickness (t^3) of the probe, and inversely proportional to the probe length (L^2), where E is the Young's modulus of the probe material. (Eq. 1) Halving the effective length of the probe theoretically increases its buckling force threshold by a factor of four, which, when designed appropriately,



Figure 3: Fabrication of PEG brace: (a) probe array and silicon holder placed in bottom mold, (b) top mold placed over array, (c) heated PEG injected into mold, and (d) holder and braced array released.

allows the buckling force threshold of the probe to surpass the force required to penetrate brain tissue. For our probes, modelled as beams that are clamped on one end and pinned on the other (k = 0.7) with a constant width of 150 μ m and a Young's modulus of 3.13 GPa (data previously collected by lab), the calculated buckling force threshold of each probe is 0.21 mN and 0.83 mN with and without a PEG brace respectively. End supports of the probes were modelled as clamped by the PEG brace on one end and pinned by friction with the agarose or brain on the other end.

$$F_{buckling} = \frac{\pi^2 E w t^3}{5.88 L^2} \tag{1}$$

Experimental design

In initial testing, the buckling force was directly measured for sham devices with and without the PEG encapsulation. Devices were driven into a metal plate using a computer controlled stepper motor until they buckled, while load was measured with a 50 g load cell (Omega, LCFA-50g). Buckling force was measured for arrays of 8 probes in addition to single probes. Values were compared to theoretical values calculated from beam theory and literature values (~1 mN) of required force for *in vivo* neural probe penetration [13].

In preparation for *in vivo* implantation, benchtop testing was conducted using brain phantoms (0.5% agarose gel solution; Figure 4) to simulate the consistency and density of brain tissue. PEG encapsulated sham arrays were inserted into gel samples using a stereotaxic stage and the following technique (Figure 1): the exposed tips of the probes were slowly driven into the agarose up to an approximate depth of 2.75 mm, at which point the PEG brace was dissolved with saline, and the remainder of the probe advanced to an ultimate depth of 5.5 mm. Probe array were monitored during implantation for signs of buckling and deformation.

In subsequent *in vivo* rat studies, sham arrays encapsulated in the PEG brace were inserted into a male, Sprague-Dawley (SD) rat weighing between 300 g to 400 g. Animal experiments were reviewed and approved by the Institutional Animal Care and Use Committee (IACUC) as well as the Department of Animal Resources of the University of Southern California (USC). During surgery, the superficial cortex above the dorsal hippocampi was exposed by removing a 2×4 mm piece of cranium from each implantation site and carefully removing dura with forceps. Three screw holes were drilled into the cranium to serve as a port for anchor screws. The animal was anesthetized throughout the procedure, as confirmed by negative toe pinch withdrawal reflexes. The neural probe array was then inserted using the technique described above at a speed of 10 μ m/s, then the subject was perfused with paraformaldehyde and sacrificed immediately post-



Figure 4: PEG brace for shortening effective length of probe, (a) top view and (b) side view.



Figure 5: Braced array inserted into 0.5% agarose brain phantom :(a) front, (b) side, and (c) top view.

surgery. Histology was performed following implantation and hippocampal slices (50 μ m thick) were Nissl stained and examined by light-field microscopy to determine the acute damage incurred to neural tissue by implantation of the probe array.

Electrochemical testing of functional devices was performed via cyclic voltammetry to confirm electrode impedance values were sufficiently low for the resolvable acquisition of neural recordings.

RESULTS

The full array of eight probes, unbraced, buckled under a mean load of 1.26 ± 0.19 mN, and were observed to consistently buckle during *in vitro* insertion tests with brain phantoms. The buckling threshold of a single probe could not be accurately recorded within the resolution of our load cell, but by extrapolating based on further experimentation, we estimated a single shank will buckle under a load of approximately 0.65 mN. As expected this is well below the loading anticipated during insertion [13], and consistent with the buckling witnessed during *in vitro* testing. Full arrays supported with a PEG brace had an average buckling force threshold of 4.24 ± 0.24 mN, and for single braced-shanks buckling force threshold was measured directly as 2.14 ± 0.25 mN. Figure 6 displays load measurements of repeated trials with a single shank supported by a brace and Figure 7 displays analogous data for full arrays with and without the brace.

Insertion of braced probes into 0.5% agarose brain phantoms during *in vitro* insertion tests confirmed what was expected following mechanical buckling measurements—that temporarily shortening shanks with the PEG encapsulation technique increased the buckling force threshold of probes and allowed for successful penetration of probes into agarose. During *in vitro* experiments arrays were inserted as described above and PEG braces dissolved within five minutes with repeat saline rinsing. Arrays were inserted into agarose without bending or buckling and could be placed accurately (Figure 5). The footprints made by the shanks in the agarose (stained blue in Figure 5, panel C) were no larger than the cross-sectional dimensions of our probes. This is a significant advantage compared to the enhanced cross-sectional insult to tissue caused by bulky overcoats and stiffeners.

Following benchtop demonstration, braced arrays were implanted into rat hippocampi. Braced arrays penetrated rat brain without buckling. One braced array experienced PEG softening upon exposure to ambient moisture causing the unsupported probes to buckle upon insertion, as visible in Figure 7. Thus, we directly observed that braced devices enable the successful penetration of rat brain, since unbraced devices buckled during insertion attempts.

Successful *in vivo* placement of the probe array in deep hippocampal layers was confirmed in Nissl stained histological slices (Figure 9). The stab wounds match probe cross-sectional dimensions and minimal damage to surrounding tissue was observed. This is a significant advantage over current insertion techniques which rely on bulky insertion aides add dramatically to the physical footprint of the neural probe.

Electrical impedance spectroscopy measurements from functional devices confirm low electrode impedance requisite for neural recordings (< 200 k Ω at 1 kHz) (Figure 8).

DISCUSSSION

Arrays encapsulated with a PEG brace were shown to successfully insert into a 0.5% agarose brain phantom as well as bilateral hippocampal sites in an anesthetized rat. This confirms the feasibility of our technique of temporarily shortening neural probes to achieve successful penetration of probes with longer shanks. Theoretically, this same strategy can be applied to probe



Figure 6: Load exerted on a single probe with PEG brace during induced buckling, three trials. Average buckling force threshold is 2.14 ± 0.25 mN (standard deviation) which exceeds the 1 mN to penetrate brain tissue reported in literature.



Figure 7: Load exerted on an 8 probe array with (solid curves) and without (dotted curves) PEG brace, three trials each. Arrays without PEG brace had an average buckling force threshold of 1.26 ± 0.19 mN and buckled during in vivo insertion, arrays supported with a PEG brace had an average buckling force threshold of 4.24 ± 0.24 mN and inserted without buckling.

shanks of any length, using an incremental PEG dissolution scheme if necessary to sequentially expose short probe segments.

Calculated and experimental buckling force measurements differed quite dramatically for probes with and without PEG brace encapsulation. A single, braced probe was estimated to have a buckling force threshold of 0.83 mN, but according to mechanical testing, had a higher threshold of 2.14 mN \pm 0.25. This discrepancy is curious, especially since the probe was approximated as a beam of constant width, which would have led to a higher estimate of the buckling force threshold. Choosing alternate end conditions for the buckling model (for example, modeling the beam as fixed on one end and free on the other end) would also only further decrease our estimation of buckling force threshold. Further study into this mismatch between expected and experimental buckling forces are warranted.

While PEG braces were created effectively using the two-part PDMS mold, heating of the PDMS in the oven sometimes resulted in contraction of the two thin PDMS layers, bending the probes in the mold. This difficulty was eliminated by lowering the PDMS heating time to 30 seconds instead of 90 seconds in order to prevent distortion of the PDMS layers.



Figure 8: Electrode impedance spectroscopy (EIS) of Pt recording site. At 1 kHz, electrode has an impedance magnitude of 163 k Ω and a phase of -65°.



Figure 9: Nissl stained transverse slices of sham array insertion into left rat hippocampus and sacrificed immediately post-surgery. Slice depth 3.82 mm.

Fully fabricated devices are being tested in insertion studies with the biodegradable PEG brace technique. Complete arrays with microfabricated metal electrodes, traces, and contact pads can experience curling do to internal material stresses upon release of the arrays from the silicon substrate. Such curvatures interfere dramatically with PEG encapsulation and insertion trials, as curved probes cannot be straightened during the PDMS mold, and enter agarose models at an angle, thereby missing their target destination and making precise positioning of electrodes in the hippocampal layer difficult. Different strategies to straighten the curved devices, including heat treatments and material etching to thin the probes in the direction of curvature, are being evaluated.

In on-going work, electrodes will be cleaned via cyclic voltammetry and checked with EIS to ensure intact traces prior to implantation. Arrays packaged with external connections will then be implanted in rats and the quality, chronicity, and density of electrophysiological recordings from active rat hippocampi will be evaluated and compared to their rigid, penetrating neural probe counterparts.

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INVESTIGATIONS OF BIO-INK RHEOLOGICAL PROPERTIES FOR DROP ON DEMAND PIEZOELECTRIC CELL PRINTING

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ABSTRACT

In this paper, the effects of increasing the viscosity of density matching bio-inks on the reliability of printing MCF-7 breast cancer cells using piezoelectric inkjet nozzles are investigated. The cell printing reliability was characterized by the frequency of occurrence of cell reflection in the nozzle: in cell reflection cells moved further away from the orifice after an ejection cycle. To decouple the effects of fluid density and viscosity on cell trajectory, three solutions were used: 1) phosphate-buffered saline (PBS) which has low viscosity and has lower density than cells; 2) low viscosity, density-matching sodium polytungstate (SPT) solution; and 3) high viscosity, density-matching Ficoll sucrose polymer solution. While cell reflection was observed for PBS and SPT solutions, it was not observed for high viscosity Ficoll solution. Propagation of pressure waves induces the transient motion of cells, and higher viscosity of solution causes increased viscous forces on the cells and prevents cell reflection.

INTRODUCTION

Inkjet cell printing has received considerable interest due to its capability to engineer tissues and organs in intricate three dimensional orientations [1, 2]. This technology has potential for applications in tissue engineering [3], cell based assays [4], stem cell research [5] or therapeutics [2]. In particular, the drop on demand (DOD) printing technology enables to precisely and accurately place biological materials (i.e., cells, extracellular matrix) onto substrates. Piezoelectric inkjet systems generate mechanical pulses to squeeze liquids out of the nozzle orifice with high speed, resulting in the formation of well controlled droplets [6]. However, the commercialization of such systems for cell dispensing remains challenging due to less controlled cell behavior.

A particle (e.g., a cell) suspended in a fluid is subjected to hydrodynamic forces. For low Reynolds number (*Re*), viscous force dominates over inertial force such that the Stokes drag force is the dominant force in dynamics of particle motion. However, during droplet ejection the Reynolds number in an inkjet system is high (Re > 10 [7]) and during this transient event the inertial force could be no longer neglected [8]. Both inertia and viscosity act on a cell and makes the cell dispensing more complex, resulting in low reliability [9]. For example, cells sometimes move far away from the orifice (termed as cell reflection) after a droplet ejection [8].

Our previous work indicates that the use of high viscosity, density-matching Ficoll sucrose polymer solution improves cell printing reliability [7][8] by reducing cell sedimentation within the suspension, giving a good match to the expected Poisson distribution of cells within printed droplets [7]. However, this previous work did not separate the effects of fluid viscosity and fluid density. In addition, high viscosity solutions may fall outside the window of operability of some inkjet nozzles. In this paper, we decoupled the effects of fluid density and fluid viscosity on cell printing by using low viscosity, density-matching sodium polytungstate (SPT) solution and respectively investigated their effects on cell motion in a piezoelectric inkjet nozzle.

MATERIAL AND METHODS Piezoelectric Inkjet Nozzle

A piezoelectrically actuated inkjet (Microfab Technologies Inc., Suite, MJ-ABP-0) was used to dispense cells within droplets. The inkjet consists of a 21.7 mm long glass cylindrical tube with an inner diameter of 0.475 mm which tapered to the orifice with a diameter of 80 µm. The middle of the glass tube was surrounded by a piezoelectric element, which consists of a sandwich structure of inner electrode/piezoelectric material/outer electrode. When applying a voltage difference, the electrical field is generated between the inner and outer electrodes, resulting in the deformation of piezoelectric (PZT) actuator. In this way the piezoelectric element enables to produce pressure wave and squeezing fluid through the orifice to generate droplets. The piezoelectric element was actuated by a bipolar waveform (Fig. 1). An arbitrary waveform generator (Agilent 33220A) was used to provide the command signal input to a high-voltage amplifier (Trek Model 603) with a DC voltage gain of 50 V/V. The amplitude and frequency of the electrical waveform depend on the rheological properties. The SPT printing used a bipolar waveform with a peak-to-peak voltage of ± 30 V and a frequency of 60 Hz.

The piezoelectric actuator generates pressure waves using the deformation of PZT under the change of electrical voltage. Notethat the deformation only occurs during the transition period (rise and fall) and ceases during the constant voltage (dwell) period. On the rising (or falling) edge of the waveform, the annular PZT expands (or contracts) its circumference, resulting in an outwards (or inwards) motion of the inner glass material and consequently a negative (or positive) pressure (with respect to the equilibrium). The pressures travels in fluid as an acoustic wave at a certain speed. Bogy and Talke examined the one-dimensional wave propagation along the glass tube (vertical to the motion of the glass) [11]. For the longitudinal acoustic wave, the displacement of the liquid is in the same direction as (or the opposite direction to) the direction of travel of the wave. The wave travels to the orifice and the supply end, where the wave was reflected and moves back towards the orifice. The backwards wave reinforces the positive pressure wave [12]. In this case the pressure wave makes fluid overcome the liquid-air interface, resulting in the ejection of droplets [13]. The motion of the cells and the surrounding fluid as well as the droplets dispensing were recorded by using a high speed camera (Phantom® Miro 4 or Phantom v611).

The back end of the nozzle was connected to a syringe reservoir through tubing (Tygon® AAC00001; OD: 2.38 mm, ID: 0.79 mm; Saint-Gobain). The pressure at the back end was continuously measured by a differential pressure sensor (PX139, Omega) and read by a microcontroller (Uno R3, Arduino). The back pressure was maintained at -500 Pa by adjusting the relative height of the external reservoir to the inkjet nozzle during the printing.



Figure 1: Schematics of a piezoelectric inkjet cell printing system in which a piezoelectric nozzle (MicroFab) is actuated by a function generator (Agilent 33220A) in series with a high-voltage amplifier. The motion of cells and droplets dispensing are visualized using a high speed camera (Phantom® v611).

Cell suspension preparation

MCF-7 breast cancer cells were cultured in an incubator (37°C, 5% CO₂) until confluency in RPMI 1640 culture media. The cells have an average diameter of 15 ± 3.8 µm and a density of 1.068 g·ml⁻¹ [7, 8]. Firstly, the MCF-7 cells were detached from the flask with 0.25% trypsin-EDTA and then treated with bovine serum albumin (BSA). BSA is able to prevent non-specific binding of cells and cell aggregation to efficiently prevent the clogging of nozzle during printing. Afterwards, the cells were stained with 0.1% Toluidine Blue, which stains the nucleic acid of the inherently transparent cells to dark blue and the polysaccharides purple in color, resulting in a higher contrast of the cells for brightfield imaging. Secondly, the sodium polytungstate (SPT, Sigma-Aldrich) was added to phosphate-buffered saline (PBS) to prepare a solution with a density of $1.068 \text{ g} \cdot \text{mL}^{-1}$ that is equal to the density of MCF-7 cells [7], resulting in a neutral buoyancy suspension. The viscosity of the as-prepared SPT solution was measured to be 1.00 mPa·s using a rheometer (Physica MCR301). Finally, the MCF-7 cells were re-suspended in the SPT solution with a concentration of 1,500,000 cells·mL⁻¹ that was determined using a hemocytometer. The viscosity of SPT cell suspensions was measured to be 1.1 mPa·s, which is not significantly different from the solution without cells. Similar cell suspensions were prepared in PBS (Sigma-Aldrich) and in 10% Ficoll PM400 (GE Healthcare) [8].

Imaging setup

An inverted microscope (Nikon Eclipse TE2000-U) was used to capture brightfield images of the inkjet nozzles (see Fig. 1). The microscopy used an objective lens (Nikon CFI LU Plan Fluor 5×) with a numerical aperture (NA) of 0.15. A high speed camera (Phantom® v611) was used to record the cell motion and jet formation with a temporal resolutions of 7.7 microseconds (μ s). Note that the objective lens has a depth of field of approximately 30 μ m which was comparable to the diameter of the cells in the inkjet nozzle. In this case, it focused on the middle plane of the inkjet nozzle. This ensured that only cells within a 15 μ m thick volume around the middle plane of the nozzle were in focus and captured through high speed imaging. Therefore, considering the size of the cells, only cells within this in-focus region were analyzed for the cell tracking and mapping. The videos captured

for out of focused cells were not processed to ensure that any optical distortion due to defocusing was minimized. The high speed camera captured at the rate of 130,232 frames per second (fps) and recorded as an 8-bit greyscale video with a resolution of 256×128 pixels (0.65 µm/pixel). Each video has a size of 5.3 gigabits with 171,178 frames containing around 90 droplet ejections on average. Such that we are able to record the continuous cell motion.

Cell tracking algorithm

The presence of a cell within the inkjet nozzle was identified through an image processing and cell segmentation algorithm using Matlab® (MathWorks). For each frame in the captured high speed video, an image segmentation routine was implemented to highlight the features of the cell present within the nozzle. The grayscale image was first filtered with a Gaussian low pass filter to remove any high frequency noise from the high speed camera. The image was then inverted and filtered with a spatial bandpass filter. The filter performed background subtraction and highlighted the round cell within the images which appeared as a bright spot on a dark background. The cell was then identified as the local maximum value within the filtered image.

Cell mapping

The inkjet printer enables to eject cells with droplets. The printing process is characterized by using the motion of a cell between two droplets. We recorded the coordinates of a cell immediately before droplet ejection was initiated. The coordinates of a large quantity of cells were mapped. Each point on the map was categorized according to the cell behavior exhibited during the droplet ejection. The map shows the distribution of cell behavior.

The map permitted the identification of localized two dimensional regions within the nozzle which exhibited a high probability of certain cell behaviors. Due to the axis-symmetric geometry of the nozzle and small depth of imaging field, the mapped regions can provide insight for the entire threedimensional volume of the inkjet nozzle.

RESULTS AND DISCUSSION

Droplet formation

Figure 2 shows a sequence of high-speed images during DOD droplet formation. For the droplet formation process, the initial state (frame = 0, t = 0, Fig. 2a) was defined as the equilibrium state immediately before the meniscus oscillation. Note that one printing period contained 2170 frames (0.0167 s) that exactly equals one period of the waveform (60 Hz). At this moment the rising edge of the waveform had activated the piezoelectric material and consequently generate the negative acoustic wave (the waveform is at the dwell period), but the wave has not traveled to the orifice. At the equilibrium state, the liquid-air interface exactly coincides with the boundary of the orifice. Afterwards, the expansion wave arrived at the orifice such that the liquid is withdrawn from the equilibrium position (see Fig. 2b, $t = 15.4 \ \mu s$). Then the arrival of the compression wave caused the emerging of liquid (see Fig. 2c, t = 77.0 μ s), which would be pulled back into the nozzle by the expansion wave. In this case, the meniscus oscillation took 60 µs for several frames. Given the speed of sound in salt water free of bubbles or suspended sediments (~1500 m/s), the round trip between orifice and the back end is calculated to take 30 µs, when taking into account the length of the glass tube of 21.7 mm. The suspended cells (and cell aggregations) may induce the scattering of acoustic waves and slow down the sound propagation [14]. To be brief, the acoustic wave caused the liquid-air interface to

oscillate about the equilibrium position at the orifice. This oscillation process took about 1617 μ s and then the liquid-air interface returned back to the equilibrium (see Fig. 2d). After 3080 μ s, the oscillation of the liquid-air interface occurred again.

On the falling edge (from 30 V to -30 V), the piezoelectric material contracted and resulted in a positive pressure (t = 0.01 s). The positive pressure squeezed liquid out of the orifice and make the formation of droplets. The formation of SPT droplets could be categorized as four phases, i.e., (e) liquid extrusion; (f) thread thinning, and consequently (g) break-off; and (h) primary and satellite droplets formation and meniscus damping. Afterwards, the meniscus damping takes around 160 µs and subsequently the liquid-air oscillation would occur twice during the dwell period as same as at the constant negative voltage. The volume of the primary droplet was measured to be 82 pL and the satellite droplet was measured to be 15 pL. The volume of the primary (or satellite) droplet was determined by detecting the droplet edges and fitting to a sphere (or cylinder). Note that the satellite droplets did not merge with the primary droplets and that is undesirable. An actuation profile with lower peak to peak voltage would probably enable optimization to avoid forming satellites.



Figure 2: High speed images of a meniscus oscillation (a-d) and jet formation including (e) liquid extrusion, (f) thread thinning and (g) break off, and (h) breakup of liquid thread into primary drop and satellites and meniscus damping. The images were captured with a lateral magnification of $5 \times$ objective and a numerical aperture of 0.15 at 130,232 frames per second with a pixel resolution of 256 × 128 pixels. This imaging system has an interframe resolution of 7.7 µs and a spatial resolution of 0.65 µm/pixel.

Cell behavior

The positive pressure waves squeezed fluid out of the orifice, resulting in the formation of droplets (see Fig. 2e-h). During the droplet formation, liquid moved within the nozzle such that cells moved. Fig. 3 shows the high speed images and the corresponding cell trajectory for various cell behaviors. In Fig. 3a, a cell (indicated by the black arrow) with a diameter around 12 μ m approached the nozzle orifice with a distance around 40 μ m. During liquid extrusion, the cell moved towards the orifice. After the arrival to the orifice, the cell merged with the meniscus (Fig. 3b) and subsequently the droplet containing the cell was dispensed out of the orifice (termed as cell ejection, see Fig. 3c). Fig. 3d recorded the cell's vertical position (along Y-axis) away from the orifice (y=0) as a function of time. Fig. 3e-g visualized the travel process of a cell. In this case, the cell (with a diameter around 12 μ m) moved closer to the orifice (Δ y=-18 μ m, Fig. 3h) during a droplet ejection, but the cell was not dispensed. This cell behavior is termed as cell travel. In Fig. 3i-k, a cell with a diameter around 20 μ m moved far away from the orifice ($\Delta y=34 \mu$ m, Fig. 31) and this behavior is termed as cell reflection. During the droplet ejection, liquid moved towards orifice, however the cell was reflected towards the opposite direction. In this case, cell reflection indicated that the fluid is unable to control the motion of cells well, making the cell printing less reliable.



Figure 3: High speed images and cell tracking of cell ejection (ad), cell travel (e-h) and cell reflection (i-l) in SPT cell suspension.

Furthermore, the coordinates of cells immediately before the liquid extrusion were recorded for more than 300 printed droplets (Fig. 4). For low viscosity, non-density-matched saline (PBS) solution (Fig. 4a), we observed three cell behaviors: cell travel, cell ejection and cell reflection. For low viscosity, high density SPT solution the cell reflection was also observed (Fig. 4b) which was completely avoided for high viscosity, high density Ficoll solution (Fig. 4c). Note that Fig. 4a (for PBS) and Fig. 4c (for Ficoll) were captured using Phantom Miro 4 high speed camera with an objective lens (Nikon CFI LU Plan Fluor 5×, 0.3 NA). The Phantom v611 camera allows us to record around 100 droplet ejection events in one single video whereas Miro 4 is normally 4 ejection events. In this case, we recorded the continuous motion of cells towards the orifice step by step (see Fig. 4b). At the region where the nozzle has a diameter of 180 µm (with a distance of 150 μ m to the orifice), the step is 4 μ m on average. In this case, the volume of the liquid is around 100 pL, which roughly equals that of a droplet (see Fig. 2).



Figure 4. Cell mapping for (a) PBS, (b) SPT and (c) Ficoll within the inkjet nozzle indicating cell travel, ejection and reflection.

Cheng *et al.* experimentally determined the fluid velocity within the same Microfab nozzle (MJ-ABP-0) using the micro particle image velocimetry [8]. The velocity of liquid could be up to $1 \text{ m} \cdot \text{s}^{-1}$ during droplet ejection and induce a large Reynolds number (>10). For this high *Re* regime, both inertia and viscosity may act on cells. The Stokes number (St) correlates a particle response time with the fluid flow characteristic time, and it is given by

$$St = \frac{\tau}{t_f} = \frac{\frac{\rho_p \cdot d^2}{18 \cdot \mu}}{\frac{t_f}{t_f}}$$
(1)

with τ the time constant of a cell (s), t_f the fluid flow timescale (s), ρ_p the cell density (kg/m³), d the cell diameter (m). The time scale (t_f) of SPT solution was determined as the half of the meniscus oscillation period (T, see Fig. 5). The tf of PBS and Ficoll were obtained from our previous work [8]. The corresponding Stokes numbers (St) were calculated according to eq. (1) (see Table. 1). The relatively large St values were found for PBS (0.67) and SPT (0.38). For St > 0.1, particles slip with respect to the fluid streamline is expected [15]. In this case some cells probably moved from a high velocity region (close to the nozzle) to a lower velocity region, that is, these cells slip with respect to the surrounding fluids. The slip behavior results in the motion of the cells to a position further away from the nozzle (compared to its position at the onset of droplet ejection), which is the so-called cell reflection behavior. On the other hand, Ficoll solution has a smaller St value (i.e., 0.09), indicating a shorter response time that a cell moves with fluid at an equivalent velocity, contributing to the elimination of cell reflection. Ongoing work will evaluate the statistical distribution of cells in SPT within printed grids.

Table 1: Rheological properties of the inks, i.e., PBS, 10% w/v Ficoll PM400, 0.02 M Sodium polytungstate (SPT) and the corresponding response time of cells.

Ink	ρ(g/mL)	μ (cP)	τ (μs)	τ _f (μs)	St
PBS	1.000	1.00	13.4	20	0.67
SPT	1.068	1.00	13.4	35	0.38
Ficoll	1.050	4.86	2.8	30	0.09



Figure 5: Meniscus oscillation: (a) Meniscus position away from the orifice as a function of time after droplet break-off (t = 0) as determined in (b). Fluid flow timescale (t_f) equals the half of meniscus oscillation period.

CONCLUSION

In this work, we compared the effects of solution density and solution viscosity on the trajectory of cells during inkjet printing. The use of a neutral buoyancy salt solution did not prevent the occurrence of cell reflection, while the use of a neutrally buoyant, high viscosity solution did suppress incidents of cell reflection. Future work will examine the effect of cell diameter on cell reflection in the PBS suspensions. Future work will also examine the effect of nozzle geometry on cell reflection.

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PARYLENE ENCAPSULATED SUB-MICRON STRUCTURES FOR IMPLANTABLE BIOMEMS

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ABSTRACT

We present a method for sub-micron machining of flexible, thin-film structures fully encapsulated in biocompatible polymer poly(chloro-p-xylylene) (Parylene C) that improves feature size and resolution by an order of magnitude compared to prior work. A low temperature electron beam lithography (EBL) process compatible with Parylene-coated silicon substrates was developed, and characterized using patterned Ti structures with critical dimensions down to 250 nm, including conducting traces, serpentine resistors, and electrodes with a nano-patterned texture. Using this newly developed technique, the first flexible, free-film Parylene-Ti-Parylene devices with nanoscale components were fabricated and characterized. One application of these sub-micron structures encapsulated in Parylene is next generation minimally invasive implants. Thus, we also demonstrated a prototype high density Parylene-based microelectrode neural probe using our nanopatterning approach.

INTRODUCTION

Polymer MEMS on flexible substrates enable novel microdevices ideal for minimally invasive biomedical implants. The typical approach is to employ surface micromachined structures encapsulated in biocompatible polymers such as Parylene C, polyimide, or PDMS. The selection of polymeric substrates over conventional MEMS materials like silicon or glass conveys several advantages: low elastic moduli, high electrical resistivity, chemical resistivity, optical transparency, and mechanical flexibility. In particular, a major motivation for the use of polymers are their mechanical properties, especially for applications involving contact with soft tissue, such as retinal or neural microprobes, where rigid substrates can induce tissue damage due to mechanical mismatch, and subsequently trigger immune responses that impede performance and device lifetime. In recent years several successful examples of implantable polymer bioMEMS have been demonstrated, including implantable neural probes [1, 2], flow sensors, and pressure sensors [3, 4].

Despite the many advantages of polymeric micromachined devices, a major limitation remains - the poor feature resolution (> 5µm) achievable with typical micromachining processes that are compatible with polymer substrates. While sub-micron fabrication is routinely employed on conventional MEMS materials, there has been limited work transitioning this capability to soft polymer substrates [5]. Nanoscale patterning has been applied to polymers to create flexible stamps and shadowmasks for fabrication on other substrates [6, 7], but direct sub-micron fabrication on polymer surfaces, or for polymeric biomedical implants remains a technology gap [8]. Existing nanolithography protocols involve exposure of substrate materials to high temperatures and energies that exceed recommended operating conditions for most commonly used MEMS polymers. Methods such as nano-imprint lithography and self-assembly benefit from their highlyparallelized approach to cover large surface areas and have been used to pattern repeated arrays of simple structures on polymers [9, 10]. However, these techniques place restrictions on material choice and are pose difficulties in aligning multi-layer patterns, such as those required for many implantable microdevices.

As such, existing polymer MEMS cannot compete with silicon devices on feature density or feature size, placing limits on structures that can be easily produced and the resulting device capabilities. For example, polymer neural microprobes typically contain less than a dozen electrodes for stimulating or recording, whereas the state-of-the-art comprises silicon devices with more than a hundred electrodes per probe [11]. This limitation precludes polymer MEMS with the capabilities, complexity or minimal footprint achievable with conventional materials.

We developed a modified electron beam lithography (EBL) protocol that enables fabrication of biocompatible polymer MEMS with sub-micron feature resolution; our design consists of thin, free-film Parylene devices encapsulating titanium structures with feature size as small as 250 nm. This resolution was achieved with minimal optimization and represents a starting point for achievable nanoscale resolution. We describe our fabrication process and present initial electrical and mechanical characterization, showing our devices as functional, flexible and robust. Finally, we present a demonstrative application of this technology: a prototype Parylene-based cortical neural microprobe, with a greater number and density of recording sites than any prior polymer probe.



Figure 1: Process flow for sub-micron fabrication on Parylene C: (a) Ti contact pads and traces were fabricated on a vapor deposited Parylene film using conventional contact microlithography, (b) PMMA bilayer (PMMA 495, 950) was coated with Cr (15 nm) then patterned with e-beam, (c) Ti features were deposited with lift-off, (d) features were encapsulated in Parylene contact pads were exposed with plasma etch, and device was released.

METHODS

Fabrication

The preparation of encapsulated structures (Figure 1) entailed a combination of conventional UV lithography, polymer micromachining, and a modified EBL method. First, silicon carrier wafers were coated in 10 μ m of Parylene C through chemical vapor deposition (CVD). Structures including alignment marks, contact pads and large electrical traces were patterned lithographically using a OAI hybralign series 200 mask aligner (OAI, San Joesa, CA), then metallized with titanium using e-beam evaporation and liftoff. A PMMA resist bilayer was prepared comprising 495k 2% solids in anisole (400 nm thick) and 950k 6%

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solids in anisole (170 nm thick). Residual solvents were driven off with a 120 °C hotplate bake for 20 minutes following deposition of each layer; the comparatively low baking temperature was necessary to avoid stress-induced Parylene cracking or polymer oxidation. A conductive chromium layer (10-15 nm) was deposited above the resist to prevent charge buildup during e-beam exposure, a consequence of the insulative properties of the Parylene layer. Representative images of the fabrication process development and results are shown in Figures 2-4.



Figure 2: Sub-micron machining on Parylene C under various conditions: (a) Parylene cracking induced by baking PMMA film at 170° C, (b) damage to Parylene film due to uncalibrated e-beam exposure; and Ti features of decreasing size (5 - 0.25 μ m) patterned on Parylene with (c) insufficient and (d) sufficient dose.

EB dose and voltage were varied (150-900 μ C/cm²; 10-20 kV) to determine suitable parameters to achieve test patterns consisting of square arrays (5 & 0.5 μ m sides). Then, squares of decreasing sizes (5-0.05 μ m) were patterned with an optimized dose (420 μ C/cm²; 20 kV) to determine smallest resolvable features for the resist settings used (Fig. 2d). Test structures used in mechanical and electrical testing were patterned at the optimized dose, and consisted of straight and serpentine traces with widths and spacings of 5-0.25 μ m. Following exposure, the chromium layer was dissolved in CR-7 chromium etchant (Transene Company Inc., Danvers, MA), and the structures developed in 1:3 ratio of methyl isobutyl ketone to isopropanol (18-30s).

Dose and size calibration structures were successfully metallized with titanium and the resulting structures examined by scanning electron microscopy. Test structures were metallized with titanium (150 nm) and then encapsulated with a second CVD Parylene layer (10 μ m). O₂ deep reactive ion etching (DRIE) was used to expose the contact pads, and cut out test devices, which were then gently peeled off the silicon carrier to complete release of devices. No observable damage to the substrate or structures resulted from the peeling process.

Mechanical and Electrical Testing

DC resistance measurements were taken across all test traces using a basic 2-point probe on unstrained devices. For DC measurements during mechanical testing, electrical contacts were made using wire and conductive epoxy (silver epoxy, MG Chemicals, Surrey, CA). Devices were first manually bent and twisted while monitoring for changes in resistance or failures of the sub-micron trace. In a second set of experiments, devices were flexed using a moving stage and stepper motor, allowing resistance change to be monitored as a function of bend angle.



Figure 3: Representative SEM images of sub-micron Ti structures on Parylene: (a) straight 5 μ m trace, (b) straight 0.25 μ m trace with inset showing detailed view at higher magnification, (c) serpentine 5 μ m trace (5 μ m spacing), and (d) serpentine 0.4 μ m trace (0.4 μ m spacing).



Figure 4: (a) Photograph of released and encapsulated test structure (400 nm serpentine Ti trace) with magnified inset. (b) Encapsulated device flexed while resistance is recorded. (c) Fractional change in resistance plotted against bend angle for representative test structure.

Neural Probe Prototype

A prototype cortical neural microprobe was fabricated using this method as a demonstrative example of a future application of sub-micron resolution Parylene devices. This microprobe consisted of a flexible Parylene shank (2 mm×150 μ m) supporting 32 electrodes, each patterned with arrays of nano-scale 'bump' structures (400 μ m) as a means to increase the effective surface area. Titanium electrode sites (15 μ m radius) were machined on a Parylene coated silicon wafer along with alignment marks using standard micromachining. The above EBL method was used to pattern the bump structures and connective electrical traces with width and spacing of 750 nm. DRIE was used to cut-out the device shape, before removing it from the silicon carrier.

RESULTS

Careful optimization of the EBL process proved critical for

working with the polymer substrate. The Parylene base layer exhibited cracking under thermal-induced stress and damage during high energy ebeam exposure. Subsequently, processing was tailored to avoid rapid heating or steps above 120 °C, and EBL was conducted at energies at or below 20 kV, with thick resist bilayers to assist in absorbing e-beam energies and protect the underlying Parylene. A metal surface layer proved necessary for high resolution EBL patterning, as without such a layer, surface charges collected and distorted the beam shape. Chromium was chosen due to its ease of removal, and low atomic mass which allowed prepatterned titanium structures to be visualized for alignment.

Exposure doses between 300 and 500 μ C/cm² at 20 kV produced the smallest structures with most clearly defined features; structures patterned at lower doses or energies failed to develop fully while higher doses and energies produced distorted features or damaged Parylene film. Titanium structures with critical dimensions as small as 250 nm could be reliably machined, and exhibited strong adhesion to Parylene even under repeated deformation. Titanium was chosen owing to its demonstrated adhesion to Parylene and proven biocompatibility, though both platinum and gold were also tested, with the latter suffering from insufficient adhesion.

Features below 250 nm were not easily resolved, likely a limit of the achievable aspect ratio of the PMMA bilayer. Test devices with widths of 5-0.25 µm, and spacing of 5-0.4 µm were successfully and repeatedly fabricated (Fig. 4) and proved flexible and robust. Serpentine traces with spacing of 0.25 µm suffered from shorts and collapsed traces, perhaps a result of proximity exposure during patterning. Otherwise, under DC characterization, traces are Ohmic with no recorded shorts or faults even during repeated flexion and torsion (bend angle $> 90^{\circ}$). Key values from mechanical and electrical testing are compiled in Table I, and Figure 5 displays trace resistance as a function of the length/width ratio, from which we calculate resistivity of the thin-film metal (3.5-6.4 $\mu\Omega$ ·m, compared to bulk values of 0.42 $\mu\Omega$ ·m) and contact resistance (50-100 Ω) at the interface between sub-micron structures and larger traces. Mean resistance was observed to vary by < 0.5% under flexion down to 500 µm radius of curvature (Fig. 4bc).

	Straight	Serpentine	
Dimension (nm)	250	400	
Resistivity ($\mu\Omega$ m)	6.35	3.54	
Contact resistance (Ω)	98	48	
Torsion (°)	> 90	> 90	
Flexion (°)	> 90	> 90	

Table 1: Tabulated values are derived from electrical and mechanical characterization of straight and serpentine traces encapsulated in Parylene.

Figure 6 shows images of the prototype neural microprobe. The completed device had 32 recording sites on a 2 mm shank, more than double the electrode density of any prior polymer neural probe, and four times that of prior work done with Parylene C. This high density was achievable due to the small pitch and tight spacing of the connective electrical connections (750 nm), fabricated here through EBL. Traces extended the full length of the 2 mm shank without short or break, and exhibited the same flexibility as traces in test devices. Line resistance for the traces on the prototype was not measured, though can be extrapolated as 150 $k\Omega$ from the regression plotted in Figure 5. Though larger than typical values of traces measuring several μ m wide, at 1 kHz (typical recording frequency for neural probe measurements) the contribution to impedance will be marginal.



Figure 5 Plot of resistance vs. length-to-width ratio of patterned test structures: straight (square) and serpentine (circle) traces.

The nano-patterned structures, arrays of raised titanium bumps in the center of each electrode, served to increase effective surface area, and lower impedance. Each structure was rectangular, 150 nm tall and 400 nm across, and the arrays were calculated to increase effective surface area by 40%.



Figure 6 (a) Prototype Parylene-neural probe machined with submicron features including (b) densely packed conductive traces (750 nm linewidth and spacing) and (c) nano-patterned recording electrodes.

DISCUSSION

Through the use of the fabrication techniques described above, flexible Parylene-based devices were produced integrating electrical traces and structures with pitch and spacing of just a few hundred nanometers. Devices were flexible and robust, with insulated traces functioning despite severe and repeated flexion and torsion. These experiments bode well for the production of a new generation of polymer MEMS, with higher feature density and/or smaller footprints, and the highly biocompatible composition is encouraging for applying this technology to implants.

High resolution patterning on organic polymers is non-trivial; as seen here, issues of temperature, radiation intensity, and charge complicate existing methods developed for semiconductor applications. But these challenges are not unmanageable, and through careful preparation and calibration high resolution patterning, such as EBL, is achievable with high fidelity and good repeatability on such unconventional substrates. The resolutions achieved here were likely limited by the aspect ratio attainable with our choice of resist, and it is likely nanoscale fabrication is possible with only slight modification of this technique.

In on-going work, we are exploring other potential applications, including free-film devices with electrical components, such as resistors and capacitors, directed written into the film using dense serpentine traces and interdigitated comb designs respectively, or high density stimulating electrodes for neural or retinal devices, with large arrays of electrodes small enough to stimulate single sites of interest. We are cognizant of the scalability limitations of this work. Though EBL offers high resolution, the cost per device in time and money is too high for technologies intended for large scale distribution. This technique is useful for prototype devices intended for research, where the scale of production is small, or for prototyping novel devices. For other applications we are investigating pattern-transfer techniques compatible with Parylene that will enable batch-scale fabrication.

CONCLUSIONS

Despite material challenges electron beam lithography can be made compatible with organic polymers for MEMS processing. Through the use of such a technique, and the conformal nature of CVD Parylene, we have produced thin-film biocompatible devices with sub-micron features. This approach has applications for novel polymer implantable devices, demonstrated here through the production of a polymer neural microprobe with high electrode density. Sub-micron processing is a necessary advance for polymer MEMS, and future work will need to strive towards matching those resolutions achievable with silicon and other materials, to match their capabilities in mechanically pliant platforms.

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ULTRA-DIELECTROPHORETIC FORCE SPECTROSCOPY ARRAY FOR PROBING INTERMOLECULAR AFFINITIES

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ABSTRACT

A multiplexed electronic force spectroscopy platform is demonstrated that can quantify the strength of the intermolecular interactions. As an example application, we demonstrate our platform's ability to characterize and differentiate specific and non-specific binding events.

INTRODUCTION

The ability to quantify the strength profile of the interbiomolecular bonds plays a significant role in various biomedical applications including biomarker detection, drug screening, and systems biology. Given the complex mechanisms and buffer conditions involved in determining the strength of the intermolecular bonds, development of a multiplexed force spectroscopy platform capable of probing a large number of interactions simultaneously and under the same buffer conditions is a necessity. The most common methods used to characterize such molecular interactions are atomic force microscopy, optical tweezers and magnetic tweezers. These techniques are limited in that they can only probe a single to a few interactions at a time. To this end, electronic-based actuation platforms are excellent candidates as they are scalable, addressable and provide precise control over applied voltage which can be programmed to exert various force profiles in order to characterize molecular interactions. The challenge with electrokinetic forces, such as dielectrophoresis lies in the inherent weakness of these forces (1-10 pN) compared to typical biomolecular interactions (~nN). Previously, we enhanced the strength of the dielectrophoresis (DEP) force by two orders of magnitude (referred to as ultra-DEP, uDEP) to overcome protein-protein interactions and to perform bead-based protein assays [1]. Here, we present a multiplexed uDEP-based platform that utilizes micron-sized beads as functional probes to examine intermolecular interactions and retrieve their force spectrums. As an example application, we demonstrate our platform's ability to characterize and differentiate specific and non-specific binding events.

RESULTS AND DISCUSSION

As shown in Figure 1, we patterned an array of proteins along a single microfluidic channel, where each element targets a secondary protein coated on micron-sized beads in the subsequently introduced sample. Below each element of the array, we have a pair of addressable interdigitated electrodes (IDEs). Through controlling the voltage applied at each IDE we can exert the desired force profiles on the beads and determine whether rupture of intermolecular bonds occurs (as evident by the bead detachment, observed optically). Here, we illustrate 16-plex actuation capability of our device to characterize the strength profile of anti-IgG/IgG (specific) and anti-IgG/IgM (non-specific) interactions. To this end, we functionalized the IDEs either with IgG or IgM, and used 2.8 μ m-diameter anti-IgG coated beads to perform our pulling assay.



Figure 1: Ultra-dielectrophoretic force spectroscopy array for probing intermolecular interactions. An array of uDEP electrodes are patterned, each functionalized with a different protein that targets a secondary protein coated on the micron-sized beads. Detachment percentage of protein-bound beads is quantified optically when ramping up the applied voltage at each of the IDE pairs one-by-one. Here, V_1 is ramped up and the subsequently produced uDEP force detaches the bound beads from the surface of the 1st interdigitated electrode pair (i.e. IDE 1). With no voltage applied at the other IDEs, the protein-bound beads on the respective elements remain attached to the surface.

The two orders of magnitude of enhancement in DEP force (to overcome these interactions) was achieved by fabricating high voltage tolerant electrodes, In particular, a pinhole free nanometerscale thin film oxide as a protective layer was deposited using atomic layer deposition. The uDEP electrodes were excited at sufficiently high frequency (1 MHz) to minimize the undesired voltage drop across the oxide film (Figure 2).



Figure 2: Voltage drop spectrum across the oxide capacitance at each electrode-buffer interface for various oxide thicknesses (simulated based on the developed circuit model).

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Representative four impedance spectrums of the IDEbuffer interface are shown in Figure 3 verifying that the resistive plateau region is achieved at > 1MHz (for 30× diluted PBS buffer).



Figure 3: Measured impedance spectrum of four representative IDEs (IDE 7 and 11 are functionalized with IgG1 and IDE 4 and 15 are functionalized with IgM).

To retrieve the force spectrum for each interaction, the applied voltage was ramped up incrementally at each IDE. As can be seen in Figure 4, the peak of the anti-IgG/IgM bead detachment occurred when 12.5 V applied, while about 15 to 17.5 V was required to detach anti-IgG/IgG beads. These results indicate our platform's promising capability in distinguishing between specific and non-specific bindings.



Figure 4: Post-processed (differential) and side-by-side micrographs of two representative IDEs, one originally coated with specifically bound beads (i.e. anti-IgG/IgG) and one originally coated with non-specifically bound beads (anti-IgG/IgM). In each frame the bright spots correspond to the detached beads.

The collective detachment profile when actuating anti-IgG/IgG-bound beads vs. anti-IgG/IgM-bound beads is shown in Figure 5. This multiplexed format enables performing force spectroscopy on multiple devices independently, in a single channel; minimizing inter-experiment variations that originate from disparities in buffer conditions and assay preparation steps.



Figure 5: Detachment percentage of protein-bound beads when ramping up the applied voltage at each of the IDE pairs one-byone. The results depict the collective detachment profile when actuating specifically- (anti-IgG/IgG) vs. non-specifically- (anti-IgG/IgM) bound beads using our 16-plex platform. The array format allows performing force spectroscopy multiple times while ensuring operation under the same buffer conditions and assay preparation steps (DEP force is proportional to the square of the applied voltage).

We envision a plethora of possibilities for performing rapid and inexpensive characterization of intermolecular affinities which is of utmost importance in studying various inter- and intracellular molecular pathways.

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A DEP-BASED LAB-ON-A-CHIP SYSTEM FOR THE DETECTION OF MULTIDRUG RESISTANCE IN K562 LEUKEMIA CELLS

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ABSTRACT

This study presents a DEP-based lab-on-a-chip (LOC) system for label-free detection of multidrug resistant (MDR) K562 leukemia cells in a cell mixture, consisting of red blood cells (RBCs) and MDR-K562 cells, for the first time in the literature. The system consists of 2 consecutive DEP units, one for the depletion of RBCs and the other for capturing of MDR-K562 cells. RBCs are depleted by 60% in the first unit. In the second unit, MDR-K562 cell detection is performed with 100% selectivity at a flow rate of 10 μ l/min and at 20 V_{pp} in a cell mixture, containing less than 25% MDR-K562 cells.

INTRODUCTION

Multidrug resistance (MDR) is a condition enabling a cancer cell to resist distinct drugs of a wide variety of structure and function targeted at eradicating the cell. In case of MDR, patients do not respond chemotherapy so MDR detection in early stages of cancer is critical to envisage the most appropriate treatment and to speed up the recovery period [1].

Resistance to drugs is effectuated by some different mechanisms: (1) Drug efflux by membrane-associated proteins (P-glycoprotein (P-gp) and multidrug resistance-associated protein (MRP)), (2) drug elimination by exocytosis, (3) changing of drug targets, including DNA topoisomerase II, (4) enhancement of detoxification of compounds by glutathione system, and (5) overexpression of human major vault protein [2]. Although there are sensitive methods for MDR detection, they require expensive laboratory equipment and trained personnel. Moreover, they are time consuming due to labeling procedures and cannot be applied for frequent use during prognosis, because of the side effects [1, 3]. Hence, a cost effective, label-free, and user friendly method is needed to provide effective diagnosis of MDR in cancer cells, without compromising the sensitivity and specificity.

Dielectrophoresis (DEP) is a MEMS-enabled technique used to manipulate particles based on different sizes and/or dielectric properties under non-uniform electric field. Recent studies show that DEP is a potential tool to be applied in rapid MDR detection in a label-free manner [1, 4-7]. It is reported that the cytoplasmic conductivity of MDR-K562 cells are 2.17 times higher than drug sensitive K562 cells, as analyzed by DEP collection spectra [4]. This variation is most probably associated with the overexpression of P-gp, modulating the volume-activated Cl⁻ channels [8]. Coley et al. also reported that human breast cancer cells (MCF7) and its MDR progenies (MCF7-TaxR, MCF7-DoxR and MCF7-MDR1) have different cytoplasmic conductivities (MCF7-TaxR<MCF7<MCF7-MDR1<MCF7-DoxR) [5]. We previously showed that imatinib resistant K562 cells could be trapped utilizing DEP, while the sensitive ones were not affected under the same experimental conditions [6]. We also reported that the same system is capable of detecting MDR in K562 cells at a flow rate of 10 μ l/min by applying 5 V_{pp}, with 100% selectivity in a mixture containing less than 1% resistant cells [1]. In addition, we also revealed that DEP can be utilized for the detection of the level of resistance in K562 cells without requiring the examination of P-gp dynamic functions [7].

In this study, an LOC system has been developed for the detection of MDR in K562 leukemia cells, by integrating a DEP-based RBC depletion unit with an MDR-K562 cell detection unit. Results showed that MDR detection in K562 cells was achieved with 100% selectivity, even the resistant cells constituted less than 25% of overall population.

THEORETICAL BACKGROUND

DEP Theory

DEP is an electrical particle manipulation technique defined as the relative motion of particles and medium under non-uniform electric field. The movement of particles is a result of the timeaveraged DEP force, which is defined for a homogenous spherical particle as [1]:

$$\langle F_{\text{DEP}} \rangle = 2\pi \varepsilon_{\text{m}} r^{3} \text{Re} (F_{\text{CM}}) \nabla E_{\text{rms}}^{2}$$
(1)

where, ε_m is the medium permittivity, r is the particle radius, $Re(f_{CM})$ is the real part of Clausius-Mossotti factor of the particle, and ∇E_{rms}^2 is the gradient of external electric field.

 f_{CM} carries information about the particle and the medium dielectric properties, and the frequency dependence of DEP force. For spherical particles, f_{CM} is formulized as [1]:

$$f_{CM} = \frac{\varepsilon_p^* - \varepsilon_m^*}{\varepsilon_p^* + 2\varepsilon_m^*} \tag{2}$$

where ε_p^* and ε_m^* are the complex permittivities of the particle and the suspending medium, respectively.

Complex permittivities of particle and medium are frequency dependent variables. Therefore, based on applied frequency, the relation between ε_p^* and ε_m^* changes. If $\varepsilon_p^* > \varepsilon_m^*$, particle is pulled towards stronger electric field region, observing positive DEP (pDEP) force. If $\varepsilon_p^* < \varepsilon_m^*$, particle is pushed towards weaker electric field region, observing negative DEP (nDEP) force. Utilizing Eq. 2, the frequency at which the particle observes no net DEP force (i.e., $Re(f_{CM})=0$) can be calculated. This frequency is named as crossover frequency (f_{cross}).

Particles with similar sizes but different f_{cross} values can be separated by pDEP or nDEP approach, at a certain signal frequency. If f_{cross} values of particles are not different enough for separation, size-based separation of particles can be achieved through DEP, due to the dependence of DEP force to particle radius (Eq. 1).

Cell Modeling

 $Re(f_{CM})$ depends on particle and medium dielectric properties. Medium properties can be attuned but particle dielectric properties are out of control and should be measured. After the

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measurement of dielectric properties of particles, they should be modeled to choose the proper structures for DEP operation.

Biological cell membrane and cytoplasm have different dielectric properties. These properties provide cell-specific characteristics as cells vary in their molecular content. This can be utilized as a discriminatory feature in DEP cell identification, manipulation or separation [1]. In this study, single shell cell modeling was utilized. Effective complex permittivity (Eq. 3) is obtained with this technique and it is used instead of particle complex permittivity ε_p^* in the expression of f_{CM} (Eq. 2) [1]. $Re(f_{CM})$ vs frequency characteristics of RBCs, drug sensitive and MDR-K562 cells were obtained in MATLAB (Fig. 1).

$$\varepsilon_{eff,p}^{*} = \varepsilon_{s}^{*} \left\{ \frac{[(r+d)/r]^{3} + 2[(\varepsilon_{i}^{*} - \varepsilon_{s}^{*})/\varepsilon_{i}^{*} + 2\varepsilon_{s}^{*}]}{[(r+d)/r]^{3} - [(\varepsilon_{i}^{*} - \varepsilon_{s}^{*})/\varepsilon_{i}^{*} + 2\varepsilon_{s}^{*}]} \right\}$$
(3)

where, d is the thickness of the cell membrane, ε_i^* and ε_s^* are the cell interior and shell complex permittivities, respectively.



Figure 1: $Re(f_{CM})$ vs. frequency graphs of RBCs, drug sensitive and MDR-K562 cells in a medium with 2.5 mS/m conductivity and 78 permittivity coefficient.

According to this simulation, f_{cross} of sensitive K562 cells was specified as 48.64 MHz in a medium with the conductivity of 2.5 mS/m as working frequency of MDR detection unit [1]. $Re(f_{CM})$ of MDR-K562 cells and RBCs were positive at that frequency and they should be pulled to higher electric field region through pDEP force, having different magnitudes. It should be distinguished that $Re(f_{CM})$ of MDR-K562 cells (point A) were 1.52 times higher than RBCs (point B) at f_{cross} of sensitive K562 cells.

DESIGN AND FABRICATION

The device schematic was presented in Figure 2. RBC depletion unit is composed of a main channel (1000 μ m W x 20

 μm H) and side channels (500 μm W x 20 μm H) connected to each other via 8 μm filtering gaps on the sidewalls to realize size-based filtration.

Planar electrodes with 15° angles were implemented throughout the channel to direct the cells through sidewall filtering gaps via DEP force. MDR detection unit has 2x29 3D-electrode arrays, with 40 μ m width and 15 μ m gaps in between, placed on the sidewalls of 300 μ m x 20 μ m parylene microchannel. The channel also contains parylene posts for hydrodynamic focusing of cells to DEP traps. Isolation of both planar and 3D-electrodes from the solution is provided by means of thin parylene coating, reducing the *Joule* heating and cell damaging.

In RBC depletion unit, all cells are directed towards the sidewalls by sliding on the electrodes through pDEP force and hydrodynamic forces, at a frequency of 1 MHz. Small RBCs pass through the filters, while cancer cells continue through the main channel.

Based on the dielectric simulations, f_{cross} of sensitive K562 cells (48.64 MHz) was chosen as working frequency of MDR detection unit [1]. This unit works based on electrohydrodynamic principles. Utilizing Newton's second law in the microchannel and assuming electroosmotic force and acceleration term as zero [9], the following inequality is obtained (Eq. 4), indicating that size and $Re(f_{CM})$ of particles determine their trapping conditions under the same hydrodynamic settings.

$$2\pi\varepsilon_{\rm m}r^3 \operatorname{Re}\left(F_{\rm CM}\right)\nabla E_{\rm rms}^2 > 6\pi\mu r v_m \tag{4}$$

where, v_m is the velocity of medium and. μ denotes the medium viscosity.

In this study, the average radius of drug sensitive and MDR-K562 cells were 5.5 μ m while RBCs were 3 μ m in radius, determined by pixel measurement software (*Pixel Ruler*). Moreover, as it is shown in Figure 1, $Re(f_{CM})$ of MDR-K562 cells were 1.52 times higher than RBCs. This implies that the DEP trapping force on MDR-K562 cells is 9.36 times higher than that of RBCs. If flow rate is optimized, the detection of MDR-K562 cells can be achieved in MDR detection unit with high selectivity.

Figure 3 and 4 show the fabrication flow and the fabricated device, respectively. Fabrication was started with dehydration of the 6" glass wafers which were cleaned with piranha and etched with BHF. Ti/Au was sputtered (*BESTEC dual chamber sputter system*) to form planar electrodes and a seed layer for Cuelectroplating. Utilizing wet etching, planar electrodes and the seed layer were patterned. To prevent parasitic capacitances between Au layer (used to short nonconsecutive electrodes) and 3D-electrodes, a parylene layer (~0.5 μ m) was coated (*Parylene deposition system*, SCS 2010 LabcoterTM 2).



Figure 2: Device schematic. The system consists of 2 consecutive DEP units for the depletion of RBCs and capturing of MDR-K562 cells.

3D-electrode places were patterned with parylene RIE (*STS Multiplex RIE system*). Cu-electroplating was carried out to obtain 20 μ m thick 3D-electrodes. Another parylene layer (~0.5 μ m) was coated for insulation purposes. Next, the channel and parylene obstacles were formed with lithography and parylene coating (~20 μ m). Finally, sacrificial photoresist was released with acetone.



Figure 3: Fabrication flow.



Figure 4: Fabricated device.

EXPERIMENTAL Materials

The Philadelphia chromosome positive K562 cell lines were obtained from German Collection of Microorganisms and Cell Cultures, Germany, and cultured in RPMI 1640 medium (Invitrogen) with fetal bovine serum and penicillin-streptomycin. To develop resistance, doxorubicin (Saba) were added to different culture medium with gradual increment in their concentration until they become resistant to 1000 nM doxorubicin [10]. Whole blood (3 mL) was sampled from a healthy adult volunteer (woman) through venipuncture, utilizing EDTA treated tube to prevent

coagulation. This sample was centrifuged at 900 rpm for 15 min. RBCs were harvested from the pellet.

Test Setup and Procedure

Tests were conducted under a fluorescent microscope (*Olympus, SZX12*). Observations were carried out under 20X magnification. A high-speed camera (*Evolve 128*) was utilized to observe the cell movement inside microchannel, under continuous flow and low fluorescence intensity. Flow rate was adjusted with a computer controlled microsyringe pump (*Lab Smith*). Real-time processing of screenshots was carried out with the computer software, *Winfluor*. DEP force was generated by energizing the electrode arrays with a signal generator (*Agilent, 81150A*). Two of its four outputs were utilized with 180° phase difference. Figure 5 shows test setup.

Cell preparation and test procedure are as follows. MDR-K562 cells and RBCs were centrifuged at 1000 rpm for 5 min and 900 rpm for 15 min, respectively. The pellets were washed twice and suspended in an isotonic medium (8.5 % (w/v) sucrose and 0.3 % (w/v) dextrose) with a conductivity of 2.5 mS/m. These medium properties were adapted from the study of Labeed *et al.* [4]. For monitoring purposes, RBCs were stained with cell tracker red (Invitrogen), a fluorescent dye with red color, and observed under red filter (*31002 TRITC, Chroma*). MDR-K562 cells were stained with fluorescein diacetate (Invitrogen), a fluorescent dye with green color, and observed under GFP filter. After 25 minutes incubation, cells were washed with the suspension medium. Finally, they were mixed and working cell mixture was obtained. The ratio of MDR-K562 cells and RBCs were adjusted as 2, 0.3, 0.1, 0.05, keeping MDR-K562 cell number constant.



Figure 5: Test setup.

RESULTS AND DISCUSSION

Detection tests were performed with 20 V_{pp} at a flow rate of 10 µl/min. Figure 6 shows the screenshots of trapping tests of MDR-K562 cells for different cell ratios (video presentation is available). Table 1 presents the ratio of trapped MDR-K562 cells to RBCs. This ratio is infinite for the condition that MDR-K562 cells constitute less than 25% of overall population. This implies that MDR detection in K562 cells is achieved with 100% selectivity.



Figure 6: Pseudo-colored images taken during detection of MDR-K562 cells, green screens. Red screens show RBCs. Working solution was the mixture of MDR-K562 cells and RBCs with different concentrations (f=1 MHz and f=48.64 MHz for depletion and detection units, respectively. Flow rate = 10 µl/min, voltage = 20 V_{pp}).

On the other hand, increase in the number of RBCs was ensued with the trapping of them in MDR detection unit since RBC depletion unit was saturated and the harmony between electrical and hydrodynamic conditions was corrupted. This challenge can be overcome by increasing the length of RBC depletion unit.

Table 1: The ratio of trapped MDR-K562 cells to trapped RBCs for 4 cell mixtures, injected into channel with different ratios of MDR-K562 cells to RBCs.

The ratio of MDR-K562 to RBCs in inlet	2	0.3	0.1	0.05
The ratio of trapped MDR-K562 to RBCs	8	∞	4	1.75

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A FLEXIBLE, LARGE-SCALE DIAMOND-POLYMER CHEMICAL SENSOR FOR NEUROTRANSMITTER DETECTION

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ABSTRACT

This paper reports a flexible, diamond-polymer chemical sensor with three microelectrodes, which features a wide aqueous potential window and low background current from diamond and mechanical flexibility from the polymer. An improved wafer transfer process is developed to transfer boron-doped polycrystalline diamond (BDD) onto a thin Parylene-C substrate with good uniformity, strong adhesion, and high yield. The as-fabricated sensor shows a much wider potential window compared to an Au electrode, and a sensitivity of ~0.018mA/mm²·mM through cyclic voltammetry for Ru(NH₃)₆ ^{2+/3+} and 0.0012 μ A/mm²· μ M through chronoamperometry for dopamine (DA).

INTRODUCTION

The central nervous system (CNS) consists of large networks of neurons that communicate with each other via electrical impulses (action potentials) along the axon and chemicals at the synapse between neuron-to-neuron. Dysregulated neurotransmitter transmission has been linked to several neurological disorders, such as Schizophrenia, Huntington's disease, Alzheimer's disease and Parkinson's disease [1], [2]. Used as a molecular diagnosis for Parkinson's Disease, dopamine (DA) is one of the most important neurotransmitters [3]. Real-time physiological monitoring of the concentration of DA will allow researchers to better understand brain diseases and evaluate the efficacy of the therapeutics toward Parkinson's disease [3].

An ideal implanted neural chemical sensor for effective sensing of DA should possess the following requirements: resistance to surface biofouling, high sensitivity, biocompatibility, a wide working potential window, mechanical flexibility, and long-term stability and reliability. Boron-doped polycrystalline diamond (BDD) has shown excellent electrochemical properties in these requirements. Unlike carbon fiber, which is composed almost exclusively of sp² carbon, requires time-consuming surface pretreatment and suffers from surface molecular adsorption, BDD shows excellent resistance to surface fouling and needs less/no pretreatment for surface activation due to sp³ carbon [4]. Besides, BDD has relative small double-layer capacitances (ranging from 5 to 40µF/cm²) [5], which can minimize non-Faradaic current (background noise) and achieve a higher signal-to-noise ratio. The biocompatibility has been demonstrated and is comparable with titanium and 315 stainless steel [6] by observing a very small amount of adsorbed and denatured fibrinogen on the diamond surface, which is used as a biocompatibility indicator [6]. In addition, BDD has a wide potential window range from 2.27V to 3.3V in an aqueous environment [7], [8] and chemical inertness [9]. However, BDD is a rigid material with a Young's modulus of $\sim 1000 \times 10^{9}$ Pa [10], which is several orders of magnitude higher than that of brain tissue (a Young's modulus ranges from $\sim 10^3$ to 10^5 Pa [11]). Such a mechanical property mismatch causes tissue irritation and damage during long-term application, which is an obstacle for the application of biochemical sensors. Additionally, BDD cannot be synthesized onto polymer substrates directly due to its high synthesis temperatures of 500-900°C. Considering these limitations, previously, we developed a microfabrication process to transfer

BDD onto Parylene-C substrates [12] (with a Young's modules of 2.8×10^9 Pa [13]), where Parylene anchors were introduced to increase the bonding strength between Parylene and BDD. The BDD was released by etching silicon (Si) using potassium hydroxide (KOH) with a custom-made jig. The capability of powering up a µLED was also demonstrated and intended for use in optogenetics, which uses light to manipulate electrophysiological activities of neurons. In this paper, we improved this wafer transfer process by replacing wet KOH etching with deep-reactive-ion dry etching (DRIE) to achieve higher yield, better uniformity and adhesion. The application of this paper is dedicated to neural chemical sensing, especially, to DA detection.

The concept diagram is shown in Figure 1. A 3-electrode BDD (in black) chemical sensor fabricated on a Parylene substrate (in gray) with a reference electrode (RE), working electrode (WE) and counter electrode (CE), is used to demonstrate the proposed fabrication method. The openings $(30\mu m \times 50\mu m)$, in white) encircled in green dash lines are created on large BDD patterns to make Parylene anchors. The overall design dimensions of the sensor are 4mm×4.7mm, and the effective surface areas of WE, CE and RE are $0.8mm^2$, $0.48mm^2$, and $0.48mm^2$, respectively.



Figure 1: Concept diagram of flexible BDD chemical sensors with design dimensions.

FABRICATION METHOD

Figure 2 shows a detailed process flow. (a) One micron (1µm) sacrificial SiO₂ was deposited on a 3 inch Si wafer using plasma enhanced chemical vapor deposition (PECVD) (PlasmaLab 80plus®, Oxford Instruments), followed by synthesis of a ~2.7μm microcrystalline BDD film using a custom-made microwave plasma assisted chemical vapor deposition reactor (MPACVD) with a gas mixture of hydrogen diborane and methane. (b) A 1.3µm thick aluminum hard mask for diamond etching was deposited using a sputtering system (Denton Desk Top Pro Sputtering System, Denton Vacuum, Inc). (c) The aluminum layer was patterned using standard ultraviolet (UV) photolithography and etched in the aluminum etchant (Type A), followed by plasma etching in an electro cyclotron resonance reactive ion etcher (ECR-RIE) (Lambda Technologies, Inc.) using SF₆/Ar/O₂ as processing gases, to pattern the BDD film. Then the aluminum hard mask was stripped in the aluminum etchant. (d) SiO₂ was over-etched in buffered oxide etchant (BOE) to create undercuts for Parylene anchors. (e) The wafer was treated with Silane A174 adhesion promoter (Sigma Aldrich, Inc) to increase the adhesion between Parylene and BDD,

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Figure 2: Device fabrication and BDD transfer processes: (a) $1\mu m SiO_2$ deposition on a 3 inch silicon wafer using PECVD and 2.7 μm BDD synthesis using MPACVD; (b) 1.3 μm Al deposition as hard mask using DC sputtering system; (c) Al hard mask patterned using photolithography and BDD etched using ECR-RIE; (d) SiO₂ undercut in BOE; (e) 17 μm Parylene-C conformal coating using CVD; (f) Backside Parylene-C removal using plasma etching; (g) Silicon etching in DRIE; (h) SiO₂ removal in BOE.

followed by a ~17 μ m conformal Parylene-C coating (PDS 2010, Specialty Coating System, Inc). At this point, Parylene anchors were formed and wrap around BDD patterns. (f) The backside Parylene was etched in O₂ plasma (PX-250 plasma system, Nordson March, Inc.). (g) The front-side of the wafer was bonded onto a carrier wafer using polyphenyl ether and the silicon substrate was completely etched from the backside using deep reactive-ion etching (DRIE). Then the BDD-Parylene layer was released from the carrier wafer with acetone soak, followed by isopropanol (IPA) and deionized water (DI water) rinse. Finally, (h) the sacrificial SiO₂ was etched completely in BOE solution.

DEVICE CHARACTERISTICS

Figure 3 (a) shows an as-fabricated BDD-Parylene sensor sitting on a penny with the left branch labeled as RE, the middle branch as WE, and the right branch as CE. The sensor layout is symmetrical to the central line, which means RE and CE have the same size. The mesh structures correspond to the openings shown in Figure 1, which enable more Parylene anchors over large surfaces to significantly enhance the adhesion between BDD and Parylene-C. Figure 3 (b) demonstrates the mechanical flexibility of the transferred BDD-Parylene film by wrapping the device on a punch



Figure 3: (a) A fabricated BDD-Parylene sensor with three electrodes. (b) A device wrapped on a 3.5mm-diameter punch tip, demonstrating its flexibility.

tip with 3.5mm in diameter.

Figure 4 (a) shows a scanning electron microscopy (SEM) image of the Parylene anchors using a low vacuum mode SEM (6610V, JEOL, Inc) with BDD in gray and Parylene in bright white. Parylene anchors which wrap around the BDD patterns are clearly observed as indicated in Figure 4 (a). The Raman spectrum taken from the nucleation side of BDD is shown in Figure 4 (b), where characteristic diamond band (1332cm⁻¹) decreases and widens and two new boron bands appear at ~500cm⁻¹ and ~1210cm⁻¹. The Raman features are comparable to heavily doped BDD thin films [14], where boron doping concentration is estimated at the order of 10^{20} cm⁻³. Energy dispersive X-ray spectrometry (EDS) was performed on the surface of BDD to confirm the complete removal of SiO₂, as shown in Figure 4 (c), which otherwise causes high contact resistance and desensitizes sensing performance.



Figure 4: (a) Scanning electron microscope images show the mesh structure and Parylene anchors. (b) A Raman spectrum shows both the boron and diamond bands from the BDD nucleation side. (c) An EDS image shows only the carbon peak, implying no SiO₂ residue was detected.

The strong adhesion between BDD and Parylene was verified by "Scotch tape" testing, as shown in Figure 5. In Figure 5 (a), the BDD side of the sensor was first attached to a Scotch tape and pressed to make sure the whole pattern was in good contact with the tape. Then, the BDD sensor was peeled off from the Scotch tape (Figure 5 (b)). No BDD delamination was observed after five peelings.



Figure 5: Scotch tape testing shows: (a) a BDD-Parylene sensor attached on to a piece of tape, with the BDD side facing down, and pressed; (b) a clean tape surface after peeling off the sensor from the tape.



Figure 6: (a) Voltammograms of Au, Pt, BDD electrodes in $0.1M H_2SO_4$ vs. Ag/AgCl. The scan rates are 0.1V/s. (b) CV curves of the BDD sensor in various concentrations of $Ru(NH_3)_6^{2+/3+}$ with 1.0M KCl solution vs. BDD. The scan rate is 30V/s. (c) Fitting curve of the cathodic peak current versus the concentrations of $Ru(NH_3)_6^{2+/3+}$ (d) Voltammograms of the BDD sensor in various concentrations of DA with 0.1M PBS vs. BDD. The scan rates are 1.0V/s. (e) Chronoamperograms of various concentrations of DA in PBS buffer solution vs. BDD at an applied potential of 1.0V. (f) Fitting curve of the background corrected current versus the concentrations of DA.

CHEMICAL SENSING TESTING

To demostrate the low background level and wide potential window of the BDD, a standard gold electrode (Au) and a platinum electrode (Pt) are used as comparisons. Cyclic voltammetry (CV) was conducted in 0.1M sulfuric acid (H_2SO_4) solution vs Ag/AgCl with a scan rate of 0.1V/s as shown in Figure 6 (a). The BDD electrode shows low and featureless background current from -0.8 V to 2V. The Au electrode shows reduction current at 0.8V and oxidation current at potentials higher than 1.4V. The Pt electrode shows a reduction current at 0.4V and strong oxidation current at potentials higher than 1.5V. This demonstrates that the BDD electrode possesses lower background current and a wider potential window than the Au and Pt electrodes.

 ${\rm Ru(NH_3)_6}^{2+/3+}$ is commonly used in electrochemistry to evaluate the reactivity of electrodes. It is classified as an outer-sphere redox couple, which is insensitive to the electrode surface microstructure, adsorption, and oxides [16]. In this case, the electron transfer rate is mainly affected by electronic properties of the electrode. Cyclic voltammetry was performed in various concentrations of Ru(NH₃)₆^{2+/3} in 1.0 M KCL solution using BDD with a scan rate of 30V/s, as shown in Figure 6 (b). The well-defined oxidation and reduction peaks were observed. The peak separation is 59mV for reversible electron transfer systems [15]. However, our BDD sensor exhibits higher peak separation values of ~400mV, which suggests possible IR drop in the system or low electron transfer kinetics [17]. The cathodic peak currents versus various concentration of Ru(NH₃)₆^{2+/3+} were measured in Figure 6 (c), which implies a good linearity of the sensor response to Ru(NH₃)₆^{2+/3+} with an R² value of 0.999.

Dopamine (DA) can be detected electrochemically according to the reaction in Figure 7, where DA is oxidized to Dopamine-o-quinone [18]. In the CV measurements (CHI 604E), the BDD WE was used to detect various concentrations of DA in 0.1M pH=7 phosphate buffer solution (PBS) vs. BDD, with a scan rate of 1.0 V/s, as shown in Figure 6 (d). Unlike Ru(NH₃)₆^{2+/3+}, where faster electron transfers were observed and the system was under mass transfer control by showing a peak-type voltammogram, DA detection had a relative sluggish electron transfer process with no peak observed. Chronoamperometry was conducted at a single step with a constant applied potential of 1.0V, and measured current decayed exponentially with time. Chronoamperograms of various concentrations of DA in 0.1M pH=7 PBS buffer vs. BDD is shown in Figure 6 (e). Background corrected current, which is the steady state current of chronoamperograms subtracting the current of background PBS buffer solution, versus different concentrations of DA is plotted in Figure 6 (f), where a linear response of the BDD sensor to DA was observed with an R² value of 0.993.



Figure 7. Dopamine / Dopamine-o-quinone redox.

CONCLUSIONS

In summary, we reported a flexible, diamond-polymer chemical sensor with three microelectrodes. An improved wafer transfer process of replacing wet KOH etching, as we reported previously, with deep-reactive-ion dry etching (DRIE) is used to transfer boron-doped polycrystalline diamond (BDD) onto a thin Parylene-C substrate with good uniformity, strong adhesion, and high yield. The flexibility of the as-fabricated BDD sensor was demonstrated by wrapping the sensor onto the tip of a micro punch. The Parylene anchors were observed clearly from an SEM image. The bonding strength was testified using a "Scotch tape" method. After five cycles of attaching, pressing and peelings, no delamination between Parylene and BDD was observed, which implies the efficacy of the Parylene anchors. Raman spectroscopy showed a typical heavily doped diamond spectrum with the diamond characteristic band at 1327cm⁻¹ and the two boron bands at 505cm⁻¹ and 1209cm⁻¹. The chemical performance of such a flexible BDD sensor was first reported. A wide aqueous potential window and a lower background current in the 0.1M H₂SO₄ was observed as compared to a commonly used gold electrode. A faster electron transfer process under mass transfer control was observed in Ru(NH₃)₆^{2+/3+} redox and a sluggish electron transfer process was observed for Dopamine/Dopamine-o-quinone redox. A sensitivity of ~0.018mA/mm²·mM for Ru(NH₃)₆^{2+/3+} and 0.0012µA/mm²·mM for DA was measured experimentally.

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A PARYLENE PERIPHERAL NERVE CUFF ELECTRODE WITH INTEGRATED MICROFLUIDICS

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ABSTRACT

We present a novel micromachined polymer peripheral nerve interface that combines both electrodes and microfluidic channels in an adjustable cuff sized to interface with rat sciatic nerve. Parylene fluidic channels enable targeted delivery of lysing agents and neurotrophic factors to fascicle sites on the nerve surface, in order to induce axonal sprouting towards electrodes. This Lyse-and-Attract Cuff Electrode (LACE) uses a simple locking mechanism that is adjustable for close contact with nerves of varying diameters. We demonstrate fully functional cuffs having electrode impedances of $< 2 \ k\Omega$ (1 kHz) and controlled microfluidic infusion at $< \mu L/min$ flow rates.

INTRODUCTION

Neural recording and stimulation technologies are needed to interface with peripheral nervous system (PNS) to create a bidirectional link between the nervous system and artificial devices (prosthesis) for restoration of sensorimotor function (Figure 1) [1-4]. Electrical interfaces to the peripheral nerves are either extraneural or intrafascicular/intraneural. Minimally invasive extraneural interfaces are placed on the circumference of the nerve which is surrounded by a collagenous sheath (epineurium) that limits access to individual nerve fibers. Improved access to individual fascicles has been achieved by extraneural interfaces that reshape the nerve by flattening it, as is the case of the previously described flat interface nerve electrode (FINE) [5]. However the signal to noise ratio is still restricted by the impedance of the epineurium, and nerve damage can be induced by reshaping.

Intrafascicular/intraneural electrodes gain greater access to individual nerve fibers, improving signal-to-noise ratio, and can selectively target small groups of axons, but are significantly more invasive [6, 7]. Electrodes may either be inserted longitudinally into the nerve and run parallel to nerve fibers, or transversely and run perpendicular to fibers. Of the two methods, transverse electrodes are associated with greater risk of nerve damage. Invasively inserted electrodes are fabricated from materials with elastic moduli several orders of magnitude greater than that of nerve, contributing to chronic tissue response and the degraded recording/stimulation stability over time.

Neither approach has been able to achieve the desired selectivity while minimizing invasiveness, and all prior attempts suffered from a lack of chronic reliability. To overcome these hurdles and achieve a long-term stable interface with high fidelity recordings, we developed a non-invasive approach in which a polymer cuff electrode targets individual fascicles within a nerve by inducing the sprouting of axonal collaterals towards electrodes embedded in the microfluidic channels of the cuff. Axonal sprouting will be selectively induced from the fascicles closest to the microfluidic outlet ports via localized delivery of lysing agents to temporarily disrupt the epineurium, followed by delivery of neurotrophic factors to promote axonal sprouting towards electrodes within the microchannels [8-11]. The design calls for a cuff with adjustable diameter to ensure localized diffusion of chemicals into the epineurium of nerves of varving diameters. This Lyse-and-Attract Cuff Electrode (LACE) aims to increase fascicular selectivity and signal-to-noise ratio for recording and/or stimulation

without traumatic invasiveness, while providing long-term stability beyond the current state of the art.



Figure 1: Peripheral nerve interfaces necessary for braincontrolled prostheses.

DESIGN

The LACE is fabricated from thin-film biocompatible polymer Parylene C (Figure 2), and incorporates 4 surface micromachined microfluidic channels (250 μ m W × 20 μ m H) for targeting four fascicles in a rat sciatic nerve. Each microfluidic channel contains a pair of Pt electrodes (300 μ m W × 1500 μ m L) for differential recording and/or stimulation. The cuff wraps around a nerve with the microfluidic outlets in direct contact with the circumference of the epineurium, and is held in position by etched serrations (Figure 2). The cuff is adjustable (1.1-1.5 mm diameter, 0.1 mm increments) to provide close fits to nerves of varying diameter. Ancillary components identified in Figure 2b include a port for external fluidic connection, Parylene ribbon cable (~2 cm long) for external electrical connection, and a reference electrode external to the microfluidic channels.



Figure 2: (a) Schematic of LACE for targeting individual fascicles within a nerve. Insert shows the cross-sectional view. (b) Fabricated LACE in which microfluidic channels are highlighted by the presence of photoresist.

Fabrication

Devices were fabricated on a silicon wafer for mechanical support during the microfabrication process (Figure 3). A 10 μ m Parylene base layer was first deposited by chemical vapor deposition. Sputtered Pt electrodes and leads (2000 Å) were then patterned by a liftoff process with negative photoresist AZ 5214 E-IR. A 10 μ m Parylene insulation layer was then deposited and selectively plasma-etched to expose electrodes and contact pads. Sacrificial photoresist AZ 4620 was spun to a height of 20 μ m and patterned to form the microfluidic channels. An 8 μ m Parylene layer

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was then deposited over the photoresist. Oxygen plasma etching created openings for microfluidic ports as well as the device cutout. Devices were released from the substrate and sacrificial photoresist was removed with an acetone soak. No stiction of Parylene structures was observed.



Figure 3: LACE fabrication process which utilizes standard surface micromachining techniques for Parylene.

Device level fluidic packaging consisted of a custom made polyurethane (PU) catheter (SAI Infusion Technologies) secured in place with MED 4210 silicone adhesive (Factor II Incorporated). A zero-insertion-force connector (ZIF, part no. 0514411072 Molex Inc) was used to establish reversible electrical connections to the cuff electrodes through the contact pads at the end of the integrated Parylene ribbon cable (Figure 4). The ZIF connector was mated to a flat flexible cable (FFC, part no. 050R10-102B, Parlex USA LLC) and a rigid PCB for external connection to the electrophysiological recording setup.



Figure 4: Cuff electrode with integrated Parylene cable is attached to a zero insertion force (ZIF) connector.

METHODS

Interlocking Mechanism

The cuff structure consisted of an elongated tapered tab that loops around the sciatic nerve, inserts into an etched slit and locks in place using etched serrated teeth (Figure 5a-c). A two-part locking mechanism was used to maintain the loop around the nerve, and includes an additional slit through which the tab is threaded to create a secure buckle structure (Figure 5d). The guide needle hole was utilized to pass a suture from the circular hole in the tab and facilitate guiding of the tab into the slits. Devices were wrapped and unwrapped around simulated agarose nerve phantoms and holding strength was evaluated under gentle handling conditions (n > 10). An agarose concentration of 1.37 % wt/wt in water was selected since it has a compressive modulus equal to that of rabbit sciatic nerve [12, 13].



Figure 5: Prototype LACE interlocking mechanism test. (a) Flat LACE, (b-c) tab threaded through first slit and kept in place by serrated teeth, and (d) tab secured by threading through second slit to form the buckle locked structure.

Microfluidic Channel Evaluation

Evaluation of the microfluidic channels and fluidic connection was conducted by driving colored dye through the channels using an infusion pump (Harvard apparatus Infuse/Withdraw PHD 2000) and imaging the progression of the dye with a microscope (Caltex Scientific HD60T). The LACE device was submerged in distilled water under the microscope, and primed by flushing the channels with isopropyl alcohol and distilled water for 5 minutes each. With the LACE device in a flat or curled orientation, a solution of dilute green dye was infused into the LACE device at varied flow rates ranging from 5 to 2000 nL/min.

Maximum possible flow rate of the LACE device was determined by pumping dye through a LACE device wrapped around a nerve phantom, and observing which flow rates induced leaks or structural failure. Flow rates were increased from 300 nL/min at increments of 100 nL/minute. Between each increment, the device was uncurled and infused with green dye in a flat orientation under the microscope in order to detect any small leaks that may have formed in the interior of the channels, then re-curled for the next flow rate.

Localization of fluid delivery was demonstrated by locking a LACE device around a simulated sciatic nerve phantom, then driving sodium fluorescein dye (Sigma F6377) through the microfluidic channels using the infusion method previously described at a flow rate of 50 nL/min.

Electrode Evaluation

Cyclic voltammetry (CV) and electrochemical impedance spectroscopy (EIS) were used to assess the stimulation and recording capabilities respectively of the cuff electrodes. Electrochemical characterization of the electrodes was performed with a Gamry Reference 600 potentiostat (Gamry Instruments, Warminster, PA). Prior to EIS measurement, CV was used to electrochemically clean the electrode surface and take initial measurements. LACE devices were immersed in 0.05 M H₂SO₄ with a constant stream of N₂ in a three-electrode cell. The working electrode was cycled between -0.2 to 1.2 V with respect to an Ag/AgCl (3M NaCl) reference, a potential range that is within the water window [14]. A 1 cm² Pt plate served as a counter electrode. Using a scan rate of 250 mV/s, each electrode was cycled for 30 cycles. EIS was performed in phosphate buffer saline $(1 \times PBS)$ at room temperature with an AC perturbation signal of 10 mV (rms) in the frequency range of 1-10⁵ Hz. As with CV, an Ag/AgCl (3M NaCl) reference and 1 cm² Pt plate counter were used.

RESULTS

Interlocking Mechanism

The locking mechanism was simple to implement and robust. A suture needle facilitated wrapping the device around an agarose nerve phantom of 1.2 mm diameter. The LACE was able to wrap tightly around the phantom without sliding of the device or damage to the simulated nerve. Once locked in place, the LACE did not disengage during fluidic and electrical testing, evidence of a secure lock. No damage to the cuff was observed despite repeatedly engaging and releasing the lock and curling and uncurling the LACE around the nerve phantom.

Microfluidic Channel Evaluation

No leakage or delamination was observed up to a flow rate of 2000 nL/min, with uniform flow through all fluidic channels and outlet ports in the flat configuration (n = 4). Figure 6a displays a sequence of images showing simultaneous and equally distributed flow out of all four outlet ports in the flat orientation. Image analysis confirmed that flow rate through channels was uniform within 6% standard error. In the curled configuration, flow was achieved in 3 out of 4 channels which is sufficient for the *in vivo* application (Figure 6b). Flow through the distal channel was blocked by pinching of the horizontal feeder channel and can be mitigated by channel design changes such as: reduced feeder channel width and incorporation of Parylene support walls along the horizontal channel [15, 16]. These changes have been incorporated and are being evaluated.



Figure 6: Sequential photographs of the infusion experiment at 830 nL/min flow rate in a (a) flat device and (b) curled device. Image analysis using ImageJ software confirmed that flow rate through channels was uniform within 6% standard error.

During maximum flow rate testing, microfluidic channel inlet failure occurred at a rate of 1000 nL/min. The leak appeared to be from delamination between Parylene layers. A safe flow rate of 900 nL/min can be achieved by the LACE device, which greatly exceeds the typical epineural space drug delivery rate of 17 nL/min that will be employed in our studies [17]. Localized drug delivery was achieved with the LACE as shown in Figure 7. In on-going testing the agarose nerve will then be imaged using a fluorescent microscope (Nikon LV100; mercury 100 W lamp, Chiu Technical Corporation; B-2A filter cube) and digitally processed in ImageJ in order to correlate fluorescent signal with dye concentration.

Electrode Evaluation

The expected standard voltammogram for Pt immersed in sulfuric acid was achieved for all electrodes. Distinct peaks of current in the cyclic voltammogram result from the process of hydrogen atoms adsorption to and desorption from the electrode surface were observed as shown in Figure 8. These peaks can be integrated with respect to the scan rate to calculate the electroactive surface area (ESA). In equation (1), Q_H is the hydrogen desorption charged measured via CV and 210 μ C/cm² is the value for the characteristic charge density associated with the monolayer of hydrogen atoms adsorbed to polycrystalline Pt [18]. Stimulation

electrodes can be characterized by their cathodal charge storage capacity (CSC), which is calculated from the time integral of the cathodic current in a cyclic voltammogram per unit geometric surface area (GSA) [19] as shown in equation (2).

$$ESA = \frac{Q_H}{210\,\mu C_{cm^2}}\tag{1}$$

$$CSC = \frac{Q_{cathodic}}{GSA}$$
(2)

The cyclic voltammogram obtained for all electrodes tested indicated a clean electrode surface. The ESA calculated was 635,714 μ m², which is different from the GSA of 450,000 μ m² due to surface topology. The CSC was 1.06 ± 0.06 mC/ cm² and the roughness factor, defined as the ratio of ESA to GSA was 1.4 (n = 6). No bubble generation was observed during the CV, which is important for safe electrical stimulation.



Figure 7: 1.37% (w/w) agarose nerve phantom after localization testing with fluorescein dye.



Figure 8: Cyclic voltammetry in H_2SO_4 of single recording site yielded standard Pt electrode characteristics.

EIS measurements on cuff electrodes yielded an electrical impedance of $1.8 \pm 0.11 \text{ k}\Omega$ and a phase of $-56.4 \pm 1.0^{\circ}$ at 1 kHz demonstrating low variation between electrodes (n = 6). A representative EIS measurement for a single electrode site post CV-clean is shown in Figure 9. Recorded impedances are expected to provide adequate neural recording potential. Baseline EC measurements on cuff electrodes demonstrated suitable electrode properties for peripheral nerve recordings and stimulation.

CONCLUSION

We developed a peripheral nerve interface that combines both electrodes and microfluidic channels in an adjustable cuff sized to interface with rat sciatic nerve. The LACE device uses a robust and simple locking mechanism that is adjustable for close contact with nerves of varying diameters. Parylene C was selected as the structural material for its biocompatibility and improved mechanical matching to tissue.

We demonstrated fully functional cuffs having low electrode impedances of $< 2 \ k\Omega$ (1 kHz) to help reduce susceptibility to noise pickup from sources such as muscle outside the cuff or recording equipment [20]. Controlled microfluidic infusion at $< \mu L/min$ flow rates and localized drug delivery was achieved, which is important to generate specific drug gradients necessary for guiding the axons into the microchannels. Future work entails characterization of modified fluidic channels to allow uniform flow through all channels in the curled orientation in order to target each fascicle inside the sciatic nerve, long-term characterization of electrodes, and preparation for *in vivo* animal testing.



Figure 9: EIS in PBS of single recording site showed a low impedance (< $2 k\Omega$) at 1 kHz.

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BIODEGRADABLE ELECTRICAL INTERCONNECTS FOR TRANSIENT IMPLANTABLE SYSTEMS

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ABSTRACT

This study presents the development and characterization of biodegradable electrical interconnects comprising biodegradable conductive polymer composites for use in transient implantable systems. The biodegradable conductive polymer composites were developed using iron (Fe) microparticles as the conductive filler and polycaprolactone (PCL) as the insulating matrix. The electrical resistivity and the mechanical and electrochemical properties of the composites were investigated during physiological degradation. The electrical percolation threshold was found at 17% iron volume fraction, but higher volume fractions exhibited more stable electrical resistivity throughout the time course of degradation. An electrical lifetime of over 20 days was achieved with 40% Fe composites, where the average resistivity was 0.3 Ω cm. Adhesion tests under physiological aging conditions were also performed. Biodegradable electrical interconnects based on 40%vf Fe-PCL composites were successfully micropattened in daisy-chain structures to illustrate process compatibility of these materials.

INTRODUCTION

There has been growing interest in the development and study of biodegradable medical devices in the medical and health industry. Since these devices comprise biodegradable materials, which can gradually decompose and be expelled from the body, patients do not need a second surgery to remove the devices from their bodies after healing. In addition, biodegradable devices overcome the negative effects associated with permanent implants, such as fibrous encapsulation and stress-shielding at the implant-tissue interface [1]. For these reasons, biodegradable implantable devices have been proposed for the monitoring and treatment of short-term ailments [2]. Biodegradable analogues of passive RF pressure sensors have been demonstrated, where degradation lifetime can be tailored based on the material composition and sensor design. For example, the degradation rate of zinc/iron inductors used in sensors can be controlled by altering the exposed area ratio of these two metals [3]. To date, biodegradable devices have been limited to either passive designs or active devices with short (i.e., minutes to hours) functional lifetime [4]. To achieve active biodegradable devices with clinically-relevant functional lifetimes, research must not only address the sensor and power source [5], but also the circuitry and electrical interconnects.

Non-degradable polymer-metal composites have been studied previously for interconnect materials because of their low process temperature, light weight, and environmental friendliness. Generally, these interconnect materials are developed using metal (e.g., silver, gold, and nickel) as the conductive filler and polymer (e.g., epoxy, silicone, polyimide) as the insulating matrix [6]. With appropriate materials modification, conductive polymer composites are interesting options for biodegradable system interconnects.

The behavior of such composite interconnects can be studied by use of percolation theory. Percolation theory is a statistical model that describes how randomly positioned sites are connected in a disordered system. Above a critical concentration of positioned sites, called the percolation threshold, a connected path is formed through the system [7]. Percolation theory has been used to interpret the electrical percolation of composite materials consisting of conductive filler in an insulating matrix. Composites behave as insulators and as conductors when the concentration of conductive filler is far below and above the percolation threshold, respectively. Further, composites show an appreciable change in electrical resistivity when the concentration crosses the percolation threshold.

We present the development of electrical interconnects comprising biodegradable conductive polymer composites for transient implantable systems. The electrical, mechanical, and electrochemical properties of the composites are investigated during physiological degradation. The percolation point of these composites is identified during this characterization, and favored compositions with reasonably stable properties are selected for subsequent testing. Finally, biodegradable electrical interconnects based on these favored compositions are micropatterned to demonstrate their compatibility with MEMS processing.

METHODS

Fabrication

Biodegradable conductive polymer composites were developed using Fe microparticles as the conductive filler and PCL as the insulating matrix, and micropatterned onto polylactic acid (PLA) substrates by screen printing to form electrical interconnects. (Fig. 1) First, the Fe powder (<10 μ m, ≥99.9% trace, Sigma Aldrich) was serially washed with dichloromethane (≥99.8%, Sigma Aldrich) and 1,4-dioxane (99.5%, Acros Organics) in triplicate. PCL (average M_n 80,000, Sigma Aldrich) pellets were solubilized in dioxane to a concentration of 200 mg/mL. Oleic acid (90%, Sigma Aldrich) was then added to the serially-washed Fe as a surfactant to facilitate the homogeneous suspension of Fe in the PCL solution. Finally, PCL solution was added to the Fe and oleic acid mixture to reach the desired volume fraction.

Screen printing was performed using 10-mil-thick polyester shim as the stencils. First, the polyester shim was micromachined with a CO_2 laser. The patterned shim was then laminated onto PLA substrates using polyvinyl alcohol (PVA) as the adhesive. The PLA substrates had been previously metallized with Fe traces 100 nm in thickness by sputter deposition through a shadow mask and laser micromachined (532 nm wavelength, 100 μ J pulse energy, 1 ns pulse width, and 10 W maximum power) to pattern the Fe traces.



Figure 1: Fabrication scheme and daisy chain structures of Fe-PCL biodegradable electrical interconnects.

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Specifically, 25% of the Fe surface area of each pad was ablated to expose the underlying PLA to promote adhesion of the composites. Finally, the Fe-PCL composite was applied and screen printed onto the pre-patterned Fe-PLA substrate. In this manner, electrically-continuous daisy chain structures were micropatterned on the PLA.

Electrical and Mechanical Characterization

As electrical conductivity and mechanical robustness are key design requirements for interconnects, the effect of physiological degradation on the electrical resistivity and elastic modulus of Fe-PCL composites was investigated. Composite films with different volume fractions of Fe were prepared and immersed in simulated body fluid (SBF) at 37°C in an incubator to simulate physiological degradation. At intermittent time points, the resistivity of the films was measured using electrical probe testing. In addition, physiologically conditioned Fe-PCL composite films were mechanically strained to 1% tensile strain at 1 Hz for 100 cycles in a uniaxial mechanical testing system (Bose Electroforce 3200) to monitor the elastic modulus of the composite with respect to degradation time. As biodegradable electrical interconnects may also be exposed to occasional mechanical strain due to physiological loading and local micromotion, the electrical resistivity of mechanically and physiologically conditioned Fe-PCL films was also monitored.

The functional lifetime of electrical interconnects may also be limited by its adhesion strength. While this is not desirable, physiological degradation may further exacerbate the effect. In order to overcome this design challenge, it was important to enhance the adhesion strength of Fe-PCL composites on biodegradable substrates and to ensure that the adhesion persists throughout physiological degradation. Towards these goals, as well as to demonstrate the processing compatibility of biodegradable Fe-PCL interconnects on similarly biodegradable substrates, Fe-PCL electrical interconnects were screen printed onto micropatterned Fe-PLA substrates. Specifically, iron traces were micropatterned onto a 250-µm-thick PLA substrate by sputter deposition through a shadow mask to form a grid of 10mm x 10mm squares of 100 nm thickness. Next, the sputtered Fe was ablated with an array of 250µm-wide square holes to expose 25% of the underlying PLA and Fe-PCL composite (40%vf of Fe) was screen printed onto the micropatterned Fe using the fabrication procedure described earlier. Samples was then diced into individual coupons comprising three test samples per coupon and immersed in SBF at 37°C for physiological conditioning. At periodic time points, samples were retrieved for optical microscopy and adhesion testing in accordance with ASTM D3359-09 guidelines [8]. Briefly, two intersecting cuts were made through the Fe-PCL composite in each sample using a razor blade; the intersection point was aligned with the center of each square pattern and the intersection angle ranged between 30° and 45°. For each test, 75-mm-long strip of Scotch permanent tape was adhered onto the cut Fe-PCL composite with the tape running in the same direction as the smaller intersecting angle. Good contact was ensured before proceeding. Within 90 ± 30 s of application, the tape was pulled off rapidly at a peel angle as close to 180° as possible. Finally, the cut area was inspected for removal of composites from the substrate and adhesion was rated based on the ASTM scale. The scale ranges from 0 to 5, which correspond to complete removal of the adhesive material (i.e., poorest adhesion) and negligible loss of adhesive material, respectively.

Electrochemical Properties Characterization

The degradation behavior of Fe-PCL composite films (40%vf Fe) were evaluated by linear sweep voltammetry (LSV) (Gamry Reference 600). The test was performed at 37°C in SBF using a

three-electrode configuration, where the working, counter and reference electrodes were the Fe-PCL composite, a platinum mesh, and a saturated silver/silver chloride electrode, respectively. The measurement scanned a 500 mV window centered about the open circuit potential at a scan rate of 2.5 mV/s after open circuit potential stabilization. The corrosion potential and corrosion rate were determined by linear fitting the results based on Tafel equations [9].

RESULTS AND DISCUSSION Optical Images

Figure 2 shows the micropatterned biodegradable daisy chain structures comprising Fe-PCL composites interconnects bridging sputter-deposited Fe traces. The interconnect width and thickness were 350 μ m and 250 μ m, respectively, which agree with the microscale dimensions of MEMS devices. Together, the micropatterned daisy chain structures demonstrate the compatibility of Fe-PCL composite interconnects with standard MEMS processing techniques. Fig. 2B shows the backside view of a daisy chain structure to visualize the laser machined Fe traces featuring 25% areal exposure of the underlying PLA substrate. The ablated regions with PLA exposure are in direct contact with the overlying screen-printed Fe-PCL composite to promote adhesion.



Figure 2: Optical images of micropatterned biodegradable daisy chain structures comprising Fe-PCL conductive polymer composites as interconnects bridging sputter-deposited Fe traces. (A) Zoomed out. (B) Backside view of bridged pad structures in a daisy chain.

Electrical Resistivity

Figure 3 shows the electrical resistivity of Fe-PCL composite films as a function of volume fraction, parameterized by duration of physiological degradation. The electrical resistivity decreased by orders of magnitude between 5% and 20% volume fractions of Fe with a percolation threshold at approximately 17%vf, above which the electrical resistivity did not appreciably change. However, with physiological degradation, the resistivity of the composites at higher volume fraction of Fe (i.e. \geq 40%) exhibited enhanced stability, potentially attributable to the smaller percentage of iron being lost at higher volume fractions as degradation proceeds (since the initial amount of iron at higher volume fractions is higher). Another potential explanation is that Fe corrosion products, such as oxides formed during degradation, present an electrical barrier to conduction.

Figure 4 shows the electrical resistivity of composites with varying volume fractions of Fe throughout the time course of degradation. The electrical resistivity of the films decreased by roughly 3 orders of magnitude after immersion in SBF. This is possibly due to electrolyte permeation into PCL, based on previous



Figure 3: Electrical resistivity of Fe-PCL biodegradable conductive polymer composite films at varying volume fractions of Fe, parameterized by degradation time.



Figure 4: Electrical resistivity of Fe-PCL biodegradable conductive polymer composite films parametermized by volume fraction of Fe as a function of degradation time.



Figure 5: Electrical resistivity of Fe-PCL biodegradable conductive polymer composite films (40% of of Fe) as a function of degradation time with and without intermittent strain.

measurements that showed the resistivity of PCL decreased by 2 orders of magnitude after immersion in SBF. The electrical resistivity of composite films with volume fractions of Fe above 17% stablized at approximately 0.1 Ω cm within 6 hours after immersion in SBF, which indicates that the composites can behave functionally within a short period of time after immersion. Although the resistivity is higher than traditional interconnect materials, the Fe-PCL composites above the percolation threshold are still candidate materials for use in low current interconnects. In addition, the electrical resistivity of composite films with volume fractions of Fe above 17%vf remained stable within 500 hours of immersion in SBF

and, thus, demonstrated a liftetime of over 20 days. After 500 hours, the resistivity of all films started to increase as corrosion proceeded. Because stability of conductivity over the functional lifetime of the interconnect is an important parameter, composites with 40%vf of Fe were selected for use in subsequent testing.

To assess the effects of intermittent strain on the interconnect conductivity, the electrical resistivity of 40%vf Fe-PCL composite films as a function of degradation time with and without intermittent cyclic tensile strain is compared (Fig. 5). The mechanical conditioning simulated the strains that an implanted chip may experience in the body. Each strained sample was subjected to 100 cycles of a 1% strain at a frequency of 1 Hz. The resistivity of the intermittently strained films decreased and stabilized to approximately 0.3 Ω ·cm within 6 hours in SBF, similar to the electrical resistivity of films that had not undergone mechanical strain. In addition, the resistivity fluctuations of both films were commensurate, which indicates that the stability in resistivity of composites with 40%vf of Fe was not significantly affected by intermittent strain under physiological degradation.

Mechanical Properties

During the intermittent strain process described above, it was possible to assess the elastic modulus of the 40%vf composite material as a function of degradation time (Figure 6). The elastic modulus of the composite was 200 MPa prior to immersion in SBF. The elastic modulus was larger than that of bulk PCL (0.8 ± 0.1 MPa) because Fe microparticles increased the modulus. The elastic modulus increase stabilized at approximately 400 MPa after immersion in SBF. A possible explanation for this behavior is the formation of oxides during degradation resulting in an increased elastic modulus. However, the elastic modulus is still lower than that of metal and semiconductor interconnects (\approx 100 GPa), which suggests that the composites are more compliant and could provide more flexibility in interconnection. The elastic modulus fluctuated within 25% over 500 hours, indicating a relatively good stability in degradation.



Figure 6: Elastic modulus of Fe-PCL biodegradable conductive polymer composite films (40%vf of Fe) as a function of degradation time under intermittent strain.

As biodegradable electrical interconnects must maintain adhesion onto similarly biodegradable substrates, the adhesion of Fe-PCL composites on Fe-PLA substrates was investigated with respect to degradation time (Fig. 7). From previous experiments, it was found that the composites showed poor adhesion on stainless steel substrates, where composites could be peeled off without effort (i.e., rating of 0 in adhesion tests). Further, it was hypothesized that the corrosion products formed during degradation could embrittle the Fe-PCL composite and deteriorate its adhesion. However, the design of the Fe traces presented in this study, which featured 25%



Figure 7: Optical images of physiologically conditioned Fe-PCL composites (40% of Fe) after adhesion tests were performed on PLA substrates with micropatterned Fe traces.



Figure 8: Adhesion test ratings of Fe-PCL composites on micropatterned Fe-PLA compared against stainless steel (i.e., bulk metal) control samples. Tests were performed on Fe-PCL composites after varying immersion times in SBF to simulate degradation conditions. Adhesion is rated between 0 to 5; higher ratings correspond to better adhesion.



Figure 9: Polarization curves of Fe-PCL biodegradable conductive polymer composite films (40%vf of Fe) after physiological degradation.

areal exposure of the underlying PLA substrate, significantly improved its adhesion with Fe-PCL composites. Specifically, the composites scored the maximum adhesion rating (i.e., 5) and retained their adhesion strength throughout the time course of degradation (Fig. 8). The results not only confirmed that the design of the micropatterned Fe traces and Fe-PCL composites can successfully resolve previous adhesion challenges, but also support their use for biodegradable electrical interconnects in transient implantable applications.

Electrochemical Properties

Figure 9 shows the polarization curves of Fe-PCL biodegradable conductive polymer composite films with 40%vf of Fe throughout degradation. The corrosion potential of the composite films was -0.537 V vs. Ag/AgCl prior to degradation conditioning in SBF, which approximates the corrosion potential of bulk Fe. The corrosion potential decreased when the degradation time increased, indicating the increasing corrosion tendency of the films. Moreover, the corrosion current density increased as a function of time, implying an increased corrosion rate as degradation continued.

CONCLUSIONS

In this study, the suitability of Fe-PCL composites as potential candidates for biodegradable electrical interconnects was investigated. The electrical percolation threshold of Fe-PCL composites was found at 17% of Fe. Stability of resistivity over a reasonable functional lifetime was achieved by utilizing composites with 40% of Fe, in excess of the percolation threshold; stability was maintained even under intermittent tensile strain. Short term adhesion testing indicated reasonable adhesive stability of these interconnect materials. These properties, together with the relatively low composite elastic modulus, suggests their potential application as interconnects. The biodegradable electrical interconnects were shown to be compatible with standard processing techniques through the formation of micropatterned daisy-chain structures.

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LOW-POWER ELECTRICALLY CONTROLLED THERMOELASTIC MICROFLUIDIC VALVE ARRAY FOR MULTIPLEXED IMMUNOASSAY

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ABSTRACT

We report a low-power, thermally actuated, and elastomer-based microvalve, which is integrated in a thermoplastic microfluidic device for multiplexed immunoassays. The thermal-actuating material used in this valve also enables a latching capability. Thirteen microfluidic valves have been integrated in a microfluidic device, enabling a network of fluid channels for reagent manipulation and sequential delivery. With a unique combination of antibodies, the device is able to analyze six proteins simultaneously. The total energy consumption for a device during the immunoassay is only 4 mWh.

INTRODUCTION

Immunoassays are extensively used in biomedical research, clinical diagnostics, and food/water safety testing [1-3]. Although they can be performed in a microplate to increase sample throughput, immunoassay in each well is generally limited to one analyte per test. Such a format is inefficient and not cost-effective for the requirement of detecting multiple analytes in one sample. For instance, a set of biomarkers should be detected for a particular disease to enhance the accuracy and reduce false positives or negatives. Similarly, a spectrum of pathogens should be simultaneously monitored in foods and drinking water.

A microfluidic network (μ FN) has potential to achieve multiplexed immunoassay in a microfluidic device [4, 5]. Figure 1 shows the process of μ FN for protein patterning. First, multiple antibodies are introduced into each channel respectively (Figure 1a) Then sample solutions are introduced through other sets of channels perpendicular to the previous ones (Figure 1b). Each intersection area is a detection area for a specific protein. Although multiple target proteins can be detected simultaneously, many time-consuming steps of manual control of flows still required. A microvalve can be utilized to solve the problem.



Figure 1: Microfluidic networks for protein patterning. (A) Each antibody is immobilized on each channel (blue). (B) The channel set for samples (in pink) is oriented perpendicularly to the previous set (in blue). Each intersection area (in black) is an assay site for a specific analyte.

Among all microvalve designs, an electrically controlled and thermally actuated microvalve is the most compact, hence portable [6-9]. However high power consumption is a drawback of this type of microvalve, especially when multiple valves are required in one device for multiplexing. Moreover, utilizing thermal actuation in a low-cost plastic device is a challenge because the maximum temperature that plastic can withstand is lower than silicon or glass.

In this study, we design a low-power, thermally actuated

978-1-940470-02-3/HH2016/\$25©2016TRF DOI 10.31438/trf.hh2016.90 microvalve array which is featured by the latching capability. The average energy consumption per valve during an immunoassay is only 0.31 mWh, which is about 300-500 times less than other thermoelastic microvalves [10, 11]. With such a low-power valve, we are able to integrate the valves into an all-plastic microfluidic device that enables portable operation. We have demonstrated a multiplexed immunoassay on 6 assay sites in the device.

MATERIALS AND METHODS Reagents and Materials

COC resin and film (Zeonor 1020R and 1420R) were purchased from Zeon Chemicals (Louisville, KY, USA). Polyurethane elastomer (Bayhydrol 110) was purchased from Bayer Material Science (Pittsburgh, PA, USA). COC sheet (8007) from Topas Advanced Polymer Inc. (Florence, KY). Paraffin wax (MP 47°C-65°C) was purchased from Spectrum Chemicals (New Brunswick, NJ, USA). UV curable epoxy is purchased from McMaster-Carr (Atlanta, GA, USA). Avidin was purchased from Fisher Scientific and the proteins C-reactive protein (CRP), transferrin (TfR), resistin (Res), and erythropoietin (EpoR) with their respective biotinylated capture and detection antibodies were purchased from R&D systems (Minneapolis, MN, USA). The dyes Alexa Fluor 647 was purchased from Life Technologies (Carlsbad, CA, USA).

Device Design and Fabrication

A low-power, electrically controlled thermoelastic-based thermoplastic microvalve consists of 4 layers, which are channel, cavity, elastomer, and heater layers, as illustrated in Figure 2a. The channel layer is formed by compression molding COC resins to 1.5-mm thick. The microchannels in this layer are 100 μ m wide, 35 μ m deep. The elastomer layer is a 4- μ m-thick polyurethane



Figure 2: (a) Illustration of a microvalve consisting of 4 layers indicated. (b) Top-view picture of the electrically controlled thermolelastic valve array in a microfluidic device. Thirteen valves and 6 inlet/outlet were integrated in the device. The metallic wire is the electrical pattern of the heater layer. The channels were filled with a red dye solution to show the channel layout.

elastomer, which is made by spin-coating a polyurethane solution

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on a sacrificial layer, and then bonding it chemically with the channel layer as described in our previous work [12]. The cavity layer is a 254- μ m-thick COC sheet containing 1-mm-radius cavities, which are made using a cutting plotter (Graphtec America, Irvine, CA, USA). This layer is chemically bonded to the elastomer layer [12] as well. The heater layer consists of a thin copper film patterned in a serpentine shape by photolithography on a COC film. One mg of paraffin wax is placed in the cavities, and melted by putting the device on a hot plate (75°C) until wax fill the cavities entirely. The cavity layer is then bonded to the heater layer with UV curable epoxy.

Figure 2b shows the top-view of the device with a microvalve array. The device consists of 13 valves and 6 inlets/outlets. To visualize channels, a red dye solution is introduced into the device. The dark metallic line is the pattern of the copper wire on the heater layer. The size of device is only 2.25 inch x 1.25 inch.

Principle of Valve Operation

Figure 3 illustrates the valve operation including closing, latching, and opening as we recently reported [13]. When power is on, the wax changes from solid to liquid, expands, and deforms the elastomer to close the channel (Figure 3b). As the power is off, the wax returns to solid while the elastomer is still under deformation. Consequently, the valve remains closed or latched (Figure 3c). The valve is reopened by applying a short power pulse with a pressurized fluid from a syringe pump (Figure 3d).



Figure 3: Principle of valve operation. (a) Initial state of a valve. (b) When the heater is on, wax melts, expands, and deforms the elastomer layer to close the channel. (c) As the heater is off, wax changes to the solid phase faster than elastomer recovery. Consequently, the valve remains closed without power. (d) A pulse of power and a pressurized flow in the channel are simultaneously supplied to open the valve.

Multiplexed Immunoassay

To achieve simultaneous immunoassays of 6 proteins (4 target proteins and 2 control sites) in our device, the scheme of valve operation and immunoassay process are described as follows. First, 1 mg/ml avidin solution is introduced into all channels and incubated for 30 min. Then by controlling an appropriate set of valves as shown in Figure 4a, two vertical channels are accessible. Two mixtures of 10 μ g/ml capture antibody solutions (antibody 1,2 and antibody 3,4) are then introduced into each channel and incubated for 30 min. Next, by controlling another set of microvalves as shown in Figure 4b, three horizontal channels in the device are open. The sample solution which contains all target proteins is introduced in top and bottom channels, while the control solution which is bovine serum albumin (BSA) is introduced in the middle channel. After incubation for 30 min, three mixtures of 2.5 μ g/mL fluorescently labeled detection antibody solution (dAb 1,3, dAb 2,4 and dAb 1,2,3,4) are introduced to each channel as shown in Figure 4b. Note that washings with a PBS buffer are performed among different steps. As a result, each target proteins can only be detected in one assay site, which is the intersection area of channels. Moreover, the total energy consumption for a device during the immunoassay process is 4 mWh.



Figure 4: Immunoassay scheme for simultaneously detection of 4 proteins with 2 assay sites as negative controls. The device consists of 13 valves (solid blue circles and open circles with a cross), and 6 wells as fluid inlets/outlets. (a) As specific valves close (circles with cross), the capture antibodies flow in the vertical direction of pink arrows and are immobilized on the channels. (B) As the valve layout changes, the sample and detection antibodies flow in the horizontal directions as indicated by pink arrows. Due to the unique combination of antibodies indicated at the inlet, 4 proteins are distinctly detected at the 4 assay sites (red circles). The number at each red circle indicates the presence of a particular protein if a signal is detected there. The middle picture illustrates the sandwich immunoassay scheme at each assay site.

RESULTS AND DISCUSSION Valve Performance Test

In order to test the valve performance, a single valve testing setup was designed as shown in Figure 5a. A resistor is connected in series with the channel containing a sodium chloride solution. The electrical current (I) indicates whether the valve is open (I \neq 0) or closed (I = 0) since a closed valve can prevent the current passing though the solution in the channel.

Figure 5b shows the result of the valve testing. The red curve indicates the electric power for the heater, and the blue curve indicates the measurement of an electrical current. First, the valve is closed with a power of 56 mW in 24 s. After power is off, the valve remains closed (latched) as the current is around zero. To reopen the valve, a pulse of power is supplied for 5 s with a 1 μ L/min flow. The valve is reopened as the current indicated ~0.5 μ A.



Figure 5: (A) Experimental setup for valve performance test. Electrical current (I) determines whether the valve is closed (I = 0) or open ($I \neq 0$). (B) Cyclic operations of microvalve. The blue Y-axis (left) and blue curve represents the state of the valve; the red Y-axis (right) and red curve shows the power for the heater. The valve closes in 24 s with power at 56 mW and reopens with the same power in 5 s (pulse).

Assay Calibration Curve

In order to verify the immunoassay protocol in our device, a CRP regression analysis is performed. The CRP concentrations of 20, 40, 70, 140, and 250 ng/mL are used to for this analysis. The detection antibody is labeled with Alexa Fluor 647. The immunoassay protocol is the same as described above, except that only one protein is detected. Figure 6 shows the calibration curve of CRP detection. The negative control in this analysis is BSA, and the data at zero CRP concentration is included in the calibration curve. The recorded data points are fitted well to a reverse exponential curve with an R^2 value of 0.99.



Figure 6: Graph of CRP calibration curve. Each data point represents the fluorescence intensity from CRP measurement (20-250 ng/ml). Data point at 0 ng/ml CRP is the negative control using BSA. The curve is fitted with reverse exponential curve (y=4970-4828 $e^{-0.0046x}$) with a R^2 value of 0.99.

Multiplexed Immunoassay

Our microfluidic device is capable of detecting 6 different proteins simultaneously. To demonstrate the multiplexed immunoassay, 4 assay sites are used to detect 4 target analytes (CRP, transferrin, resistin, and erythropoietin) and the remaining 2 assay sites are used for negative controls (BSA). Since the real biological sample always contain multiple analytes, we mimic this situation by mixing all target proteins and BSA together as the sample solution. Figure 7 shows the fluorescence intensity in 6 assay sites. The signal-to-noise ratio for CRP (100 ng/ml), TfR (300 ng/ml), Res (198 ng/ml), and EpoR (134 ng/ml) are 5.4, 2.2, 3.0, and 3.1 respectively. Furthermore, as the fluorescence intensity of CRP is fitted with the calibration curve from Figure 6, the corresponding CRP concentration is 85 ng/ml, which gives 15% forecast error.



Figure 7. Fluorescence intensity of each assay sites with their corresponding protein concentration.

CONCLUSION

A thermoelastic microvalve using wax as the actuation material has been fabricated and characterized by investigating its latching behavior of the microvalve. The thermoelastic microvalve is controlled electronically, making the device portable as compared to pneumatic valves which require bulky accessories such as a gas tank. The latching capability reduces the power consumption, enabling us to integrate these valves in a low-cost, all-plastic microfluidic device. Using a microfluidic network strategy, we have successfully demonstrated a multiplexed immunoassay in the device. Moreover, the total energy consumption during the 1 hour immunoassay process was 4 mWh, which can be provided by a 9 V lithium battery. The low power, low cost, compact, and electrically controlled microvalve has a potential to enable portable, battery operated, disposable microfluidic devices for point-of-care diagnosis.

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MEMS AIR-MICROFLUIDIC LAB-ON-A-CHIP SENSOR FOR PERSONAL MONITORING OF AIRBORNE PARTICULATE MATTER

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ABSTRACT

We present the design, fabrication and experimental results for a novel air-microfluidic lab-on-a-chip sensor, which measures the mass concentration of airborne particulate matter (PM), such as tobacco smoke or diesel exhaust. The sensor detects PM having aerodynamic diameter (AD) of less than 2.5μ m, (PM2.5) directly by measuring the deposited PM on a mass-sensing resonator, at flowrates two orders of magnitude lower than the closest comparable devices. We report on an improved sensor design, new mask-less dispenser-printed SU-8-based bonding process, and recorded sensitivity of our newly-developed design.

INTRODUCTION

Particulate matter (PM), a mixture of small solid particles floating in the air, is an important type of air pollutant. The US EPA categorizes PM by size into coarse (A.D > 10 μ m) and fine (A.D < 2.5 μ m) particles. According to the EPA there are serious health hazards associated with fine particles. They are able to penetrate deep inside the human respiratory system and can cause adverse health effects such as increased respiratory symptoms, aggravated asthma, and cardiovascular problems leading to premature deaths in people with heart or lung diseases.

In this work we present a MEMS air-microfluidics lab-on-achip sensor for personal monitoring of PM2.5. A preliminary version of the sensor was reported in [1-2]. In contrast with other miniature PM sensors (such as. [3]), the device presented in this work uses a microfabricated film-bulk acoustic resonator (FBAR) to *directly* measure the mass of the deposited PM. In this work we present a newly optimized design of the virtual impactor (VI) and microchannels for balancing flowrates through minor and major channels, the development of a new mask-less adhesive wafer bonding technique using dispenser printing of SU-8, as well as characterization of the VI using a custom opto-gravimetric method. Data are also shown demonstrating sensor response to incense particles. The working flowrate is set to 6 mL/min and experimental results show the sensitivity of the sensor to be approximately 5 Hz × m³/µg × min, with a limit of detection (LOD) of 1 µg/m³.

Device operation

A conceptual block diagram and an image of the MEMS PM2.5 sensor are shown on Figs. 1 and 2, respectively. The sensor consists of three main components fabricated on three wafers (quartz-siliconquartz) bonded together. In the air-microfluidic circuit, PM particles are separated by the aerodynamic diameter using a virtual impactor (Fig. 2-a). PM2.5 and smaller are transported through the major flow channels while the larger particles are exhausted from the sensor through the minor flow channel. PM2.5 is then deposited by a thermophoretic precipitator (Fig. 2-b) on a mass sensitive FBAR. A change in the FBAR's resonant frequency occurs as the mass loading increases, and the rate of this frequency change corresponds to the particle concentration in the sampled air volume. Thermophoretic precipitation is driven by a microfabricated heater suspended just below the capping quartz wafer of the microfluidic circuit. Forward-looking Infrared (FLIR) analysis (Fig. 2-c) shows the temperature distribution (around 100 °C) of the heater while dissipating 50mW of power, which is typical for the thermophoretic precipitator in our designs.



Fig. 1. Conceptual functional diagram describing the operation of the MEMS PM2.5 sensor.



Fig. 2. Air-microfluidic MEMS PM2.5 Sensor. The sensor consists of a microfabricated virtual impactor (a), and a thermophoretic precipitator and mass sensing FBAR (b). FLIR analysis of the active heater dissipating 50mW is shown in (c). Air inlett (i) and outlet (ii) is annotated in the lower pane.

FABRICATION

The sensor is fabricated using a stack of three wafers. A CAD drawing in Fig. 3 schematically displays all the wafers. The fabrication process for the new sensor is shown on Fig. 4. It consists of separately fabricated middle and top wafers components, sub-

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dicing, and subsequent bonding of the two wafer die. A novel adhesive (SU-8 3025) bonding method was used to bond released microfabricated heaters (top wafer) with the etched channels of the middle wafer.



Fig. 3. CAD drawing of all three wafers of the PM2.5 sensor.

The bottom quartz wafer with gold electrodes contains the FBAR and its CMOS driver, providing an oscillating signal at the resonant frequency of the FBAR. The process is finished by attaching the bottom wafer to the stack of middle and top wafer.



Fig. 4. Fabrication process flow for the middle wafer (a1-3), top wafer (b1-3), dispenser printing of SU-8 and bonding of top and middle wafers (c1-4), bottom wafer fabrication (d1-3), and final assembly (e-1)

Thermophoretic heaters (top wafer)

The fabrication process for the thermophoretic heaters is illustrated in Fig. 4-b. First, a 1 μ m thick layer of phosphosilicate glass (PSG) is deposited using low-pressure chemical vapor deposition (LPCVD) process (2:4:1 SiH₄, O₂, PH₃ at 450°C) to form the sacrificial layer for HF release. A 2 μ m thick layer of *in-situ* doped polysilicon is then deposited through a LPCVD process (1:1 SiH₄, PH₃ at 615°C) over the PSG layer. To form low resistance connections to the heaters, a 100 nm thick layer of gold (Au) with a 5 nm chrome adhesion layer (Cr) is evaporated on top of the

polysilicon layer (b-1) and subsequently patterned using a photolithography process and etched using an iodine-based gold etchant and CR-7. To create the resistive heater elements, the polysilicon layer is etched by a reactive ion-etch (RIE) process (90% SF6, 10% O2, 66 sccm) (b-2). The wafer is subsequently diced and the individual die are soaked for 10 min in 49% HF to release the heaters from the substrate (b-3).

Microfluidic channels (middle wafer)

The fabrication process of the air-microfluidic channels is illustrated in Fig. 4-a. 1µm thick layer of silicon dioxide is thermally grown on a silicon-on-insulator (SOI) wafer with 200µm device layer (a-1). This layer serves as the hard mask for subsequent deep reactive ion etch (DRIE). Channels are then photo-lithographically patterned and etched through the device layer using a two-step DRIE processes (a-2) (Oxide etch: C₄F₈/H₂/He 15sccm, 8sccm and 174 sccm at 4mT, 1500W, Bias 350W at 13.56 MHz. Si etch and passivation: 7sec SF₆/O₂ 100sccm/13sccm at 18mTorr, 5sec C₄F₈ 100sccm at 18mTorr and 600 W, 15 W bias, 380 KHz). A two-step backside DRIE etch (handle and BOX) is subsequently performed to open the inlet (i), outlet (ii), and FBAR insertion opening (a-3).

Resonator and circuitry substrate (bottom wafer)

The bottom wafer fabrication process is illustrated in Fig. 4.(d-1 - d-3). A 5nm/100nm layer of Cr/Au is evaporated on a quartz wafer and patterned and etched lithographically to define electrodes and provide connections between electronic components, followed by the sub-dicing of the wafer. A CMOS chip and a $475\mu m \times 1mm$ \times 1mm silicon pedestals are die attached to the board and FBAR is attached on top of the pedestal by a non-conductive epoxy followed by a 60 min. curing at 150 °C. The FBAR is then wire-bonded to the CMOS and CMOS is wire-bonded to the gold electrodes. Then an 80µm thick adhesive polymeric seal is attached around the perimeter of the FBAR and CMOS to provide a seal for the opening through the middle wafer. Assembling the bottom board to the stack of the middle and top wafer is illustrated in Fig. 4.(e-1). The thickness of the seal also sets the height that the FBAR penetrates into the microfluidic channel. Finally, bypass capacitors and RF cables are soldered to provide off-board power and RF connections.

Wafer bonding

Bonding of the air microfluidics wafer (middle wafer) to the quartz wafer containing the released microfabricated gold heaters (top wafer) is a critical part in the assembly of PM sensor. Perfect sealing of microfluidic channels is necessary because even a small air leak can affect the flow rate in the minor and major channels and impair the functionality of the sensor. In this case, there are considerations which limit the use of some common wafer bonding methods such as direct bonding or anodic bonding. Different wafer materials (silicon and quartz), non-conformal geometry, and dielevel bonding, precludes the use of common direct bonding methods. Lack of alkali ions in quartz precludes the use of anodic bonding. In contrast, adhesive bonding which is a popular wafer bonding method for MEMS applications and microfluidics [4] is favored because of its compatibility with different substrate materials and non-planar geometries, ease of application and relatively inexpensive process, and possibility of selective bonding.

SU-8 polymer (negative photoresist) is frequently used in MEMS adhesive bonding applications; however, spin coating of SU-8 on either of the middle or top wafers is not practical because it can fill the microfluidic channels or damage the heaters suspended just beneath the quartz wafer. Thus selective deposition of the SU-8 adhesion layer via a dispenser printer is a better approach to apply SU-8 onto the desired areas.
Dispenser printing and subsequently the wafer bonding process are schematically displayed in Fig. 4-(c1-c4). SU-8 3025 (Microchem) is dispenser printed using a custom printer on the middle wafer to provide an adhesive seal around the perimeter of the minor and major channels, as well as around the virtual impactor (c1). After printing, the SU-8 is soft baked at 95°C for 3 minutes on a hotplate (c2). Then, the top wafer is aligned with the airmicrofluidics wafer and placed on top of it, followed by the application of a constant bonding force (such as adding weights on top) (c3). The SU-8 is then exposed to UV light for 60 seconds through the quartz wafer for polymerization (c4). Following exposure the SU-8 is further cured at 95°C for 5 minutes to provide a strong bond. Fig. 5(a-b) shows optical micrographs taken during the SU-8 dispenser printing process, while, Fig. 5(c) shows an image of the middle wafer after the SU-8 pattern is printed but before the bonding.



Fig. 5. (a-b) Optical micrographs showing the dispenser printed SU-8 (i) around the collection probe of the microfabricated VI. The nozzle of the dispenser printer is visible (ii). (c) Dispenser-printed pattern of SU-8 prior to bonding with the top wafer.

SU-8 spreading and transformation

Spreading of the printed SU-8 after applying the top wafer and a given bonding force must be taken into consideration to avoid the adhesive from reaching into microchannels or unwanted areas. Spreading of a liquid droplet placed on a solid surface is a common phenomenon. Under equilibrium, the well-known Young-Laplace equation governs the behavior of a liquid droplet placed on a solid surface and the angle of contact is calculated based on the interface tension between solid-liquid, solid-vapor, and liquid-vapor interfaces.

It has been shown in [5-6] that the area of contact of a droplet with a solid surface under a force will increase with time at first, but then it reaches a saturation value and does not increase significantly beyond that point. By calculating the saturation area we are able to precisely include safety margins in the design of printing pattern map. An inverse configuration space transformation can be applied to convert the desired pattern to the pattern that will be printed by the dispenser printer.

The saturation area of contact of a constant volume of liquid V_f with the solid surface under the load of M (kg) is calculated from (1), which is derived by Nag et al. in [5]:

$$A_{sat}^4 - A_0^4 = \frac{8\pi v_{\max} V_f^2 M}{3\mu} , \qquad (1)$$

where it is assumed that the load M is dropped and the maximum kinetic energy is dissipated by the viscous droplet as the load plate

comes to a rest. Thus the corresponding maximum velocity v_{max} can be calculated at the point of contact. A_0 denotes the initial area of the contact while A_{sat} denotes the saturation area and μ is the dynamic viscosity of the fluid.

In our case, as the majority of printing patterns of SU-8 are rectangles with very high aspect ratio (very thin and long lines), the spread of the SU-8 along the length is insignificant compared to spread along the width, and can be neglected.

$$(w_s l_s)^4 - (w_0 l_0)^4 = \frac{8\pi v_{\max} V_f^2 M}{3\mu}$$
(2)

 $l_s/l_0 \approx 1$ (for rectangular patterns with high aspect ratio compared to w_s/w_0),

$$w_s^4 - w_0^4 = \frac{8\pi v_{max} w_0^2 h_0^2 M}{3\mu l_0^2}$$
(3)

It should be noted that the constant volume of liquid is obtained from $V_f = w_0 h_0 l_0$ where w_0 and h_0 are the initial width and height of the pattern of SU-8. These can be modified by the dispenser printer's specifications such as syringe's tip size and air pressure.

A series of experiments have been designed and carried out to evaluate Eq. (3). Four lines of SU-8 3025 with width of 100 μ m, height of 40 μ m and length of 5 mm were printed to form a square shaped frame. Then the top plate (glass with weight of 20 g) followed by a load of 0.4 kg were placed on top. The process was captured using a camera (Canon EOS) at the rate of 30 frames per second and the images were analyzed using Adobe Premiere. The velocity v_{max} is attained as the load comes to the rest, calculated at 1m/s. The area of contact is saturated after ~60 seconds. Specification of SU-8 3023 from are listed in Tab 1.

Table 1: Specifications of SU-8 3025

	1 3 3	
Density	Kinematic	Dynamic
(kg/m^3)	Viscosity	Viscosity
	(cSt)	(kg/ms)
1143	4400	50.292

From Eq. (3) the saturation width is calculated to be $128\mu m$, which is consistent with the experimental results obtained by the image and video analysis. A $28\mu m$ spread (width) was measured from a line $100\mu m$ wide, however this does not take into account the spread as a result of unwanted lateral movements of the top wafer during the bonding process.



Fig. 6. Inverse configuration space transformation for a high aspect ratio line pattern (a,b) and a square test pattern (d,c).

The idea of the inverse configuration space transformation is displayed Fig. 6. Based on Eq. (3), the desired pattern (gray and black, (b) and (d)) is obtained by printing SU-8 in black areas only ((a) and (c)).

EXPERIMENTAL RESULTS

A representative result from exposing the MEMS PM2.5 sensor to smoke is shown in Fig. . The flow through the sensor was controlled using an external pump and the flowrate was set to 6 mL/min. The experimental results indicate the sensitivity of our sensor at 5 Hz × m³/µg × min, with a (LOD) of 1 µg/m³. The sensor showed a tendency to lose some mass after excessive deposition of smoke on the FBAR surface, likely due to the evaporation of semi-volatile constituents of the incense as the smoke equilibrates with ambient air. This is a common feature with continuous measurement methods and can be potentially used for PM speciation.



Fig. 7. Experimental results from the exposure of the MEMS PM2.5 to incense smoke in the range of 100 - 300 nm AD.

CONCLUSION

Due to the serious health hazards associated with fine PM (PM2.5), development of personal PM monitors is of great interest. In this work we presented the design, fabrication, and initial testing results of a MEMS air-microfluidics lab-on-a-chip PM2.5 sensor with improved VI design, fabrication process, and novel SU-8 dispenser printing based wafer bonding. The sensor has shown the sensitivity of 5 Hz × m³/µg × min, with a detection limit of 1 µg/m³. This small footprint air-microfluidic lab-on-a-chip presents an initial platform for future more complex air-microfluidic circuits.

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MICROELECTRODE ARRAYS IN THIN-LAYER CELLS FOR RAPID, SENSITIVE, AND CALIBRATION-LESS DETERMINATION OF ARSENIC IN μL VOLUMES BY ANODIC STRIPPING COULOMETRY

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ABSTRACT

Recent contamination events have emphasized the need for widespread heavy metal monitoring over extended periods of time. The customary and laborious random "grab sampling" for later analysis at central laboratories does not adequately satisfy this need. Our group is developing an electroanalytical approach that allows highly sensitive, calibration-less measurements to quantify heavy metals in microliter volumes. This approach offers promise for economical, miniaturized, remotely-deployable sensor networks. This study reports the application of microfabricated gold microelectrode arrays within a thin-layer cell and demonstrates sufficient sensitivity to detect sub-5-ppb arsenic concentrations in a microfluidic sample volume (< 5 μ L).

INTRODUCTION

A number of recent events have highlighted the need for heavy metal monitoring at a large number of sites in a simultaneous fashion over extended periods of time. Such events include contamination of drinking water from buildings with Pb plumbing [1], accidental spills of wastewater from storage sites into the environment [2], and the recently enacted regulations for heavy metals in power plant effluents [3]. In addition to these recurring events, heavy metals such as As, Pb, Hg, and Cd, have been of longstanding concern and are ranked #1, #2, #3, and #7, respectively, on the Agency for Toxic Substances at the >1300 National Superfund sites [4].

Current heavy metal monitoring and analysis methods rely on sophisticated techniques such as inductively coupled plasma mass spectrometry and graphite furnace atomic absorption spectroscopy, which are performed in central laboratories **[5, 6]**. This approach necessitates sustained, labor-intensive and coordinated "grabsampling" efforts to collect and transport samples to the central laboratory, placing severe limitations on the number of sites that can be monitored and the sampling frequency at these individual sites. This need for more frequent monitoring at a multitude of sites has, in fact, led to several types of portable 'suitcase' instruments; however, this approach also relies on regular site visits by a mobile technician.

The desire to reduce reliance on the above labor-intensive sampling practices has led to a number of automated 'on-line' metal analyzers [7-11]. In automated analyzers, electrochemical methodologies have been widely practiced since the associated instrumentation is amenable to direct control and automation by integrated electronics, and the electrochemical stripping analysis of heavy metals is a well-established and highly sensitive technique. A recent review of 172 on-line stripping methods spanning a 12 year period, however, reveals the ubiquity of automating continuous-flow, bench-scale (mL) analyses and the need for frequent calibration with standards or blank solutions [12].

At present, the automated electrochemical heavy metal analyzers based on the above approaches are necessarily complex and costly, in order to accommodate the calibration and sample pretreatment functions for large sample volumes. And even with this capacity for automation, such analyzers are effectively cabinet-sized appliances intended for centralized, industrial process monitoring, which from an economic perspective, makes them unsuitable for environmental and/or decentralized monitoring applications.

Our group has therefore been developing an autonomous, economical, remotely deployed sensor platform for continuous monitoring of heavy metals in water intended for use at the superfund sites, which may also be suitable for other decentralized heavy metal monitoring applications. The approach combines elements of microfabrication, microfluidics, and electrochemistry which synergistically provide inexpensive, simple to automate, mass-producible, redundant sensor networks with minimal requirements for on-site power and reagents [13-16]. This approach has been designed from the outset to take advantage of the wellknown but little-used electrochemical coulometry method, which is one of very few analytical chemistry techniques that is capable of calibration-free measurements [14, 16].

Further, coulometry can be combined with another electrochemical technique: stripping analysis, which is a wellestablished and remarkably sensitive technique for the analysis of heavy metals. The remarkable sensitivity of stripping analysis arises from the preceding pre-concentration step in which dissolved species such as metal ions are accumulated on the electrode surface. Naturally, the sensitivity is greatly enhanced when a large volume of sample is analyzed, since more of the dissolved species can be pre-concentrated on the electrode surface. Given that even trace (low ppb) heavy metal concentrations are of environmental concern, it stands to reason that the required sensitivity is particularly challenging to attain in microfluidic devices, as we propose.

In this work, it is shown that sufficient sensitivity is attainable within a microfluidic device (analysis volume $< 5 \ \mu$ L) to detect sub-5ppb arsenic concentrations, which even meets the EPA mandated regulations for arsenic. Further, the overall analytical sensitivity and practical performance of the sensor is shown to be responsive to parameters which, in principle, can be controlled during design and manufacture of the sensor.

THEORY

The basic premise of the sensor relies on Faraday's 1st law of electrolysis, first proposed by Michael Faraday in 1834 **[17]**:

$$Q = n \times F \times N$$
 Eqn. 1

where Q represents the charge, n represents the number of exchanged electrons per mole of analyte, F is the Faraday constant, and N is the number of moles of analyte. The number of moles of analyte can be represented as:

$$N = C \times V$$
 Eqn. 2

where C represents the analyte concentration and V represents the sample volume. For a stripping process, according to **Eqn. 1** and **Eqn. 2**, the stripping charge (Q_{strip}) ought to correspond exactly to the prevailing analyte concentration according to:

$$Q_{strip} = n \times F \times C \times V$$
 Eqn. 3

when the redox reaction (n) and the cell volume (V) are known, so

978-1-940470-02-3/HH2016/\$25©2016TRF DOI 10.31438/trf.hh2016.92 Solid-State Sensors, Actuators and Microsystems Workshop Hilton Head Island, South Carolina, June 5-9, 2016 long as sufficient time is allowed for electrolysis to proceed to 100% completion during the pre-concentration step. In order to achieve complete pre-concentration of dissolved metals rapidly (in about one minute), our group has developed a thin-layer cell, the schematic of which is shown in **Figure 1**. The cell features a working electrode as one wall of a thin-layer fluidic cell, which is isolated from the counter/reference electrode compartment by a semi-permeable membrane.



Figure 1: Partial schematic of thin-layer cell for coulometry with structural layers omitted for clarity

One practical limitation of **Eqn. 3** is that, under experimental conditions, the measured charge (Q_{total}) represents not only the faradaic processes of interest (e.g., the stripping of plated metals, Q_{strip}), but also any background and non-faradaic processes (e.g., double layer charging current at the electrode/electrolyte interface, Q_{dl}). Accordingly the subtraction of Q_{dl} is necessary for accurate estimation of Q_{strip} According to:

$$Q_{strip} = Q_{total} - Q_{dl}$$
 Eqn. 4

Although direct measurement of Odl can be obtained by performing the experiment under identical conditions in a blank solution, a simpler approach for in-the-field analysis is to perform this measurement of Q_{dl} directly in the sample solution. Accordingly, our group has developed a method we have termed double potential step anodic stripping coulometry (DPS-ASC) where the deposition potential is applied briefly (100 msec), followed by the stripping potential (pulses 1 and 2 of Figure 2). Since no appreciable metal pre-concentration occurs during this rapid step, the total charge (area under the curve) for pulse 2 is an approximation of the Q_{dl}. This pulse sequence is repeated, only with a much longer deposition (pre-concentration) pulse (60 seconds, pulse 3 of Figure 2) to exhaustively plate all dissolved metals in the thin-layer cell volume. Subsequent stripping in Pulse 4 of Figure 2 represents Qtotal. The charge corresponding solely to Qstrip can be visualized by overlaying pulses 2 and 4 (Qdl and Qtotal, respectively) on the same axis, which in principle, is equivalent to the operation shown in Eqn. 4.



Figure 2: Illustration (not to scale) of time vs. recorded current for DPS-ASC. Positive pulses are -500mV deposition potential; Negative pulses are +500mV stripping potential. Signal obtained by overlaying pulses 2 and 4 on the same axis and integration to obtain charge within crescent (Figure 3)

EXPERIMENTAL

The flow cell consists of two polycarbonate fixtures, rubber gasket layers, a 100 µm thick pyrolytic graphite sheet (Panasonic Electronic Components, Secaucus, NJ) as a counter electrode (CE), and a 200 MWCO membrane (SelRO MPF-34, Koch Membrane Systems, Inc., Wilmington, MA). An access hole on the top polycarbonate fixture allows insertion of a custom-made Ag/AgCl 0.8 mm OD miniature reference electrode (RE), as previously described in [14]. Assembly yields a three-electrode, membraneseparated, dual-compartment cell with independent stoppable flow paths (Figure 1).

 As_2O_3 AAS standard solution (1000 ppm) is diluted into a supporting electrolyte solution containing 10 mM HNO₃ and 10 mM NaCl to make the concentrations indicated in the text below.

Electrochemical measurements were carried out using a BASi Epsilon potentiostat (Bioanalytical Systems, West Lafayette, IN). In all cases, a 10 second pre-conditioning step (at the stripping potential) is followed by the sequence of potentials in **Figure 2**. Briefly, this consists of an in situ blank deposition for 100 msec followed by stripping for 320 msec, then deposition for 60 seconds to exhaust the cell volume of all dissolved metals followed by stripping for 320 msec. The deposition and stripping potentials for arsenite are -500 mV and + 500mV, respectively.

RESULTS

The measured charge values (crescent shaped areas resulting from overlaying pulses 2 and 4) are shown for the indicated arsenite concentrations in **Figure 3**, where a macro-electrode comprised one wall of a 1.85μ L thin-layer cell. Under these conditions, the crescent obtained for the 100 ppb arsenite signal is discernably larger than the crescent obtained in the absence of any arsenite. However, it is also apparent that detection of the lower arsenite concentrations would benefit from a smaller crescent at 0 ppb arsenite. This is also reflected in numerical plots of the charge vs. arsenite concentrations shown in **Figure 5**. Despite a clearly linear dependence of charge on the arsenite concentration, it is evident that the non-zero intercept of the line contributes significantly to the signal at the lowest arsenic concentrations. This is due to the fact that, in practice, the DPS-ASC background correction only provides an approximation of Q_{dl}.



Figure 3: Obtained charge for elliptical macro electrode as one wall of 1.85μ L thin layer cell. Pulse 2 (grey) is background. Pulse 4 (black) is arsenic stripping and background. All traces overlay three sets of data.

Numerous experiments have shown that the value of the intercept (in the absence of any metal solutions) is directly related to the total electrode area. In order to reduce the electrode array we have therefore microfabricated microelectrode arrays (MEAS) by depositing silicon nitride on the gold surface, followed by traditional photolithographic patterning and a liquid-based HF vapor etching in the fume hood, yielding >99% electrochemically active MEA elements (see copper plated MEA in **Figure 4**). Photographs of the arrays are shown in **Figure 4** and the array geometries are summarized in **Table 1**.



Figure 4: Side view schematic of the MEA sensor chip (top left); and optical microscopy photos of MEAs

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chip design	number of electrodes	edge to edge distance	electrode area	geometric area*
Ellipse	1	NA	0.232 cm ²	0.23 cm ²
50 µm	3008	50 µm	0.059 cm ²	0.23 cm^2
20 µm	6394	50 µm	0.020 cm ²	0.23 cm ²
5 µm	12711	50 µm	0.002 cm^2	0.23cm ²

The data obtained for the microelectrode arrays are shown in **Figure 5**, where the crescents for much lower arsenite concentrations were discernible from the crescent at 0 ppb arsenite. For instance, the 5 μ m array of **Figure 5A** allows clear visualization of sub-5-ppb arsenite concentrations. In all cases, the lines on either side of the crescent are overlaid triplicates from three trials (i.e., three grey traces and three black traces). This inter-trial reproducibility is a notable feature of the DPS-ASC method {RSD typically <5%}. In addition, the data of **Figure 5** show that the stripping signal is much faster on the microelectrode arrays than the single macro-electrode. This observation is in agreement with the accepted behavior for microelectrodes [18].



Figure 5: Signals obtained with 5 μ m array (A); 50 μ m array (B). Note scale bars compared to figure 3 and discernibility of <5 ppb arsenic concentrations. All traces overlay three sets of data.

To enable a more direct comparison to the macro-electrode, the charge values shown in **Figures 3** and **5** are plotted vs. the arsenite concentration in **Figure 6**. As noted above, the error bars reflect the inter-trial reproducibility. The variations in the slope of the arsenite calibration lines of reflect the fact that the volume of



Figure 6: Calibration plot of charge (crescent area) vs. concentration at macro electrode (black), 50 µm array (blue), 20 µm array (green), and 5 µm array (red). Note improved accuracy/lower intercept of MEA in inset.

the thin-layer cell varied slightly from assembly to assembly. Since the slope is defined as the analytical sensitivity, thin-layer cells of different volumes may, in the future, be designed to meet performance requirements for different samples. Nonetheless, the stripping charge obtained under each set of conditions were linear over a wide range of the concentrations. At higher concentrations, the slope of the lines decreased (not shown) since metallic As acts as an insulator, in agreement with previous observations [19].

As shown on the inset of **Figure 6**, the intercepts at 0 ppb arsenite decreased with the electrode area. Correspondingly, the charge values within the crescent at 0 ppb decreased in proportion to the decreasing electrode area, as shown in **Table 2**. Further, the charge obtained at this intercept value represented a smaller fraction of the measured charge, which is particularly important at the lowest arsenite concentrations.

Table 2: Charge within crescent at 0 ppb arsenite as a smaller portion of the measured charge at 100 ppb (100 ppb not measured on the 20 μ m array, 50 ppb shown).

	Charge within crescent (μ C)							
	(rel	(relative standard deviation)						
[Arsenite] (ppb)	macro- electrode	50 μm array	20 μm array	5 μm array				
0	1.54 (2.4 %)	0.49 (11.1 %)	0.22 (2.6 %)	0.039 (3.4 %)				
50		1.06 (2.2 %)		0.26 (3.2 %)				
100	2.19 (1.6 %)		0.86 (1.5 %)	0.42 (9.2 %)				

CONCLUSIONS

This paper describes progress towards sensing trace arsenite concentrations in microfluidic volumes utilizing double potential step anodic stripping coulometry (DPS-ASC). The use of microelectrode arrays is shown to significantly enhance the capability to perform As measurements below 5 ppb. In addition to the performance enhancement attained by utilizing microelectrode arrays to reduce the area of the working electrode and the contribution of Qdl to the measurement, it was confirmed that the slope of measured standards reflects the volume of the assembled electrochemical cell. Thus, for a given microelectrode array geometry, improvements in the overall sensor performance at the lowest concentrations can be further enhanced by designing thinlayer cells with larger volumes. Finally, we are incorporating the performance of this cell into an autonomous, low-power, calibration-less system intended for autonomous use in remote locations.

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CAPILLARY MICROFLUIDICS-INTEGRATED IMPEDANCE SENSOR FOR RAPID LABEL-FREE ANTIBODY SENSING

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ABSTRACT

We report on a microsystem integrating capillary microfluidics and impedance sensors for controlled on-chip nanoreceptor assembly and label-free antibody sensing. Leveraging engineered surface tension in open-channel capillary microfluidic pumps and stop valves, genetically engineered *Tobacco mosaic* virus-like particle (VLP) nanoreceptors are autonomously delivered and assembled onto the impedance microsensor. The evaporation-assisted VLP assembly shortens the time required for sensor functionalization to 6 minutes, a significant reduction in time compared with the VLP self-assembly process. Such VLP-functionalized impedance sensors can perform label-free antibody detection down to 10 ng/mL within 5 minutes. This platform has demonstrated great potential in on-demand portable biosensing.

INTRODUCTION

In recent years, there are growing needs for decentralized and personalized biomedical devices to better understand the body's functions in real-time for both care-takers and patients. These new incentives have promoted the development of a variety of portable electronics. Lab-on-a-chip biosensors are the most successful examples of these portable biomedical devices. They combine sensitive and selective transducers with microfluidics to facilitate complex sample delivery, manipulation and analysis on a portable platform [1, 2]. However, most lab-on-a-chip devices rely heavily on external pumps and valves in sample analysis, which increases complexity of the final device operation. On the transduction aspect, the limited active area of a portable sensor platform often confines the amount of biorecognition elements available on sensors, compromising the sensitivity of a transducer. Therefore, it is necessary to develop high performance biological receptors and the associated sensor functionalization processes to simplify microsensor functionalization while enhancing sensor performance.

VLP, a derivative of the *Tobacco mosaic virus* capsid, is a high surface area biomolecule featuring a large number of genetically programmable sites on its nanorod surface [3]. Genetic modifications include cysteines to promote self-assembly onto various materials and binding peptides to enable selective target molecule binding. Previously, VLPs were successfully utilized as biosensing nanoreceptors for detecting antibodies in sandwiched immunoassays, which relied on labeling the bound target molecules with enzyme-linked secondary antibodies [2, 4]. The labeling, necessitated most likely by low VLP assembly density, adds to the overall cost and time of sensing.

In this work, we integrate VLP bio-recognition elements on an impedance microsensor through an autonomous microfluidic platform to address the aforementioned challenges. High aspect ratio KMPR capillary micropumps and stop valves are created on top of the impedance sensor surface. Leveraging capillary action and surface evaporation, the utilization efficiency of VLPs in sensor functionalization is greatly improved compared with VLP self-assembly (which requires emersion of device surface in VLP suspension for 18 hours). An impedance microsensor was developed on the microsystem platform to study the accelerated VLP assembly process as well as real-time monitoring of biological binding events between the target antibody and VLP sensing probes. The VLPs assemble and form a dense functional receptor layer on the sensor, which increases the biosensing efficacy and enabled label-free antibody sensing rapidly after the sensor functionalization.

METHODS

Capillary microfluidics-integrated impedance sensor

The capillary microfluidics-integrated impedance sensor microsystem aims at achieving rapid as well as controlled sensor functionalization. The microsystem is composed of an impedance microsensors, three open-channel capillary micropump units and an array of microfluidic stop-valves. The benefits of using capillary microfluidic devices are the autonomous liquid sample delivery and manipulation using capillary action.



Figure 1: Schematic of the impedance sensor and capillary microfluidics-integrated microsystem.

Fig. 1 shows a schematic of our microsystem. Micropillar structures on a silicon wafer create the main geometries of the capillary micropump. The hydrophilic sidewalls of the pillar structures provide capillary driving forces to advance the liquid meniscus. The stop valve array is made of sharp "nozzles", which dramatically change the sidewall-meniscus angle during liquid filling. This creates a localized low surface energy pocket to prevent the liquid sample from flowing to the venting capillary pump on right [5]. A capillary pump that is connected to a smaller inlet provides the only triggering mechanism. For antibody sensing, a triggering fluid is introduced from the small inlet in the middle. The fluid breaks the liquid-air interface of the meniscus at the stop valves, generating continuous flow while performing antibody sensing.

The impedance microsensor features interdigitated electrodes with 4 μ m width/spacing and an overall area of 2×2 mm². The capillary microfluidic layer is 60 μ m thick with an aspect ratio (channel depth : channel width) of 2.4:1 at the micropump area and the aspect ratio of 4:1 at the stop valve. From theoretical calculation, the desired sidewall contact angle should be larger

978-1-940470-02-3/HH2016/\$25©2016TRF DOI 10.31438/trf.hh2016.93 Solid-State Sensors, Actuators and Microsystems Workshop Hilton Head Island, South Carolina, June 5-9, 2016 than $40^{\rm o}$ in order to generate positive capillary driving force to the liquid.

VLP sensing probe assembly and antibody sensing procedure



Figure 2: Schematics of (a) the evaporation-assisted VLP assembly and (b) the label-free antibody sensing process.

Fig. 2 shows the functionalization and biosensing processes, which are continuously monitored by the impedance microsensor based on the induced changes in the double layer capacitance. The VLP construct (VLP-FLAG) is genetically modified with both cysteine residues and FLAG-tag peptide sequences. In the sensor functionalization, the probes are assembled on the impedance sensor due to gold/cysteine interactions and van der Waals forces during evaporation at room temperature (Fig. 2a). Since VLP concentration increases during evaporation, an accelerated VLP assembly process and a denser VLP layer are expected.

During sensing, the binding event between the VLP sensing probes and antibody targets is directly analyzed. The triggering fluid is introduced from the small inlet in order to break the liquidair interfacial energy at the stop-valves; thus, the antibodies can continuously flow through the functionalized impedance sensor area. The target primary anti-FLAG antibodies in 5μ L of Trisbuffered saline (TBS) (1X) solution were introduced after sensor functionalization with VLPs (Fig. 2b). Antibodies show much lower dielectric constants compared to water. The substitution of water molecules by antibodies on the impedance sensor lowers the effective dielectric constants of both the double-layer and dielectric capacitances between electrodes.

Microfabrication and surface treatments

In the microfabrication of the sensor microsystem, interdigitated electrodes were fabricated using Cr/Au, the microfluidic components were made by patterning KMPR 1050 – a thick negatively photosensitive polymer layer. The challenges in the microfabrication is to simultaneously create hydrophilic sidewalls while keeping the substrate more hydrophobic.



Figure 3. Microfabrication process flow of the microfluidics integrated impedimetric sensing microsystem: (a) patterning of photoresist, (b) e-beam deposition of Cr/Au, (c) lift-off of photoresist to create interdigitated impedance sensor electrodes, and etching of SiO2 in BOE, and (d) creating capillary microfluidic layer through patterning of KMPR.

The fabrication process flow of the microsystem is demonstrated in Fig. 3. A 500 µm-thick silicon wafer with a 200 nm-thick SiO₂ passivation layer deposited using Plasma Enhanced Chemical Vapour Deposition (PECVD) was utilized as the substrate. Negative photoresist NR9-1500PY (Futurrex Inc., NJ, USA) was spin-coated at 3000 RPM for 30 seconds to form a 1.5 µm-thick photoresist layer. The photoresist was patterned using photolithography with an exposure energy dose of 280 mJ/cm² at 365 nm wavelength (Fig. 3a). After development, Cr/Au (20 nm/140 nm) was coated on the patterned wafer using NEXDEP Ebeam Deposition (Angstrom Engineering, Ontario, Canada) (Fig. 3b). The metals deposited on the photoresist were then removed in a lift-off process in acetone, creating the impedance sensor features on the wafer. A 45-second buffered HF etch was performed to expose the silicon (Fig. 3c) which is a more hydrophobic substrate material compared to silicon oxide.

The driving capillary force is mainly provided by the changing interfacial energy on the sidewall, which largely depends on the sidewall geometries. After impedance sensor fabrication, KMPR 1050 (MicroChem Corp., MA, USA) was spin-coated at 3000 rpm for 30 seconds, to form a 60 µm-thick layer. The KMPR

was soft-baked at 100 °C for 20 minutes before exposure at a dose of 1000 mJ/cm² at 365 nm with UV light. The wafer was then postbaked at 100 °C for 4 minutes. The KMPR development process was completed in SU-8 developer for 4 minutes followed by rinsing in isopropyl alcohol. The KMPR was then patterned on the impedance sensor and formed a high aspect ratio capillary microfluidic layer (Fig. 3d).

After fabrication, the integrated sensing microsystem was exposed to O₂ plasma (50 W, 0.5 Torr) for 5 minutes in order to remove residue generated during the microfabrication process as well as render the KMPR microfluidic layer hydrophilic. Interestingly, owing to the exposure of silicon substrate in the BOE etching, the substrate will show more hydrophobic behaviour compared to the sidewall (with the contact angle > 60°), which agrees with observations in literature [6, 7]. This will eventually contribute to a higher dependency of flow movement with the sidewall geometries, and a better controllability of the flow condition in the capillary microfluidic device.



Figure 4: Scanning electron microscopy (SEM) image showing the capillary microfluidic components after microfabrication.

Fig. 4 shows the scanning electron microscopy (SEM) images of the patterned capillary micropumps and stop valves. The microfluidic components show high resolution and high aspect ratio features after one-step photolithography. The wafer was then wet etched in buffered HF (BOE) for 45 seconds to expose the silicon layer at the bottom of microfluidic channels. This can improve the controllability of the capillary microfluidic sidewall to the flow. These microfluidic components facilitate autonomous sample delivery and localized VLP assembly in the impedance sensing area. The device surface was activated using O₂ plasma reactive ion etching (50W, 0.5 Torr) for 5 minutes to change the 80° inherent contact angle of KMPR to 6°, rendering the microfluidic devices hydrophilic.

RESULTS AND DISCUSSIONS

Accelerated VLP assembly and impedance monitoring

The VLP assembly morphology was investigated using SEM, and the assembly density and coverage were studied using the ImageJ image processing tool. In order to get a good contrast in SEM imaging, the VLP-coated surfaces have gone through electroless coating of Ni. The Ni coating method has been previously reported in detail in [8].



Figure 5: SEM image of the impedance sensor electrode after VLP assembly.

The SEM image in Fig. 5 validates the dense VLP coverage on the impedance sensor surface after evaporation-assisted assembly. During VLP assembly, the impedance was continuously measured. Fig. 5 also shows that the VLP-FLAG sensing probes form a dense functional layer on both the impedance sensor electrodes as well the spaces in between. The density of the evaporation enhanced VLP assembly in the open microfluidic channel is much higher compared to that previously achieved in a closed chamber [9].

The original SEM image of the VLP-FLAGs assembled on gold impedance electrodes and silicon surfaces (Fig. 5) was analyzed using ImageJ to understand the VLP surface coverage quantitatively. The original image was first processed by setting threshold values, and then converted into binary images where the white color represents VLP and the black color represents uncovered areas. The VLP coverage can then be calculated using the average grey value of each image. The specific VLP assembly on gold reaches 91% while the non-specific assembly of VLP on silicon is 41%. The non-specific assembly of VLP may result from van der Waals force during evaporation-assisted assembly process. Still, there are significantly larger numbers of VLP rods assembled on the gold surface compared to the silicon surface. This validated the effect of covalent binding between the gold and thiol groups on the cysteine modified VLPs.



Figure 6: Percentage changes of impedance in the presence or absence of VLPs.

Fig. 6 plots the percentage change in reactance ($\Delta |Z''|$) during VLP assembly, showing a rapid increase to 80% within the first 2 minutes. The impedance is then gradually increased to 120% within 6 minutes, a significant reduction in time compared to the previous VLP self-assembly process [2]. In the control experiment using buffer solution, the impedance increase is less than 5%, which may resulted from the fluctuation of ionic strength during evaporation of pure sodium phosphate buffer.

This impedance evolution has indicated a much more accelerated VLP assembly process through evaporation of VLP-containing buffer solution on the impedance sensor surfaces. Previously, without evaporation, the VLP can only saturate the sensor surfaces with the same areas with a 3-hour self-assembly process [9].

Rapid and label-free antibody sensing

Specific (target) and non-specific (control) binding antibodies were introduced onto the VLP-assembled impedance sensors through the capillary microfluidic system to evaluate the biosensing efficacy.



Figure 7: Percentage change in impedance for label-free sensing of target or control antibodies with increasing concentrations.

Fig. 7 shows the subsequent label-free antibody sensing using the VLP-functionalized impedance sensor. The sensor generated the maximum impedance shift over 30%, and the minimum impedance increase of 9% at the lowest anti-FLAG IgG (target) concentration of 10 ng/mL. In comparison, the maximum relative impedance change when introducing anti-rabbit IgG (control) is only 6%, which is attributed to non-specific bindings between the VLP sensing probe and anti-rabbit IgG.

The VLP-based sensor can reliably detect target antibody concentrations down to 10 ng/mL, equivalent to 55 pM antibody concentrations, over a 100-fold improvement compared to the 1.2 μ g/mL achieved with labeling previously [2, 9]. The sensor also showed dynamic response to a wide antibody concentration from 10 ng/mL to 100 μ g/mL. Thus, the results have demonstrated the great feasibility of the VLP-based impedance microsensors as selective, sensitivity and label-free biosensing platforms for ondemand biological analysis.

CONCLUSIONS

We developed a microsystem platform for accelerated VLP sensor functionalization and rapid biosensing. The sensor

microsystem utilized capillary action to autonomously facilitated VLP assembly on an impedance sensor within 6 minutes, a significant improvement compared to the 18-hour process in the prior art. With a densely covered VLP functional layer, the impedance microsensor was able to detect the presence of 10 ng/mL target antibody without relying on labels.

This work is the first demonstration of using capillary microfluidic devices to control the bioreceptors assembly in order to elevate the sensitivity for label-free biosensing. The integration of impedance microsensors and capillary microfluidics provides a microsystem solution for rapid and controlled sensor functionalization. This technology offers on-demand microsensor programming with specific bioreceptors according to actual needs shortly before the sensing, keeping the optimized biosensing efficacy. Meanwhile, with the growing inventory of binding peptides, the VLPs can potentially be developed as universal vehicles for expressing high density receptor peptides in miniaturized sensor system. Therefore, the integration of VLP with microsystems will greatly benefit future applications of pointof-care diagnostics.

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REDUCED GRAPHENE OXIDE SENSOR FOR QUANTIFICATION OF METABOLITES IN EXHALED BREATH CONDENSATE

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ABSTRACT

This work reports nitrite detection in EBC samples using reduced graphene oxide as the sensing material. Nitrite content in EBC can be a potential biomarker for inflammation in the respiratory system. The performance of the sensor was characterized in standard nitrite solutions using cyclic voltammetry and square wave voltammetry and then was applied to patient EBC samples. The sensor has a sensitivity of 0.21 μ A μ M⁻¹ cm⁻² in the range of 20-100 μ M and 0.1 μ A μ M⁻¹ cm⁻² in 100-1000 μ M nitrite concentration with a low detection limit of 830 nM in EBC matrix.

INTRODUCTION

Exhaled breath condensate (EBC) has been widely studied as a noninvasive source of biomarkers in chronic respiratory diseases. Although EBC has great potential as a source of biomarkers in many chronic respiratory conditions such as asthma, the low samples volume and concentrations of biomarkers within EBC present analytical challenges.

Exhaled breath contains gaseous materials such as nitric oxide and carbon monoxide, and EBC contains non-volatile compounds including small molecules such as water, hydrogen peroxide, nitrite and nitrate, and larger molecules such as eicosanoids, proteins, and even DNA [1]. Among these different substances, in this work we are interested in detection of nitrite content. The main source of nitrite in the inner lining of the airway is nitric oxide (NO) that is produced from L-arginine. In aqueous solution NO reacts rapidly with reactive oxygen species to form more stable form of nitrogen oxide such as nitrite (NO₂) and nitrate (NO₃) [2]. Increased level of NO has shown correlation to inflammatory conditions such as asthma and cystic fibrosis [3]. The increased level of NO in asthma has been suggested to be due to an increased expression of inducible NO synthase (iNOS) in the bronchial epithelium [4].

Common methods for detection nitrite are the spectrophotometric assay (Griess reaction), which is a fluorimetric method, chemiluminescence or ion chromatography [5]. The detection limit of the fluorimetric method is 0.1 μ M. The Griess method, however, has a higher detection limit compared to the chemiluminescence method, which has a lower detection limit in the nM range. Nitrite content detected in EBC is in the μ M range, which is compatible with these methods detection limits.

However, inspite of the low detection limit of these methods, often they require pretreatment steps on EBC samples to enable a suitable reaction or eliminate interfering compounds such as chlorine [6]. Usually electrochemical methods do not need this type of pretreatment and easily can be miniaturized to be used for point-of-care diagnostics and can potentially detect nitrite at a specific redox potential in real-time without interference from other compounds in the EBC matrix. However they do not have a detection limit as low as the optical techniques and require milliliters of sample volume.

Electrochemical detection of nitrite is based on either oxidation or reduction of the nitrite [7]. Oxidation of nitrite is usually preferred with the final product of NO₃ since main limitation of interference sources such as oxygen in reduction can be avoided. However, the problem of oxidation is its high over-potential [8] thus many attempts have been made to develop novel electrode materials in recent years [9]. Among of them, graphene based electrodes have been widely used due to small residue current, wide potential window, excellent chemical stability and easy functionalization. In this work we used graphene based electrodes for detection of nitrite. We fabricated a nitrite sensor using a combination of benefits offered by screen-printed electrodes and electrochemically reduced Graphene Oxide with low detection limit. To the best of our knowledge, this is first electrochemical sensor that directly measures nitrite in EBC samples with this level of sensitivity.

Another issue common in many graphene based electrochemical sensors, is that they usually use simple drop cast methods to deposit GO on top of the electrode which can result in very different results from sample to sample and has an agglomeration problem. Here we used a modified drop cast method to have a thinner layer on the electrode surface. Also by using the PDMS membrane as the electrochemical cell, voltammetric measurement of low volume EBC samples (100 μ l) can be easily done.

EXPERIMENTAL

To calibrate the sensor response we tested sodium nitrite spiked into acetate and phosphate buffer. Graphene oxide was prepared using hummers method. The morphology of graphene oxide was characterized using field emission scanning microscopy (SEM) with Shimadzu SS 550 SEM instrument. AFM was performed using Digital Instruments Nanoscope IV in tapping mode with standard cantilevers with spring constant of 40 N m⁻¹ and tip curvature <10 nm. FT-Raman spectra were recorded using Horiba Johin-Yvon Micro Raman Spectrometer, equipped with a microscope having a 532 nm laser as the source of its excitation beam. Electrochemical measurements were performed with PSTAT Princeton instrument in ambient condition. All potentials are with respect to Ag/AgCl reference electrode.

The steps of modification of the gold electrode with rGO are shown in figure 1. First, a 3 μ L aliquot of graphene oxide suspension that was synthesized from graphite powder by hummers method was dropped on the gold electrode surface. Then thin glass slide was placed on top of droplet and extra solution was removed and surface was dried under room temperature. Then GO thin layer was reduced electrochemically in acetate buffer with a pH of 5.5 using cyclic voltammetry between -1.6 to 0 V with scan rate of 25 mV for 30 cycle under continuous N₂ purging.

For fabrication of an electrochemical cell with microliters of

volume, a thin layer of PDMS was applied on top of the SPE electrode holder and then the top PDMS well containing a 8 mm hole was bonded onto it using O_2 plasma. During O_2 plasma treatment, the rGO layer and the connection pads were protected with glass slide.



Figure 1: Schematic of nitrite sensor fabrication steps: a) screen print electrode with working and counter electrode from gold, reference electrode from AgAgCl past. b) drop cast solution contains GO. c) Using glass slide on top of drop to have very thin layer of GO. d) Fabrication PDMS electrochemical cell for low volume EBC samples. e) Electrochemically reduction of GO.

RESULTS AND DESCUSSIONS

Characterization of Modified Electrodes

Characterization of GO morphology has been done with AFM and SEM. The level of reduction of GO also has been quantified with Raman spectroscopy. Figure 2a shows AFM images of GO on Si/SiO₂ substrate. In most areas there is a uniform flat GO layer with wrinkles and some agglomerated area that can be produce during vaporization of solvent which perhaps can be avoided using lower concentration of GO solution or drying in vacuum, however because we are interested in the electrochemical properties instead of the absolute electrical properties of GO, and because the electrochemical edges of GO are more sensitive than the flat layer, we intentionally did not try to remove these defects. However, this can be further studied using impedance spectroscopy to learn more about the effect of these defects on electrochemical behavior of the rGO layer.

Also an SEM image was taken from a larger area from the GO layer directly deposited on gold electrode (figure 2b). It shows that even in the gold working electrode with a roughness of several microns we have a covered uniform layer in most of the area. In comparison to common drop cast methods that usually are used in electrochemically-modified electrodes this method can give a much larger area of a few GO layers covered without agglomeration.

The efficiency of electrochemical reduction of GO also was investigated using raman spectroscopy. Representative data shows the average of three measurements in different areas of each sample. Figure 2C is the raman spectrum of GO layer before reduction and figure 2D shows the raman spectrum of rGO after 30 cycles of reduction. In carbon-based materials, the main features in the Raman spectra are the G and D peaks. These peaks arise from vibration of sp² carbon, appearing around 1580 and 1350 cm⁻¹ respectively. The overtone of the D peak appeared around 2700 cm⁻¹ is called 2D peak. Unlike mechanically exfoliated graphene, because GO is more disordered, thus its 2D band usually has low intensity. The peaks that can be used for their ability to distinguish between GO and rGO are G and D peaks and their ratio. Also the G peak of GO and rGO with respect to graphene and graphite gets shifted into higher frequencies (1598 cm⁻¹) because of defects. Even in the case of thermal reduction of GO usually I_D/I_G remains constant, the increased I_D/I_G ratio of rGO after electrochemical reduction has been reported in theliterature. In this study, this ratio exhibited a significant increase compare to GO (from 0.87 to 1.1). This shows restoration of sp² carbon and a decrease in the average size of sp² domains after electrochemical reduction of GO. An increase in the 2D peaks also suggests better graphitization [10].



Figure 2. a) AFM, b) SEM of GO thin layer. c) Raman of GO, d) Raman of rGO.

Electrochemical response toward nitrite

After we achieved properly reduced GO, we get through electrochemical performance of layer toward our analyte of interest, nitrite. As our final goal is fabrication of a miniaturized sensor that can be operate in ambient conditions, so even O₂ can react with analyte, we preferred to do all our experiment in presence of O₂ to understand how the sensor works in ambient conditions. As seen in the results of the EBC samples, the voltage of the oxidation peaks gets shifted to positive voltages, it is better to work in a lower over-potential thus acetate buffer with pH 6 was selected as electrolyte. Also pH in EBC samples of patients with inflammatory disease has been reported as acidic [11]. Thus pH of 6 is more similar to the pH of EBC samples obtained from asthma patients. Also the performance of rGO modified electrodes compare to SPE and GO deposited electrode was investigated. Figures 3A and 3B shows the response of GO and rGO modified electrodes in presence of 100

to 1000 µM nitrite respectively in 25 mV/s scan rate.

The performance of the fabricated electrodes was confirmed in standard electrolytes containing different concentrations of nitrite. This motivated us to test the electrode in EBC to understand the effect of the complex biological matrix, which was necessary before trying to measure basal levels of nitrite content in EBC.

We fabricated an electrochemical sensor capable of detecting nitrite content in real EBC samples in the μ M range. Cyclic Voltammetry and Square Wave Voltammetry were used to measure the current from different concentrations of nitrite solution in buffer spiked into EBC. Also, the analytical performance of the nitrite sensor was explored using square wave voltammetry (SWV). SWV measurements were conducted from 0 to 0.9 V. Figures 5A and 5B display the square wave voltammograms of nitrite in the range of (2-1000 μ M). The peak current in around 0.7 V depends on nitrite concentration. Figure 5C and 5D shows calibration curve based on SWV method.



Figure 3. Cyclic voltammetry of GO and rGO in a) buffer solution contains $100 \ \mu M$ and b) 1 mM nitrite.

Seven different EBC samples were tested to determine the accuracy of the sensors in detecting nitrite for different EBC samples. SWV was measured for each of the samples separately (figure 5a). The slight difference between the oxidation potential may be because of the different complex matrix of EBC from patient to patient. Based on their oxidation current; their concentrations were compared to data that was collected with ozone based chemiluminescence [13]. Figure 6B shows the calibration curve based of current respect predicted

concentration using chemiluminescence method. As can be seen there is a linear relationship between them.

CONCLUSION

In this study the enzyme-free nitrite sensor was fabricated using the unique properties of Graphene combined with SPEs. The use of the PDMS membrane allows us to measure low volumes of EBC samples. After optimization of various parameters in purified buffer solution, we tested real EBC samples. We obtained the calibration curve using various spiked nitrite concentrations in EBC. The sensitivity of the sensor is 0.21 µA μ M⁻¹ cm⁻² in the range of 20-100 μ M and 0.1 μ A μ M⁻¹ cm⁻² in 100-1000 µM with detection limit of 830 nM. The data for a small number (7) of real EBC samples was also collected and compared with chemiluminescence methods. The sensor was demonstrated to measure nitrite content with high accuracy in the µM range of concentration which for most cases is the typical range of nitrite in EBC samples. Detection was even demonstrated in the nanoMolar range, however enabling robust operation requires further improvement. This can be done either through modification of the geometry of the sensor or through use of graphene-nanoparticle composites.



Figure 4. a,b) SWV of different concentrations of nitrite spiked in EBC sample. c,d) calibration curves. Inset of c is calibration curve for lower concentration range.



Figure 5. a) SWV of seven different EBC samples, b) calibration curve using photoluminescence nitrite concentration data.

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THE BIOSENTINEL BIOANALYTICAL MICROSYSTEM: CHARACTERIZING DNA RADIATION DAMAGE IN LIVING ORGANISMS BEYOND EARTH ORBIT

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ABSTRACT

We report details and initial lab test results from an integrated bioanalytical microsystem designed to conduct the first biology experiments beyond low Earth orbit (LEO) since Apollo 17 (1972). The 14-kg, 12 x 24 x 37-cm³ BioSentinel spacecraft will assay radiation-responsive yeast in its science payload by measuring DNA double-strand breaks (DSBs) repaired via homologous recombination, a mechanism common to all eukarvotes including humans. S. cerevisiae (brewer's yeast) in 288 microwells are provided with nutrient and optically assayed for growth and metabolism via 3-color absorptimetry periodically during the ~ 12-monthlong spaceflight mission. BioSentinel, one of several secondary payloads to be deployed by NASA's Space Launch System (SLS) as it carries Exploration Mission 1 into space in 2018, will receive commands and telemeter data to Earth from tens of millions of km.

INTRODUCTION

Small autonomous satellites, called nanosatellites or cubesats, simultaneously reduce cost and increase accessibility for spacescience experiments [1]. Leveraging and integrating advances in nano-, micro-, and miniature technologies in fields from biotechnology to microfluidics to telecommunications, small satellites are being developed worldwide by over 100 universities, numerous small ventures, several large aerospace companies, multiple developing nations, and all major space agencies. Despite their diminutive size, they support complex science [1-4]. By coupling autonomy and telemetry in these space "free flyers", near-real-time experimental data are provided from environments that may be challenging or costly for human missions [2-4].

For life to live and thrive beyond low Earth orbit (LEO) requires understanding and managing multiple unique perturbations. While the International Space Station (ISS) and other orbiting spacecraft provide reduced- (including) micro-gravity environments, the complex radiation environment of interplanetary space comprises many particle types, each with its own energy spectrum and resultant effective biological dose rate. This complex environment is not reproduced by any terrestrial facility: radiationexposure facilities typically provide one or two particle types at one or two energies simultaneously; a dose equivalent to months in outer space is delivered in a matter of minutes to hours. Because of the capacity of biological systems for self-repair and cell-cell communication, the response to acute vs. chronic dosage cannot be assumed to be identical, even for the same total dose. Thus, complete radiation-response characterization necessitates chronic exposure while monitoring live organisms beyond the shielding effects of Earth's magnetosphere for periods of months [5].

BioSentinel's strategy is that of 'the canary in the coal mine': assess health risk for long-duration human missions beyond LEO using a model organism with important similarities to humans, in this case the mechanism by which DNA double-strand breaks are repaired [6]. Of the categories of biological damage created by low-to-moderate levels of chronic radiation exposure, damage to DNA is a major concern: resulting mutations can cause cancer.

In order to better understand how biological effects can be related to physically measured radiation dosage in the space environment [7], and to help validate biological radiation-effect models, BioSentinel will also carry two types of solid-state radiation sensor: a total ionizing dose (TID) dosimeter and a miniaturized linear-energy-transfer (LET) spectrometer (details below).

BioSentinel's biological radiation experiment utilizes multiple independently actuated sets of microfluidically-serviced microwell arrays to monitor DNA DSBs, measuring their impact on viability as well as their rate of successful repair. Three strains of Saccharomyces cerevisiae are utilized to accomplish this: (1) a wildtype, serving as control, which is minimally affected by the dose range anticipated during the BioSentinel mission; (2) a mutant strain, $rad51\Delta$, which is defective in DNA double-strand break repair by homologous recombination and, therefore, more likely than wildtype yeast to be made non-viable by DSBs; (3) an engineered strain with a genetic defect the prevents it from producing an amino acid (e.g., leucine) essential to its growth. This so-called biosentinel strain of yeast, when fed with growth medium lacking leucine, neither reproduces nor exhibits the levels of metabolic activity associated with growth; however, when its DNA DSB repair process is activated, for example by a high-energy radiation damage event, that genetic defect can be simultaneously repaired. Thus, if even one yeast cell in one microwell suffers a DSB from one radiation 'hit' and successfully repairs its genetic defect, this cell acquires the capacity to synthesize the missing amino acid and, through exponential reproduction, generates readily measurable metabolic activity and population growth in that microwell.



Figure 1: BioSentinel spacecraft views: exploded (left) and assembled (upper right). Solar panels are pointed toward the sun and radio antennas toward Earth by the integral guidance, navigation, and control unit. The bio payload container occupies ~ 4 L.

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DESIGN AND EXPERIMENTAL DETAILS

Figure 1 shows a solid model of the BioSentinel spacecraft and its major subsystems and components. Its deployment from the SLS launch vehicle upper stage will place it in a heliocentric, so-called 'disposal orbit' (never to return to Earth) in the range of 0.93 - 0.97 astronomical units (AU) from the sun and well beyond Earth's magnetic field and radiation belts. Due to its orbit, BioSentinel's orbital velocity will cause its distance from Earth to increase with time: after 12 months, *BioSentinel* is expected to be $\sim 10^8$ km away. It is therefore designed for operation in deep space and features a deployable, gimbaled triple-junction solar panel array that generates up to 35 W; an integrated guidance, navigation, and control (GN&C) unit, including 3 orthogonally oriented momentum wheels, a star tracker, multiple sun sensors, and an inertial measurement unit; a cold-gas micropropulsion system for detumbling upon initial deployment and for desaturating the momentum wheels; lithium ion batteries and an electric power management system; a radio/transponder and multiple 'patch' antennas for communication with NASA's Deep Space Network of antennas; and overall electronics tolerance through a combination of part selection, localized shielding, and 'watchdog timers' to the radiation dose anticipated in one year or more in deep space. These subsystems, together with the mechanical structures, occupy \sim half of the total spacecraft volume, leaving ~ 4 L volume for the experimental-biology-and-radiation-monitoring payload.

Figure 2 shows one of *BioSentinel's* 18 microfluidic "minicards", fabricated by precision machining, laser/blade cutting, and lamination with pressure-sensitive adhesive (PSA). Each well is 3.5 mm in diameter x 10 mm deep, for a contained volume of 100 μ L/well. Integral "track-etched" (Whatman Nucleopore) translucent polycarbonate membranes (1.0 μ m pore diameter; a single common layer covers the tops of the minicard microwells and a second common layer covers the bottoms) provide crosstalkfree inlet/outlet filtration, confining the various strains of yeast to their respective microwells while permitting initial microwell filling from the dry state (displacement of air), exchange of medium, and high-precision light-transmission measurements.



Figure 2: Exploded (left) and assembled (upper right) views of the microfluidic minicard with integral thermal control and optical measurement components. Bottom right: photo of a functional prototype fluidic minicard; 15 wells contain alamar blue and one well contains the same dye that has turned pink due to yeast metabolic activity. (For microwell cross section, see Fig. 3.)

Minicard bodies and microchannel-defining layers are made of polycarbonate, as are the Nucleopore membranes; the acrylic PSA maintains bonding strength at temperatures above 130 °C. Thus, the entire fluidic minicard can be sterilized by autoclaving at 121 °C, a process not typically tolerated by thermoplastic microfluidic systems but which is simple and provides reliable sterility.

After minicard sterilization, yeast cells are dried onto microwell walls in preparation for spaceflight; cards are sealed and integrated with a fluid-supply manifold (Fig. 2) and the entire system is integrated, including reagents and media contained in fluoroethylene polymer bags (American Fluoroseal), an integrated fluid-delivery system, control electronics, and MEMS pressure (Freescale) and humidity (Sensirion) sensors. The integrated biosensor system is sealed inside the hermetic bio payload container.

When properly dried, yeast can survive for months to years. This is critical because the fully integrated *BioSentinel* spacecraft will be delivered to the launch provider several months prior to its launch into space; also, the mission itself has a nominal one-year duration, with the expectation that one or more minicards will be activated (i.e., their yeast will be grown) near the end of this time.

To help ensure the longevity of the dried yeast while awaiting activation, *BioSentinel* includes a 'refrigeration' feature. One large face of the spacecraft (Fig. 1) is pointed, whenever feasible, towards dark space; it is coated to enhance thermal emissivity. The integrated fluidic minicards, Fig. 2, are topped by a conformal thermally conductive gasket pressed into contact with the inside face of the container that is thermally coupled to the cold spacecraft face. Thermal modeling suggests a temperature of ~ 4 - 8 °C can be maintained within the unheated minicards—which are monitored and heated only if necessary to avoid freezing—dependent on the details of the orbit and spacecraft operation. Only the minicard(s) with wetted yeast in the active growth phase are thermostatted at the higher growth temperature, ~ 20 °C.

Figure 3, a cross-section of a single fluidic well, shows the optical measurement and thermal control components. Three surface-mount LEDs provide illumination (570, 630, and 850 nm, sequentially); absorbances are calculated from the output of a dedicated intensity-to-frequency light sensor (ams/TAOS) at each well (the optical system has zero moving parts). Minicard cover layers are optical-quality, permeation-resistant poly(cycloolefin) (1-mm-thick Zeonor injection-molded plaques).

The lower part of Fig. 3 shows the design of a nine-minicard manifold that provides fluid in/out connections, houses solenoid valves and check valves, and supports an optical calibration cell (visible at the near end of the lower bank of minicards). This cell is used to measure the absorbance of alamar blue to confirm its concentration and provide an optical calibration datum at all three wavelengths for each minicard at its time of filling.

The 16-well fluidic minicards will be activated either singly or in pairs across the duration of the one-year mission in deep space in order to quantify time-dependent accumulated DNA DSBs. Fluid filling and metering are accomplished by a miniature peristaltic pump (Takasago) and 3-way solenoid valves (Lee Co.). *S. cerevisiae* grow after microwells have been filled with a 9:1 mix of synthetic complete (SC) growth medium (minus one deliberately omitted amino acid, as described above) and alamar blue indicator dye. This dye is chemically reduced from dark blue to (fluorescent) pink, then to colorless, due to cellular metabolic activity that produces reducing agents such as NADH and FADH [2,4].

In addition to its bioanalytical microsystem, *BioSentinel* includes two physical radiation sensors to measure total ionizing dose and linear energy transfer. Given the shielding provided by the spacecraft structure, the estimated one-year accumulated ionizing radiation dose inside the payload is estimated at 5 Gy. The

TID dosimeter (Teledyne uDOS001; volume 9.2 cm³; mass 20 g) is a silicon-based solid-state charge-integrator-based device that has been utilized for multiple space missions. It features a resolution of 140 nGy (the dose received, on average, in ~ 0.9 sec during the *BioSentinel* mission) and full-scale of 400 Gy.



Figure 3: **Top:** Cross section of one fluidic microwell with integral filter membranes and connecting microchannels. Yeast dried onto walls grow and bud when medium is introduced, displacing air through porous membranes. Three LEDs per well, with an intervening thin-film polymer diffuser, provide green, red, and near-infrared (IR) illumination to monitor metabolic activity and track yeast growth via an intensity-to-frequency detector at the bottom of each well. Patterned metal-on-kapton heaters with aluminum thermal spreader plates maintain uniform, stable growth temperature ($\sim 20 \pm 1 \, ^{\circ}$ C) via closed-loop control; a Pt resistance-temperature device is embedded in each minicard. **Bottom**: Nine fluidic minicards will be integrated and manifolded together for spaceflight; two such sets (288 microwells in total) will be included in the hermetically sealed 4L bio payload container (Fig. 1).

The LET spectrometer, developed and provided by Johnson Space Center's Radworks group, is based closely on a predecessor that has flown on the ISS as well as the first test flight of NASA's Orion spacecraft in 2014. The charge-coupled Si device records the passage of each high-energy particle as a track or 'blob' of pixels with accumulated charge (Figure 4). The associated controland-measurement electronics and software convert the track/blob data into the energy deposited by each high-energy particle per unit length of Si traversed, thus defining its potential to cause (biological) damage. Closed-loop feedback adjusts the integration time to keep frame pixel occupancy \leq 3%, making deconvolution of the tracks (more) straightforward. The sensor measures radiation over the 0.2 to 300 keV/µm LET range, which is most relevant for biological damage, using an active chip volume of 59 mm³. On-board software computes the LET value of each radiation event (particle hit) and increments the count in an appropriate bin; each of the 256 counting bins is defined by an LET width of ~2.9% of its center value. The binned event counts are reported hourly by the LET spectrometer to the spacecraft's main processor for storage and eventual telemetry to Earth, providing an hourly 'space weather report' of the types and numbers of ionizing particles encountered by *BioSentinel*.



Figure 4: Typical radiationevent data frame recorded by the CCD-based LET sensor; each locus of points is associated with the passage of a single particle or ray.

The inclusion of TID and LET sensors enables the BioSentinel biological experiment to respond to changes in space weather. In particular, during a solar particle event (SPE), radiation flux can spike to a thousand times nominal levels or more; it is therefore a goal of the BioSentinel mission to measure the effects of such an event, should it occur during the year-long mission. Yeast (and other cells) are generally significantly more damaged by radiation when hydrated than when dry, at least in part because OH• species, created from water by radiation, cause oxidative damage. With 18 fluidic minicards to be tested periodically across 12 months, and a growth duration measured in hours, there may or may not be a wet, actively growing minicard ready to respond to the enhanced flux of an SPE. Therefore, onboard algorithms will monitor both TID and LET outputs and, should the flux rise sufficiently above background, a "special" minicard will be activated so that its cells are wet and ready to grow as the main part of the SPE arrivestypically a fraction of one to a few hours after radiation levels initially begin to surge.

PRELIMINARY LABORATORY RESULTS

Figure 5 shows biological growth and metabolism results from laboratory testing of a flight prototype of the integrated optical/fluidic/bioanalytical system. The results are for wildtype yeast grown in SC medium at room temperature in the presence of "1x" alamar blue viability dye. As growth progresses, green absorbance increases as the bright pink color of reduced alamar blue appears, then decreases as pink fades to colorless. Red absorbance decreases as the blue dye turns pink, then increases due to cell population growth. Near-IR absorbance, unaffected by all forms of the dye, tracks cell population due to the linear relationship between turbidity and cell number. 16 such curves are produced simultaneously from one growing fluidic minicard.



Figure 5: Growth curves (*left*) recorded using spaceflight optical/fluidic/thermal prototype shown in the photograph (*right*) for wildtype (WT) S. cerevisiae with added alamar blue (*filled symbols*) and cell-free control microwells (*open symbols*); LED wavelengths are as indicated.

CONCLUSIONS

Directly studying biology's response to the interplanetary space radiation environment has never been attempted apart from a few costly, human-operated beyond-LEO missions. Robust, autonomous bioanalytical microsystems compatible with small satellites are poised to change this paradigm. *BioSentinel* is supported by NASA's Advanced Exploration Systems division.

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2DEG-HEATED AlGaN/GaN MICRO-HOTPLATES FOR HIGH-TEMPERATURE CHEMICAL SENSING MICROSYSTEMS

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ABSTRACT

Fully-suspended AlGaN/GaN micro-hotplates that leverage self-heating of the two-dimensional electron gas (2DEG) have been modeled, micro-fabricated, and characterized at elevated ambient temperatures (from 25°C to 600°C in air). An input power of ~75 mW heated the micro-hotplates to approximately 270°C from 25°C ambient conditions. In addition, finite element analysis (FEA) showed high-temperature uniformity across the micro-hotplate (<1% variation) and fast transient response times (~ 2 ms rise and fall times). These results support the use of the AlGaN/GaN-on-Si platform for high-temperature activation of chemical sensing catalysts and in-situ chemical sensing within high-temperature environments (e.g., combustion exhaust, industrial process, and downhole).

INTRODUCTION

The global market of chemical sensors is forecasted to reach about 31 billion US dollars by 2020 [1]. Data collected by chemical sensors can aid in emission control, energy savings, environmental monitoring, and catastrophe prevention [2-5]. For chemical sensing, a functional sensing layer (catalyst) transduces the chemical signals into electrical signals, which are then detected by the underlying sensor platform. To ensure reliable signals and reduced baseline drift, the sensing layer needs to be regenerated (burned off) periodically at elevated temperatures. In addition, many functional sensing materials exhibit increased sensitivity at elevated temperatures. For example, ZnO nanorods demonstrated enhanced sensitivity to hydrogen at approximately 250°C [6]. To sustain elevated temperatures during operation, a heating element is often integrated into the sensor system. Micro-hotplates, which are suspended and thermally isolated (reduced power consumption) structures with embedded heaters, are often utilized.

Commonly used micro-hotplates are composed of silicon oxide/nitride membranes and metallic heaters. Such systems suffer from reliability issues caused by electro-migration of metal atoms [7]. In addition, localized stress and voids in metal traces often lead to premature rupture of the membranes and drift of the heating characteristics [7]. In addition, micro-hotplates with metal heaters often employ complex heater geometries to achieve uniform temperature distribution [8]. Thus, micro-hotplates made from doped semiconductor materials (e.g., Si and SiC) have been used to mitigate the reliability issues caused by metal electro-migration. Additionally, as the semiconductor plate itself serves as the heater, such micro-hotplates can produce uniform temperature distributions using simple layout geometries. For high-temperature chemical sensing (> 300°C), self-heated silicon carbide (SiC) micro-hotplates have been developed, but custom substrates and advanced membrane release chemistry are required [9], which makes integration with electronics challenging.

In contrast, the self-heated AlGaN/GaN micro-hotplates reported here utilize commercial AlGaN/GaN-on-Si wafers and a common Si release process (XeF₂), which supports monolithic integration with GaN electronics and high-temperature operation. Monolithic GaN-based microsystems are appealing for sensing applications in harsh environments, such as combustion exhaust



Figure 1: The influence of heater arm geometry on heating efficiency and mechanical robustness (out-of-plane displacement per unit perpendicularly applied pressure) of four-arm micro-hotplates studied by analytical modeling and FEA.

pipes, industrial manufacturing processes, and oil & gas wellbores, due to the inherent temperature- and chemical-tolerance of GaN. AlGaN/GaN heterostructures have been commercialized for high-frequency and high-power electronics thanks to the highly conductive two-dimensional electron gas (2DEG) at the AlGaN/GaN interface [10]. In addition, the 2DEG is sensitive to surface charges and thus can be used for chemical sensing applications [11-12]. Moreover, due to the wide bandgap of GaN, AlGaN/GaN devices have demonstrated successful operation up to 600°C [13]. Furthermore, GaN energy harvesters and resonators [14-15] leveraging the piezoelectric properties of GaN can also be integrated on a single chip to make self-powered wireless sensing nodes. This paper presents the modeling, micro-fabrication, and characterization (interpolation-based) of the first 2DEG-heated AlGaN/GaN micro-hotplate architecture reported in the literature. In addition, uncertainty analysis of the characterization method and future work are discussed.

DESIGN AND MODELING

Analytical modeling and finite element analysis (FEA) were used to predict the effect of micro-hotplate geometry on heating efficiency (maximum temperature rise per unit input power) and mechanical robustness (maximum out-of-plane displacement per unit perpendicularly applied pressure) (Fig. 1). The results reveal that increasing the ratio of the heater arm length (L) to width (W) can enhance the heating efficiency but reduces the mechanical robustness of the micro-hotplate. It should be noted that the mechanical robustness falls at a much faster rate than the heating efficiency improves. To ensure that the micro-hotplate has sufficient mechanical robustness. L/W = 5 was chosen for this work (L = 100 μ m and W = 20 μ m). For this work, a spiral four-arm layout was chosen due to the fact that given the same L/W, a spiral four-arm micro-hotplate (Fig. 2a) spans a much smaller area than a conventional four-arm design (schematic shown in the inset of Fig. 1) while maintaining a similar heating efficiency and mechanical robustness. To ensure reliable operation, the thermal stress in the spiral four-arm micro-hotplate

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Figure 2: Optical image of a microfabricated AlGaN/GaN micro-hotplates (a), and infrared (IR) image of a micro-hotplate showing the temperature profile during operation (b).

used in FEA.
130 W/(m-K) [16]
815 S/m for design phase;
Temperature dependent for
micro-hotplate characterization
300 GPa [17]
0.23 [18]
Temperature dependent [19]
491 J/(kg-K) [16]
$6.15 \times 10^3 \text{ kg/m}^3 \text{ [20]}$

should be smaller than the yield stress of the GaN membrane. For the geometry of L = 100 μ m and W = 20 μ m, the maximum von Mises computed by FEA was approximately 630 MPa when the plate is heated to 330°C (ambient temperature at 25°C). This is lower than the yield stress of GaN at that temperature (> 1000 MPa) [21]. The maximum stress occurs at the intersection of the plate and the heater arms.

Detailed FEA (COMSOL Multiphysics®) of the spiral four-arm AlGaN/GaN micro-hotplate was conducted taking into account the nominal microfabricated geometry (GaN membrane thickness = $3 \mu m$, heater arm length = $100 \mu m$, heater arm width = 20 μ m, and central plate size = 50 μ m by 50 μ m). In addition, the temperature-dependent properties (electrical conductivity of 2DEG and coefficient of thermal expansion) were utilized. The material properties of the micro-hotplates modeled in FEA are summarized in Table 1. Using these properties, the temperature profiles of the micro-hotplate (nominal geometry) were computed and the temperature profiles were visualized. The FEA results showed very high temperature uniformity across the main plate (~1% variation) in agreement with the infrared (IR) images taken during operation Fig. 2b. In addition, the FEA transient simulation results (Fig. 3) reveal that the micro-hotplate has fast heating (rise) and cooling (fall) transient response times (~2 ms).

MICROFABRICATION

The micro-hotplates were fabricated on commercial GaN-capped AlGaN/GaN-on-Si wafers grown by metal-organic chemical vapor deposition (MOCVD). The wafer had a sheet resistance of ~409 Ω / \Box at room temperature. The electron mobility was measured to be $\sim 1400 \text{ cm}^2/\text{V-s}$ and the sheet electron density $\sim 0.9 \times 10^{13}$ cm⁻². The fabrication process (Fig. 4) starts with a mesa etch using a chlorine/boron chloride (Cl₂/BCl₃) plasma to define the 2DEG heater geometry. Then, Ti/Al/Pt/Au metal contacts were e-beam evaporated and pattered with lift-off. A rapid thermal anneal at 850°C for 35 seconds in a N2 ambient was conducted to



Figure 3: Simulated transient responses of spiral four-arm AlGaN/GaN micro-hotplate. In the simulation, a heating voltage (15V) was applied at t = 0, and removed at t = 3 ms.



Figure 4: Process used to micro-fabricate the fully-suspended AlGaN/GaN micro-hotplate with 2DEG heater.

make the contacts Ohmic and improve reliability at high temperatures [22]. Silicon dioxide (SiO₂) was then deposited by plasma-enhanced chemical vapor deposition (PECVD). The GaN and buffer layers were patterned with Cl₂/BCl₃ plasma using the PECVD SiO₂ hard mask to define the micro-hotplate membrane shapes. The SiO₂ was stripped with buffered oxide etch (BOE) after the plasma etch. Then, alumina (Al₂O₃) was deposited with plasma-enhanced atomic layer deposition (PE-ALD) for passivation of the micro-hotplate surface. Al₂O₃ was later patterned with BOE to expose metal contacts for interconnection, and to expose the gaps between the 2DEG heater arms and the central plate for later XeF₂ release of the membrane. The micro-hotplate membrane was released from Si substrate with XeF₂ vapor. The optical image of a fabricated AlGaN/GaN micro-hotplate is shown in Fig. 2a with a surface temperature map during operation (~90 mW power input) imaged by an infrared camera (Fig. 2b).



Figure 5: Interpolated heating characteristics of three microfabricated AlGaN/GaN micro-hotplates at ambient temperature of 25°C. The inset shows the simulated temperature profile of the micro-hotplate.

CHARACTERIZATION

The AlGaN/GaN micro-hotplates were electrically characterized at various ambient temperatures (from 25°C to 600°C) in air using a high temperature probe station (Signatone Inc.) and a semiconductor parameter analyzer (Agilent B1500A). Interpolation of electrical measurements and FEA simulation results were used to approximate the maximum plate temperatures. The characterization procedure is as follows:

- The electrical resistance values of several micro-hotplates were measured at various ambient temperatures up to 600°C in air.
- A numerical fit of the measured resistances versus ambient temperature was obtained using a least-squares quadratic model.
- The temperature-dependent electrical resistivity was calculated from the fitted quadratic model and fed into the FEA model (the model is shown in the inset of Fig. 5).
- The FEA model was used to compute the maximum plate temperatures (T_{max}) achieved by applying various voltages at various ambient temperatures, as well as the total current flowing through the micro-hotplate. The resistance (R) of the micro-hotplate was then calculated by dividing the applied voltages with the corresponding currents.
- The R versus T_{max} (R-T_{max}) curves at various ambient temperatures were obtained, and fitted with least-square linear regression models.
- The resistance values of the micro-hotplates under various applied voltages at various ambient temperatures were measured using the high-temperature probe station and SPA.
- The maximum plate temperatures of the micro-hotplates at different ambient temperatures were interpolated using the fitted R-T_{max} curves and the measured resistances.

The characterization results using the interpolation method are shown in Figs. 5 and 6. As shown in Fig. 5, an input power of ~75 mW generates a maximum plate temperature of about 270°C in 25°C ambient temperatures. This corresponds to a heating efficiency of about 3°C/mW, which can be further increased by geometry optimization (Fig. 1). The fabricated micro-hotplates demonstrated successful heating operation at elevated ambient temperatures (300°C to 600°C) in air using a high-temperature probe station (Fig. 6). As the ambient temperature is increased, the maximum temperature rise on the plate with respect to the ambient



Figure 6: Operation of multiple AlGaN/GaN micro-hotplates at elevated ambient temperatures up to 600°C in air.

Table 2: Summary of uncertainty analysis of the maximum temperature values obtained from the characterization procedure.

Error source	Estimated Error		
The error induced by the	Average ~ 3.4%		
simplifying assumptions made in	(Max ~9%)		
FEM			
FEM convergence	<0.1%		
Error in quadratic fit of measured	Average ~2.6%		
resistances	(Max ~9%)		
Uncertainty induced by the	~0.13%		
variation in released membrane			
width (10 µm variation)			
Error induced by the uncertainty	Approx. zero		
in thermal conductivity and	(Fig. 9)		
geometric parameters			
Neglecting contact resistance	<0.25%		
SPA measurement error	Approx. zero		
Chuck temperature reading error	<4% (1°C error at		
	25°C)		
Error in linear fit of R-T _{max}	Average ~0.15%		
calibration curves	(Max ~ 0.2%)		
Total	Average ~11%		
	(Max ~22%)		

temperature is decreased because the mobility of electrons in 2DEG is decreased due to increased phonon scattering [23]. Thus, the electrical conductivity of 2DEG is decreased and less current can be pumped through into the micro-hotplates for self-heating at higher temperatures. Nevertheless, T_{max} of ~680°C was achieved in 600°C ambient with an applied voltage of 30 V (Fig. 6).

UNCERTAINTY ANALYSIS

The uncertainty sources for the maximum plate temperatures obtained from the characterization procedure are summarized in Table 2. The total average uncertainty was estimated to be approximately 11%. The three most significant sources of uncertainty are 1) neglecting heat loss to air and radiation in FEA, 2) least-square quadratic fitting of measured resistance values with respect to ambient temperatures, and 3) the error in chuck temperature readings. It should be noted that the uncertainties in the thermal conductivity, thickness and 2D layout geometry of the micro-hotplate do not induce errors in the FEA-computed R-T_{max} curves. This is validated by the FEA: the (R, T_{max}) pairs resulting

from different thermal conductivities, thicknesses, and L/W values all fall on one single curve, which is the one generated by varying the voltages applied to a nominal device geometries with material properties summarized in Table 2.

DISCUSSION AND CONCLUSION

2DEG-heated AlGaN/GaN micro-hotplates have been modeled, microfabricated, and characterized in this work. A heating efficiency of approximately 3°C/mW has been achieved by the device prototypes, which is a suitable activation power for many sensing applications. To further improve the heating efficiency, longer and thinner heater arms can be utilized. In addition, to preserve mechanical robustness while enhancing heating efficiency, spring-shapes (or "zigzag" shapes) heater arm layout can be employed. A reduction in heating voltage for a certain maximum plate temperature can be achieved by reducing the sheet resistance of the AlGaN/GaN heterostructure through material optimization. Sheet resistances values of near 300 Ω / and lower have been reported in literature [24]. In conclusion, 2DEG-heated micro-hotplates have been successfully fabricated and operated in air and demonstrated heating temperatures as high as 680°C (from 600°C ambient temperature), supporting the development of monolithic AlGaN/GaN microsystems for in-situ chemical sensing in high-temperature environments.

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DEVELOPMENT OF A MEMS PRECONCENTRATOR (PC) - GAS CHROMATOGRAPH (GC) FOR THE SPACECRAFT ATMOSPHERE MONITOR FOR ISS AND ORION

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ABSTRACT

In this paper, we report a MEMS preconcentrator (PC) - gas chromatograph (GC) that is a crucial part of the Spacecraft Atmosphere Monitor (S.A.M.). The S.A.M. is a highly miniature gas chromatograph - mass spectrometer (GC-MS) for monitoring the atmosphere of crewed spacecraft for both trace organic compounds and the major constituents of the cabin air. The S.A.M. instrument is the next generation of GC-MS, based on JPL's Vehicle Cabin Air Monitor (VCAM), which was launched to the International Space Station (ISS) in April 2010 and successfully operated for two years [1, 2]. The S.A.M. employs a unique MEMS PC-GC technology that replaces the macro PC-GC unit in the VCAM. We report herein the current progress of the MEMS PC-GC for the S.A.M. instrument.

INTRODUCTION

The S.A.M. instrument is JPL's atmospheric monitoring module for ISS that is based on a GC-MS system consisting of a Quadrupole Ion Trap MS (QITMS) interfaced with a MEMS PC-GC unit and a small gas reservoir. The S.A.M. is intended for assessing trace volatile organic compounds and the major constituents in the atmosphere of present and future crewed spacecraft. As such, S.A.M. will continuously sample concentrations of major air constituents (CH4, H₂O, N₂, O₂, and CO₂) and report results in two-second intervals.

The S.A.M. offers further reduction in the overall instrument size to a 9.5" x 9" x 7.5" envelope, as compared to 10.8" x 18.1" x 20.4" for VCAM. This reduction in size is mainly achieved using the MEMS PC-GC unit as well as simplified electronics and other peripherals.

Trace gas analysis is based on a unique MEMS PC-GC module developed through NASA SBIR/PIDDP projects and built at JPL, which enables us to proceed with reduced power and small carrier gas consumption. A few MEMS PC-GCs have been developed before [3-5] but none of them has been devised to work with a QITMS, which requires low carrier flow (less than 0.1 sccm) and high vacuum-tight design (10⁻⁸ Torr for the JPL QITMS). Many efforts have been made to build miniaturized PC-GC systems for various applications, including homeland security and many on-site industrial applications, but no NASA mission has used MEMS PC-GC technology for life support, human exploration, or planetary research. JPL has ramped up the MEMS PC-GC technology readiness level for ISS and Orion, which significantly reduces the footprint, consumables, and power.

In this paper, we present current progress of the MEMS PC-GC system for the S.A.M. instrument.

SYSTEM CONFIGURATION

Figure 1(a) illustrates a conceptual schematic of the three MEMS devices – PC, microvalve (MV), and microcolumn (MC) that connects to the QITMS. Their chip footprints, shown in Fig. 1(b), are only 14 x 14 mm² with a 1-2 mm thickness, and they only weigh a few milligrams. To ensure that all of these devices operate properly with the QITMS, it is necessary that all the devices, their packages and fittings are vacuum-tight. Vespel housings with

ValcoTM fittings are designed and manufactured to meet this sealing requirement. Figure 1(c) shows a 3D image of a three-layer Vespel block (PC top/Via/MV bottom) and indicate where PC and MV are inserted. By using a Via block, the PC and MV chips are separated and can be treated independently. The Via block also provides thermal isolation between the devices. Figure (d) shows an addition of the MC bottom block with the MC chip after the assembly of PC top/Via/MV bottom. This will finalize the complete assembly of PC, MV, and MC with the housing blocks.



Figure 1: (a) A conceptual schematic of the MEMS PC-GC that connects to the QITMS. (b) Photographs of the PC, MV, and MC devices compared to a U.S. penny. (c) A 3D image of three-layer Vespel blocks – PC top/Via/MV bottom – as well as where PC and MV are inserted. (d) An addition of the MC bottom block with the MC chip after the assembly of PC top/Via/MV bottom.

PRECONCENTRATOR (PC)

The PC uses an embedded silicon heater that employs a siliconon-insulator wafer. Due to the thermally isolated heater design as shown in Fig. 2(a), a two microliter PC chamber can be heated to 250°C in less than a second. Flash heating to high temperature is a key factor for a high gain PC and separation resolution in the GC downstream. Carboxen 1000 adsorbent particles are packed in the PC chamber in two layers.

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Figure 2: (a) Thermally isolated Si heater in the middle chamber where Carboxen particles a adsorbents packed (left); microposts for attaching micro/nano adsorbents (middle); Carboxen 1000 particles to be packed into the middle chamber that has no micropost (right). (b) Heater layer that is made of a silicon-oninsulator (SOI) wafer. (c) Carboxen layer where the Carboxen particles are packed (the well is on the backside), (d) Inlet/outlet layer where the gas is routed in and out.

Figure 3 shows series of acetone desorption peaks after the PC chamber adsorbs 320 ppm of an acetone vapor sampled from a Tedlar bag for an hour. The calculated gains that count all the desorbed peaks and only the first peak are 12,000 and 4,000, respectively. Head pressure is 10 psig and a DC voltage of 85 V is applied for 5 sec every 25 seconds. We find the potential of high capacity and gain for the PC since the first heating solely obtains a gain of 4,000, without including all the desorption peaks. This implies less Carboxen particles will enable PC to be completely desorbed at a single heating, which can potentially deliver the similar gain and faster heating profile.



Figure 3: Periodic desorption peaks with a flame ionization detector (FID), 10 psig Helium head pressure and 85 V applied for 5secs every 25 secs after 60 min of 320 ppm acetone vapor adsorption. The calculated gains that count all the desorbed peaks and only the first peak are 12,000 and 4,000, respectively.

MICROVALVE (MV)

The MV is designed to be integrated with the PC. Fig. 4(a) shows a MV schematic. The MV is composed of two highly doped Si layers that sandwich a polyimide membrane (MEM), which is electrostatically operated between valve closing/top cap (VC/TCAP) and valve opening/bottom cap (VO/BCAP) Si layers for closing and opening actions [6]. Figure 4(b) illustrates assembly of the MV layers. The VC and VO are bonded to TCAP and BCAP, respectively. Au hermetic diffusion bonding is used to simplify the final number of bonding layers to the MEM layer, as well as to minimize the leak rate of the MV. The MEM is bonded to VC/TCAP and VO/BCAP using polymer adhesive bonding. The polyimide

MEM layer has four conductive layers that serve as four membrane valves: Sample (S), Vent (V), Carrier (C), and Injection (I). These independent membrane valves are integrated in the MV chip with two additional bypass (BP) valves – BP1 and BP2 – to implement complete operation of sampling and sweeping the desorbed analytes to the MC. A unique feature of this valve is the composite membrane that has an anti-stiction coating (purple color in Fig. 4(a)), a high dielectric strength, and conductive layers. Other unique features include the center pad to reduce opening voltage and charge buildup; pressure balancing (PB) mechanism to lower differential pressure across the membrane, lowering stress and allowing the valve to open against high pressure; interface treatment to prevent charge build up which is the main failure mode of most electrostatic valves [7].



Figure 4: (a) Composition of MV layers: Two highly doped Si layers that sandwich a polyimide membrane (MEM). (b) The two highly doped layers are Au bonded stacks of valve closing/top cap (VC/TCAP) and valve opening/bottom cap (VO/BCAP) Si layers. MEM layer is electrostatically operated between VC/TCAP and VO/BCAP layers. MEM is bonded to VC/TCAP and VO/BCAP using polymer adhesive bonding.

Figure 5 shows a plumbing and operational diagram of the MV that is composed of (a) sampling step, (b) desorption step, and (c) injection step. In (a), S, V, and BP2 valves are open allowing the PC to adsorb the sample by drawing the sample in using a diaphragm pump (D250SS, TCS micropumps, UK) from a Tedlar bag (or ambient). Non-adsorbed sample in the PC exits at V. Operations of BP1/BP2 are dependent on V/I valves: BP1 is open when V is closed and BP2 valve is open when I is closed, and vice versa. In (b), and prior to PC heating, all the valves are closed except BP1/BP2. During the heating of the PC chamber the adsorbed analyte is released but still confined to the PC. In (c), C/I/BP1 valves

are open allowing the desorbed analyte to be swept into the column/FID by the carrier gas. PC is still heated to attain complete desorption.



Figure 5: MV plumbing and operational diagram (a) Sampling step, (b) Desorption step, (c) Injection step.

Figure 6 shows periodic methane peaks from an FID as a MV is operated along with the steps in Fig. 5. No heating is applied. Sampling/desorption/injection steps last for 5 s/1 s/5 s. DC 138 V is applied until 2.6 min then the power is shut off. Regular and stable injection pulse are generated, which means the MV captures the methane in the chamber and injects to the FID in a uniform and accurate way.



Figure 6: Periodic methane peaks from an FID as an MV is operated along with Fig. 5. Heating is not applied. Sampling/desorption/injection steps last for 5 s/1 s/5 s. DC 138 V is applied until 2.6 min when the power is shut off.

MICROCOLUMN (MC)

Figure 7 shows (a) a microscopic view of the serpentine channel (left) for an MC and its two-layer composition with the serpentine layer and an Au coated capping layer to facilitate diffusion bonding to the serpentine layer. The overall footprint of the serpentine channel is $60 \ \mu m$ (W) x 100 μm (D) x 1 m (L) whose hydraulic diameter is $86 \ \mu m$. The serpentine microchannel generates better separation than that of spiral channel design in the micro level

of the chip design. The waved turn geometry counteracts the



Figure 7: (a) One meter of serpentine channel whose footprint is $60 \mu m$ (W) x 100 μm (D) x 1 m (L) (left); Au coated Si layer caps the serpentine channel layer to form an MC device (right), (b) thickness comparison of 2 m vs. 1 m MC, and (c) 2 m vs. 4 m MC.

formation of Dean Vortices, providing lower flow dispersion. To improve GC capacity and resolution, we develop 2 m and 4 m MCs by stacking two 1 m serpentine MCs, and having 1 m serpentines on both sides of two wafers, thereby eliminating the need to expand the chip platform size. Figure 7 (b) and (c) show thickness comparisons between 2 m and 1 m, 4 m and 2 m, respectively.

Figure 8 illustrates the composition of the 2 m (left) and 4 m (right). Au diffusion bonding is used to hermetically seal all of the layers. Since the MC is the closest PC-GC component to the QITMS, hermetic sealing of the MC is most critical to the overall sealing of PC-GC among all the MEMS devices. Via layers are used to connect serpentine layers with the same diameter hole as the hydraulic diameter of the serpentine channel to avoid dead volume at the junction which leads to dispersion in the chromatogram.

Figure 9 illustrates a 2 m MC chromatogram of 15 SMAC compounds using an Agilent flame ionization detector (FID). The MC is dynamically coated with 10% OV-5 (Ohio Valley, U.S.A.). A mild pressure across the MC (<10 psig) facilitates uniform coating, which improves the peak shape, tailing and dispersion. GC operating conditions are 16 psig head pressure and 0.5 ml/min flow



Figure 8: Configuration schematics to illustrate how 2 m MC can be composed of (left), and how 4 m MC can be composed of (right). Au diffusion bonding is used to hermetically seal all the layers.

rate. The injection volume is 0.02 µL with a 500:1 split ratio.



Figure 9: Two-meter MC chromatogram of 15 SMAC compounds using the Agilent FID; 15 SMAC compounds are (1) Dichloromehtane, (2) Nitromethane, (3) Chloroform, (4) 1,1,1trichloroethane, (5) 1,2-Dichloroethane (6) n-butanol, (7) Benzene, (8) Trichloroethane, (9) 2-ethoxyethanol, (10) Hexane, (11) Toluene, (12) Hexanal, (13) Diacetone alcohol, (14) Ethylbenzene, (15) O-xylene



Figure 10: (a) MV operation under injections of varying duration. Elution profile exhibits five injections of perfluoropropane/fluorobenzene mixture. Injections last for 10, 20, 50, 100 and 200 milliseconds; corresponding mass spectra of perfluoropropane for shortest and longest injection times are shown in (b) and (c).

INTEGRATION TESTING

Figure 10 illustrates the results of a series of injections using the MV and sample mixture consisting of 150 ppm of acetone, 250 ppm of perfluoropropane and 250 ppm of fluorobenzene in helium. The sample is administered from a pressurized gas cylinder. A 1 m MC and a three-meter transfer line are connected between the MV and the QITMS. A PC is not used. Starting from the left side of plot in Fig. 10(a), subsequent injections last for 10, 20, 50, 100 and 200 ms. The first chromatography peak corresponds to perfluoropropane, followed by a fluorobenzene peak. Figure 10(b) and (c) contain representative m/q lines belonging to perfluoropropane and are extracted from the integral spectra by time-gating on the counts corresponding to chromatography peaks and are corrected for the underlying background spectrum. Background segments are sampled 10 s prior to each injection and are indicated by green color areas of elution profile found in Fig. 10(a).

The background mass spectra consists mostly of oxygen and acetone whose abundances increase twofold following each injection indicating a leak in the sample introduction line or MV packaging. Perfluoropropane and flurobenzene elutions peak are sampled in 2 s segments and related mass spectra for shortest and longest injection times are shown in Fig. 10(b) and 10(c), respectively. For 10 ms injection times, the presence of fluorobenzene is barely visible in both the elution profile and the mass spectra (not shown here), which will need the PC to boost the signal intensity. This is in sharp contrast to the perfluoropropane signal, which remains a dominant feature in both the elution profile and the 69 Da mass spectra line.

CONCLUSIONS

We have demonstrated the individual performance of PC, MV, and MC as well as MV/MC integration performance. Based on the current progress, combining all of these PC-GC devices is expected to enable a dramatic reduction in size, power consumption, and carrier gas without loss of capability for the S.A.M. instrument.

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MICRO-SCALE ELECTROCHEMICAL GAS SENSOR FOR SELECTIVE DETECTION OF VOLATILE POLLUTANTS

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ABSTRACT

A micro fabricated chemical gas sensor based on the working principle of an electrochemical cell is presented along with tests in the detection of CO as a model pollutant. The sensor is composed of two Pt electrodes and a *Nafion* membrane acting as proton transporting medium. The fabrication process is presented as well as the test setups used for characterizing the sensor. The early results show that the sensor is capable of performing cyclic voltammetry when subjected to a gas flow and show the presence an oxidation peak of CO into CO₂ at a precise applied potential.

INTRODUCTION

The most promising and developed technology for micro fabricated chemical gas sensor is based on the measurement of a resistance change between two electrodes as an chemical specie adsorbs on the medium in between them. That sensing layer is usually a semiconducting metal oxide such as ZnO [1], CuO [2], TiO₂ [3] and SnO₂ [4]. The sensing layer is heated to a high temperature (about 300 °C) by a heater situated under it which induces an increase in the concentration of reactive oxygen ions at the surface of the sensing layer [5]. Those anions react with the adsorbed pollutant inducing an electron variation in the semiconductor and thus a resistance variation.

Besides being completely compatible with conventional micro fabrication processes, this type of sensor can also benefit from all of



Characterization results			
Process step	Measurements		
Oxidation	920 nm		
Adhesion layer	9.6 µm		
Nafion deposition	300 – 600 nm		
Metallization	110 – 120 nm		

Figure 1 : Topology, layout and characterization results of the micro electrochemical gas sensor.

the various nanofabrication techniques developed for metal oxides to increase substantially their sensitivity [6].

However, in order to achieve any degree of selectivity, it is necessary to combine several of this type of sensors, in an array, with different sensing layers [7].

In order to achieve selectivity on a single sensor and at room temperature, one could use the redox potential which is a characteristic of any chemical specie. To do that, the species can be oxidized or reduced in an electrochemical cell configuration. Upon reaction, an increase in current is generated, which is measured as the redox potential of the specie is applied.

However, it is necessary to have a third electrode, called reference electrode, to be able to identify the species getting oxidized and reduced. This electrode is composed of a redox couple whose redox potential is well known and does not vary in the experimental conditions. It is by measuring the voltage between that reference electrode and the working electrode that it is possible to identify the species engaged in the electrochemical reactions thanks to the Nernst equation.

The development of a gas sensor based on this working principle would require fabrication of a fully solid state electrochemical cell. Among other possibilities, such as ionic conducting hydrogels [8] or zirconia based materials [9], the liquid electrolyte can be replaced by an ionic conductive polymer such as *Nafion*[®].

Nafion is proton conducting membrane largely used in fuel cells [10]. In particular in *Proton Exchange Membrane Fuel Cells*



Figure 2: Electrochemical characterization setup (A) and sensor test setup (B).



Figure 3 : Electrochemical characterization under N_2 (A) and under CO (B).

(PEMFC) which are reducing O_2 and oxidizing H_2 in acidic conditions and at room temperature. In this example, the *Nafion* membrane is playing the role of the electrolyte, transporting the protons from one side to the other of the cell. It is a polymer composed of a teflon-like backbone and a sulfonic head. When hydrated, it forms clusters around water [11], and when acidified, the sulfonic groups allow proton transport [12].

Besides of being used in conventional fuel cell as described earlier, *Nafion* is also a promising material to be used in micro-fuel cells were it is used to separate the cathode and the anode while transporting protons [13]. The design developed in those devices have largely inspired the present work [14].

The sensor

The sensor proposed in this paper is an attempt to prove the feasibility of the electrochemical analysis of organic species dissolved in the gas phase. To obtain a fully solid state sensor, *Nafion* will be used as ionic conductive electrolyte, and the

electrodes will be made of Pt. Pt is chosen because its electrochemical and electrocatalytic properties, and its poisoning by CO in fuel cells is well documented [15], [16].

EXPERIMENTAL SECTION

Fabrication process

First, 100 mm diameter, 525 μ m thick L-test Si wafers were processed with standard cleaning procedure in the following order: i) 90% H₂SO₄/H₂O₂ (piranha), ii) 50:1 HF (clean) iii) 5:1:1 H₂O:H₂O₂:NH₄OH iv) 5:1:1 H₂O:H₂O₂:HCl (diffusion clean). A 1 μ m thick Si oxide layer is then grown on the substrate by wet oxidizing annealing at 1100 °C for 2 h 15 min.

Our studies have showed that the *Nafion* adheres poorly to the SiO₂ substrate, which is why a 10 μ m thick SU-8 2010 layer was first spin coated on the substrate at 1500 rpm for 5 sec and 3000 rpm for 15 sec. The layer is then exposed to a 17 mW.cm⁻², 365 nm *OAI*® UV exposer for 7 sec, and backed at 85 °C for 2 min to ensure hardening and drying of the SU-8. Prior to deposition of the *Nafion* layer, the SU-8 coated substrate is subjected to 30 sec of O₂ plasma etching.

The *Nafion* layer is deposited by spin coating a D1021 *Nafion* \circledast aqueous dispersion in three consecutive identical steps, which have been optimized to obtain a thickness of 300 nm, of 5 sec at 500 rpm followed by 30 sec at 1500 rpm. Each spin coating step is followed by a 2 min back at 65 °C. The deposition process is finished by a 1h backing at 110 °C to ensure evaporation of all solvent for upcoming photolithography steps.

The patterns of the electrodes are transmitted on the wafer by a typical liftoff process. The HMDS treatment generally performed prior to the coating of the resist is skipped to avoid any chemical modification of the *Nafion* layer. A 3 μ m thick *SPR220-3* photoresist is spin coated and backed at 90 °C for 2 min. The wafer is then exposed to a 15 mW.cm⁻², 365 nm UV light, through a transparency mask displaying the geometry of the sensor showed on Figure 1 in a hard contact mode for 3 sec.

The geometry of the sensor is simple and composed of two interdigitated electrodes with two larger pads to allow electrical contact as shown on Figure 1. After exposure, the photoresist is by spin coating of the corresponding chemical developer, rinsing and drying.

As final step, a 100 nm thick Pt layer is deposited by e-beam evaporation at a 1 Å.s⁻¹ rate, and the resist is stripped in an acetone bath for 2 h. The sensors are then rinsed, dried and diced. Prior to characterization or testing, the *Nafion* layer needs to be rehydrated and acidified in order to have optimum proton conductivity. The sensor is immersed in a series of baths at 80 °C for 15 min in each: i) H₂O₂ 2%vol. in H₂O, ii) H₂SO₄ 0.1 mol.L⁻¹ iii) H₂O. After that treatment the sensors are ready to be characterized and tested. The final design of the sensor is presented on Figure 1.

Characterization and test methods

The topological characterization of the sensor was made with a profilometer and the results are presented in the table of Figure 1. All the measured parameters are in the acceptable range compared to the target thicknesses. Concerning the *Nafion* layer, the measurement of its thickness revealed a quite important irregularity spanning from 300 to 600 nm across the wafer.

The sensors are also characterized by electrochemistry in a conventional electrochemical cell in a three electrodes configuration. The test setup is presented on Figure 2A and



Figure 4 : Curves of the cyclic voltammetry performed in the gas phase (A) and in the electrochemical cell in a two electrodes configuration (B).

represents the sensor with its two electrodes being respectively the working and counter electrodes, connected to an external *Mercury/Mercury Sulfate Electrode* (MSE) reference electrode ($E^{\circ}_{MSE} = -0.654$ V vs SHE) through and *Biologic*® SP-300 potentiostat. All three electrodes are immersed in a 0.1 mol.L⁻¹ H₂SO₄ solution and cyclic voltammetry is performed between various potentials and at different sweep rates. In cyclic voltammetry, the potential (in V) is applied and swept between the working and the counter electrodes. The corresponding current (in mA) flowing between the working and counter electrodes is recorded and plotted against the voltage measured between the working and the reference electrode.

The testing of the sensors for the detection of volatile pollutants are performed in a custom made test chamber. The electrodes are connected to the same potentiostat in a two electrodes configuration. The test chamber is under constant gas flow from either N_2 or pure CO with a flow rate of 20 cm³·min⁻¹. While the test chamber is under gas flow, the voltage is swept between the two electrodes of the sensor from -1.0 to 1.0 V with a 200 mV.s⁻¹ sweep rate. The complete test setup is presented on Figure 2B.

RESULTS AND DISCUSSION

Design of the sensor

The sensor has been designed with a few considerations in mind. First, the fabrication steps should impact the least possible the *Nafion* layer to keep its chemical integrity. In terms of redox chemical reaction, the key features are the "triple points", which are the interfaces were Pt, *Nafion* and the gas phase meet. With this in mind, the Pt electrodes do not need to be very thick, and it is anticipated that their thickness will not impact the sensitivity significantly. However, the main constraint for the geometry is the separation between the electrodes fingers because the ionic conductivity of *Nafion* drops drastically above 500 µm.

Keeping the design simple is also necessary to prove that the proposed sensing mechanism might be compatible with mass production.

Electrochemical characterization

The electrochemical characterization of the sensor consists of a set of two experiments performed in the configuration of Figure 2A. The first one is done with N₂ bubbling in the cell, and the second one is with CO bubbling.

The experiment with N₂ is necessary to check if the Pt electrode are well positioned at the surface of the *Nafion* layer and that the *Nafion* layer is indeed able to conduct protons as expected after the fabrication process. The results are shown on Figure 3A were one can see a typical CV curve for polycrystalline Pt in acidic medium [17]. The quality of the Pt surface as well as the ionic conductivity of the *Nafion* are confirmed by the presence of all expected electrochemical signals. The observed signals are H adsorption/desorption located between 0 and 0.25 V vs SHE, the Pt oxide formation starting at 0.7 V vs SHE, the capacitive region plateau and the Pt oxides reduction peak at 0.7 V vs SHE. The experiment is conducted at several sweep rates from 50 mV.s⁻¹ to 200 mV.s⁻¹. All electrochemical signals are increasing in current with the increasing sweep rate, which is expected for cyclic voltammetry.

The second electrochemical characterization is conducted in similar conditions but with CO bubbling. The resulting curve is showed on Figure 3B. On can see that the general shape of the curve is similar to the one obtained under N₂ and that an oxidation peak appears at 0.15 V vs MSE. This peak corresponds to the oxidation of CO into CO₂ [16] according to its position on the potential scale when converted on the SHE scale ($E_{(Pt)}$ (CO₂/CO) \approx 0.8 V vs SHE). The presence of this oxidation peak confirms that the sensor is indeed capable of detecting CO by measuring an increasing current at a specific applied potential corresponding to its redox potential. In addition of the presence of this oxidation peak, one can also see a slight decrease in current of the H ads/des peaks. This phenomena tends to confirm the nature of the oxidation of CO into CO₂ as the adsorption of CO on Pt occurs on the same catalytic sites as H [18].

CO detection tests

As model pollutant CO was chosen, and its oxidation into CO₂ for validation of the electrochemical capabilities of the sensor. The experiment is conducted on the setup showed on Figure 2B. As the

voltage is cycled between -0.5 and 0.5 V with a 200 mV.s⁻¹ sweep rate, the gas flow is alternatively switch from N₂ to CO and to N₂ again. In this configuration, the voltage is swept from one limit to the other in 5 s, which means that the observed changes in signal are happening on that time scale. The first cycles under N₂ allows to obtain a baseline to better identify the signal obtained under CO. The last cycles under N₂ are to show the quick response of the sensor.

The resulting curve shown on Figure 4A shows that under N₂ (black line), no signal is detected at any applied voltage. When the CO flow is turned on, immediately an oxidation peak appears at 0.2 and -0.2 V with increasing current over cycles. The charge of both peaks being very close (0.3 μ C), it seems that they are the signal of the same redox process happening alternatively on both electrodes as the polarization changes. As the gas flow is switch from CO to N₂ again, the oxidation signal disappears immediately, showing that the sensors response is relatively quick (ca. 5 s).

In the absence of reference electrode, it is impossible to identify the nature of that redox reaction. However, in order to confirm that it is indeed CO oxidation into CO_2 , a similar experiment is performed in liquid environment.

The sensor is placed back in the electrochemical cell with the same electrolyte, but this time, the reference electrode is not connected. Again, N₂ and CO are bubbled alternatively in the cell, and the resulting curve is shown on Figure 4B. In this two electrodes configuration, the shape of the curve observed previously is lost and becomes symmetrical with respect to 0 as is the curve obtained in the gas phase. The only shape which is observed under N₂ is the capacitive region of Pt. However, as CO is bubble in the cell, an oxidation peak appears at 0.2 and -0.2 V, just before the capacitive region, exactly as it was observed on Figure 3B. This peak can be attributed to CO oxidation, and its position in this configuration matches well with the position of the peak observed in the gas phase experiment. Obtaining this curve confirms that the result from Figure 4A is indeed CO getting oxidized in CO₂ and thus that the sensor is capable of performing electrochemical measurements in gaseous conditions, and can identify the specie being adsorbed unlike conventional metal oxide based sensors measuring a change in resistance indicating that a specie is present, but cannot be identified [19].

CONCLUSION

In summary, the design and fabrication process of a micro electrochemical gas sensor were presented along with both topological and electrochemical characterizations. It has been showed that the sensor behaves just as a typical working/counter electrodes couple when connected to an external reference electrode in a conventional electrochemical cell.

Testing of the sensor for the detection of CO as model pollutant has proved that the sensor is capable of electrochemical measurements in the gas phase.

To further improve the current sensor, the research effort is now focused on the implementation of a reference electrode in order to achieve identification of species directly in working conditions.

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MICROFABRICATED THERMAL PRE-CONCENTRATOR WITH INTEGRATED CANTILEVER-BASED RESONANT SENSORS

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ABSTRACT

This paper introduces the design, fabrication and initial testing of a smart MEMS-based micro thermal pre-concentration (μ TPC) system with integrated mass-sensitive chemical sensors for volatile organic compound (VOC) sensing. The presented suspendedmembrane μ TPC design features a hybrid of a high surface-area-tovolume ratio and a low thermal mass, resulting in a potentially high pre-concentration factor and a short thermal time constant. Additionally, the system co-integrates upstream and downstream resonant cantilever sensors, operated in an in-plane resonant mode, to monitor the VOC uptake into the μ TPC in real time, but also offering the possibility of a fully integrated chemical sensing system with a dead volume of <10 μ L.

INTRODUCTION

Thermal pre-concentration is an effective approach for VOC detection at sub-ppm levels [1]. VOCs can be used to identify the state of many types of systems, e.g., the environment [2], personal health through breath analysis [3], [4], and plant health [5]. In each of these applications, accurate monitoring of certain VOCs is crucial to provide input for any corrective action. However, the concentrations for many analytes of interest in these systems are low ppm or ppb, with some trace VOCs existing in ppt concentrations [6], presenting a challenge for many types of chemical sensors with higher limits of detection (LOD). Pre-concentration can temporarily increase the concentration for many compounds allowing for low-concentration analyte identification by a variety of downstream sensing mechanisms, e.g., mass spectroscopy, gas chromatography, and MEMS based sensors [7].

Existing microfabricated pre-concentrators typically fall into two classes: (1) pre-concentrators that feature three-dimensional (3D) structures directly etched into the bulk (silicon) substrate [8], which often yields large sorbent volumes at the expense of long thermal time constants and large power consumption or (2) quasi-2D pre-concentrators based on thermally insulated suspended membranes, which allow for fast thermal rise times and low power consumption [9], but limit the sorbent volume. The proposed μ TPC is a hybrid of these designs, incorporating 3D high-aspect-ratio microstructures with a suspended membrane design, and coupling this sensor design with an effective packaging scheme that minimizes chamber dead volume.

Moreover, most microfabricated pre-concentration systems currently lack the ability to, in real-time, detect analyte uptake within the sorption film and, therefore, adjust the collection time based on the loading condition. An exception is the integration of a pre-concentrator on top of a pivot-plate-type mass-sensitive resonator [9], which can monitor the analyte uptake in the sorption film in real time. However, the integration of a pre-concentrator and sensor into one suspended microstructure limits the sorbent volume and the performance of the mass-sensitive sensor. Thus, in this work, micromachined resonant sensors were fabricated on the same chip next to the pre-concentrator to monitor analyte uptake upstream and downstream of the TPC. As the concentration of analyte within the μ TPC film starts to saturate, the detected concentrations at the upstream and downstream sensors will converge due to less uptake by the TPC. While the detection of many analytes of interest (possibly ppt or low ppb in concentration) still remains a challenge with these sensors, other analytes remain at concentration levels, ppm or high ppb, that can be detected without pre-concentration [10], [11]. The signals from the optimized resonators can thus serve as a benchmark for the analyte uptake within the actual preconcentrator and can be used to monitor and optimize the preconcentration process in real time.

The achievable pre-concentration factor (CF) for an equilibrium-based pre-concentrator depends on the sorbent volume, the partition coefficient K of a particular sorbent-analyte combination, and the dead volume of the pre-concentration unit:

$$CF = \frac{K * Absorption Volume}{Dead Volume}.$$
 (1)

To increase the CF, this work also presents ways to minimize the dead volume using wafer-bonding techniques.

DESIGN & FABRICATION

Figure 1 shows the system design, comprising of multiple resonant cantilevers and three μ TPC membranes on a single chip. The μ TPCs consist of a suspended membrane with integrated heating and temperature sensing resistors, defined in the device layer of a silicon on insulator (SOI) wafer and formed through boron diffusion with an oxide mask. The suspended membranes feature high-aspect-ratio pillars or ridges, etched in the handle layer of the SOI substrate, to increase the surface area by ~10-100x.



Figure 1: CAD cutaway view of smart μ TPC fabricated on an SOI wafer through traditional MEMS bulk micromachining techniques with integrated upstream and downstream mass-sensitive chemical sensors.

The fabrication process of the μ TPC system follows a recent process used for resonant chemical sensors in our group [12], [13] and, thus, enables a smart, single-chip pre-concentration module with integrated chemical sensors. The high aspect-ratio deepreactive-ion-etch (DRIE) from the backside of the wafer, used to

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Figure 2: Simulated temperature distribution across heated $2x1 \text{ mm} \mu TPC$ with ridges using COMSOL. The applied heating power of 500 mW causes a fairly uniform 230 °C temperature elevation in the membrane and ridges.

release the resonant sensors in the previous work, now also serves to define the pillars and ridges used for pre-concentration [12]. Additionally, the device release etch, carried out from the top side of the wafer, now patterns the suspended membrane in addition to the sensing cantilevers. As such, no additional masks or fabrication steps are required to fabricate the pre-concentrators next to the resonant cantilevers.

The sensors are cantilever-based resonators with a semicircular head region, which are coated with an appropriate sensing film after fabrication. Different implemented sensors designs have fundamental in-plane resonant frequencies of 300-800 kHz. Two



Figure 3: SEM micrographs of a fabricated μ TPC and resonant cantilever sensor die prior to polymer deposition. Three individually addressable and thermally isolated 1x2 mm μ TPCs, as well as the mass-sensitive sensors, are visible from both front side (top) and backside (bottom) of the chip.

sensors are placed at each inlet and outlet port to measure the analyte concentration in real time. The cantilevers are excited electrothermally, similar to our previous work, by a diffused resistor at a corner of the anchor. Four piezoresistors arranged in a U-shaped Wheatstone bridge preferentially detect the in-plane vibration mode and provide for continuous operation within an amplifying feedback loop [12], [14]–[16]. To compensate for temperature effects, one resonator is left uncoated to serve as a reference.

Figure 2 shows a thermal simulation of a pre-concentrator on top of a 1 mm by 2 mm suspended membrane with 20 μ m silicon thickness using COMSOL. The membrane, with an integrated 3-D ridge structure, is uniformly heated to approx. 230 °C using a heating power of 500 mW. Besides heat transport by conduction through the silicon, a constant heat transfer coefficient of 50 W/m²K was assumed to approximate the heat transfer through the surrounding air. The small thermal mass associated with the suspended membrane structure assures a short thermal time constant, simulated to be approx. 0.4 s for this device.

Figure 3 shows SEM images of a fabricated die with μ TPCs and mass-sensitive sensors. Each of the three suspended membranes is connected to the substrate by four legs connected to two anchor points. Various die configurations incorporate either two or three individually addressable μ TPCs with varying device geometries (with up to 2x2 mm membranes featuring a total surface area of 53 mm²) and incorporating either ridge or pillar like structures. The long supporting legs (1500-2250 μ m in length) provide thermal isolation, thus reducing the required heating power.

FUNCTIONALIZATION & PACKAGING

Maximizing the CF in Eqn. 1 necessitates a high absorptionvolume to dead-volume ratio. Thus, special consideration must go into the post-fabrication functionalization and packaging steps. As already mentioned, the pillars/ridges significantly increase the surface area of the device; this allows for approximately 0.1 µL of absorption volume per 2 µm of polymer thickness for a 53 mm² device. Thin sorbent films allow for quick desorption from the film, causing a sharp increase in concentration under continuous flow testing. Thicker films, on the other hand, allow for larger CF. Figure 4 shows a typical coated device, which will have 5-20 µm sorbent film deposited through a shadow-masked spray coating. The microresonator is functionalized with either the same or a different sorbent film. For the sample devices in this paper, the uTPC pillars were coated with dimethyl silicone gum (silicone OV-1) and the microresonators were spray-coated with polyisobutylene (PIB)



Figure 4: SEM micrograph of polymer coated pillar structures. Shadow-masked pillars (bottom-left and top-right) are left uncoated, while pillars near the center are coated with a thick absorbing layer.



Figure 5: Fully packaged and wire-bonded device with ferrules aligned to laser ablated holes and epoxied to the borosilicate glass package for inlet and outlet ports.

The μ TPC is packaged by bonding borosilicate glass wafers, patterned with a layer of Microchem SU-8 3005 for thermocompression bonding, to the μ TPC's top and bottom. This achieves a low dead volume, approx. 7μ L, which is required for a high CF. The borosilicate glass also thermally insulates the package, further minimizing the power required for sufficient temperature elevation (100-200 °C). In addition to bonding, the SU-8 allows for appropriate spacing between the microresonator and the glass surface, which would otherwise prevent the resonator from oscillating. An additional epoxy layer is added outside the package in select areas to provide strength for high pressure testing.

After bonding, laser machining is used to form the inlet and outlet ports and ferrules are mounted with epoxy, providing for



Figure 6: (a) Measured average temperature elevation of $1x2mm \mu TPC$ as a function of applied heating power. Dashed lines represent linear fits through the data sets up to ~200 °C. (b) Measured and simulated thermal time constants for varying device geometries. Dashed lines represent the simulated data.

tubing access. Figure 5 shows a packaged device. The μ TPC can be operated in stop flow or continuous flow. While operating in stop flow, the μ TPC temporarily increases the concentration in the dead volume for downstream analysis by rapidly ejecting, through thermal desorption, analytes that had accumulated into the sorbent material over the sampling window. As the flow rate increases, the desorption window broadens, causing the maximum preconcentration factor to decrease.

A sample device in this work was coated with approx. $0.31 \,\mu\text{L}$ of OV-1, resulting in an active volume to dead volume ratio of ~1:23. Thus for analytes with a partition coefficient greater than 23, pre-concentration will occur. With partition coefficients greater than 1000 for various aromatic compounds [17], we expect pre-concentration factors of 40 or higher based on Eqn. 1.

SYSTEM CHARACTERIZATION

To test the μ TPC, the two integrated 500 Ω diffused resistors (connected either in series or parallel) are used for joule heating, while an additional meandering diffused resistor can be used for temperature sensing. The resistive temperature sensors of the μ TPC system are initially characterized in an environmental chamber up to 150 °C. The measured data was fit to a second order polynomial to extrapolate temperatures beyond 150 °C. Using this calibration data, the µTPC heating using the embedded heating resistors is characterized with a source-meter unit. Figure 6a shows the average temperature of a fully-packaged and sorbent-coated preconcentrator module with 2x1 mm membranes as a function of the heating power. As expected, the temperature elevation is linear with the applied heating power and approx. 700 mW are needed to heat to ~200 °C, or 4.4 mW/°C of temperature elevation, needed for VOC desorption. This is a larger power requirement than was simulated and it is thought to arise from the effect of the package on the thermal characteristics of the device. Figure 6b compares the simulated and measured thermal transients, indicating short thermal time constants of less than 1 sec even for the largest preconcentrators. The continued increase in temperature after the initial rapid increase reflects the package and surrounding medium heating.

The resonant cantilevers were also characterized before and after functionalization/packaging. Figure 7 shows the successful integration of a cantilever sensor within the sealed package. The added polymer mass causes the shift in resonance frequency and slight drop in Q-factor.



Figure 7: Amplitude transfer characteristics of a resonant cantilever before and after functionalization and packaging with PIB. The added polymer mass contributes to a -4.2 kHz shift in resonant frequency while maintaining a high Q-factor, >1500.

CONCLUSION

This paper demonstrates the design, fabrication, packaging and initial characterization of a smart μ TPC with integrated microresonators to sense VOC uptake in real-time. By utilizing pillar or ridge structures, a high surface-area is created allowing for large absorption film volumes compared to planar designs, while

still allowing for quick thermal rise times. The borosilicate glass packaging maintains an extremely low dead volume, which allows for higher pre-concentration factors.

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A LOW-VOLTAGE MICROFLUIDIC VALVE BASED UPON A REVERSIBLE HYDROPHOBICITY EFFECT

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ABSTRACT

We report a microfluidic valve based on a reversible hydrophobicity effect via the growth and retraction of non-volatile nano-textured dendritic silver filaments on the surface of a solid electrolyte. A 6 V DC bias grows or dissolves, depending on the polarity, filaments which are tens to hundreds of nanometers in height. The valving function of these nano-filaments, which occurs within approximately 25 sec, was demonstrated by monitoring the fluid flow in a 250 μ m wide, 25 μ m deep PDMS-enclosed channel.

INTRODUCTION

Valves in microfluidics control routing, timing and separation of fluids [1]. Most currently employed valves are made of PDMS and their operation relies on the deformation of thin film or bulk PDMS to obstruct or permit fluid flow [2-6]. However, such techniques, in general, require large mechanical structures, complicated integration into microfluidic channels and cumbersome external pneumatic accessories [7-12].

We report a unique mechanism to control the fluid flow - the growth or retraction of nano-scale dendritic metallic filaments (Figure 1a&b). The nano-filaments manipulate hydrophobicity and contact angle hysteresis on the interface between the fluid and the channel surface [13], rather than the bulk of the flow, to provide flow regulation. Microfluidic channels are typically 10s or 100s of µm in depth, while our valve operates on a scale of 10s to 100s of nm (Figure 1c&d). This nano-structure changes the critical dynamic characteristics on the bottom surface of the channel, emulating the transition from the lotus effect to the petal effect. The petal effect was found to be based upon hierarchical micro- and nano-structures which significantly contribute to the adhesive force on a fluid droplet [14,15]. The nano-scale dendritic filaments enhance the roughness to interrupt flow [16,17] and modulate the interface topography on the bottom of a PDMS-enclosed microfluidic channel (Figure 1e).



Figure 1: Electrodeposition of metallic nano-filaments on the surface of a solid electrolyte. **a.** Schematic of the growth of dendritic silver nano-filaments on a chalcogenide solid electrolyte surface. **b.** The growth and retraction processes of nano-filaments depending on the polarity of 6 V DC bias. **c.** Filament morphology taken by scanning electron microscope (SEM), showing maximum of 200 nm height of nano-filaments. **d.** Optical profilometer image illustrating filament morphology. **e.** The cross-sectional view of the valve.

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The bulk flow is pinned by the adhesive force from the channel bottom where the liquid-solid interface forms, which clearly differentiates our approach from existing work that incorporates the liquid-air interface of the droplet exposed to the air. In the case of microfluidic flow, the attractive force between liquid molecules and the cohesive force between solid surface and liquid molecules dominate the flow characteristics [18]. When the fluid molecules adjacent to the channel bottom are stopped, the molecules inside the bulk microfluidic also stop accordingly due to the attractive intermolecular force [19]. From the macroscopic viewpoint, the dynamic viscosity of microfluidic flow is extremely high, which impedes sliding between adjacent laminar flow layers occurring at significantly low Reynolds number [20,21]. This is a unique property of low Reynolds number microfluidic flow which does not occur in macroscale fluidic settings as the viscous force fails to hinder the movement of the bulk in these.

Instead of the cumbersome pneumatic elements and air tubing, the nano-scale valve is actuated by a low DC bias ($\leq 6V$), allowing the external unit to be a simplified standard DC power supply. Unlike the pneumatically actuated devices, where the air may penetrate through the thin PDMS membrane and dissolve into the biological / chemical samples in the channel, the nano-scale valve is free from such contamination.

RESULTS

A constant DC supply, rather than a pulse modulated voltage, was found to be preferential to facilitate rapid and directional nano-filament growth, (Figure 2a). In both 50% and 80% duty cycle pulse-width-modulated (PWM) supplies, multiple nucleation sites were observed during the growth process. These sites initiated the nano-filaments independently and formed rather randomly dispersed growth of nano-filaments from the cathode tip. Figure 2b shows the exponential dependence of the growth rate on applied DC voltage ($\leq 6V$), matching well with simulated values. The simulation is based on an electrodeposition model which with electrochemical couples theory а random walk/diffusion component to multiple incidences of dendrite growth for each condition [22]. At 6V DC, the growth rate became 18-20 µm/sec, thus the nano-filaments grow from the cathode and reach the anode within 12-14 seconds for a 250 µm wide channel.



Figure 2: Control of nano-filaments growth. *a.* Filaments generated by DC bias and pulse width modulation (PWM) with different duty cycles. *b.* Filament growth rate as a function of applied DC voltage.

The petal effect requires two attributes: i) hydrophobic surface with contact angle (CA) $> 90^{\circ}$, and ii) high contact angle hysteresis (CAH) responsible for high adhesive force (Table 1). Three hydrophobic materials, including rough Ag, smooth Ag, and parylene, and two hydrophilic materials, including chalcogenide glass (Ge-Se) and SiO₂, were evaluated for CA and CAH. Note that the measured CA of rough and smooth silver showed little difference, yet the CAH measurements gave significantly different results: 74° for rough Ag vs. 54° for smooth Ag. Figure 3a illustrates the roughness, R_{f_i} incurred by the nano-filaments, which promote high CA and CAH of hydrophobic (solid lines) surfaces, yet lower the CA and moderately improve the CAH of hydrophilic (dotted lines) surface. The silver nano-filaments were covered by a thin parylene film, 200 nm thick, to prevent direct contact to the bio-sample in the channel to avoid silver contamination (Figure 3b), and their nano-scale rough topography was projected through the parylene layer to generate significant roughness features above the parylene surface (Figure 3c). If either CA or CAH requirement is unmet, the valve will not work. For instance, a valve with a SiO₂ patch showed the roughness of protruding filaments but failed to stop the flow because SiO₂ is hydrophilic.

Table 1. Measured contact angle (CA) and contact angle hysteresis (CAH) of different materials using a Rame-Hart Goniometer.

Materials	CA [Deg] $\pm s.d.$	Advancing angle [Deg] ±s.d.	Receding angle [Deg] ±s.d.	CAH [Deg] ±s.d.	$cos\theta_{rec}$ - $cos\theta_{adv}$
Rough silver	94±1	98±2	24±2	74±3	1.05
Smooth silver	93±1	114±2	60±2	54±3	0.91
Parylene	87±1	97±2	64±2	33±3	0.56
Chalcogenide	69±1	46±1	22±1	24±1	0.23
Silicon dioxide	44±1	39±1	25±1	14±1	0.13


Figure 3: Characterization of the nano-filaments. **a.** Contact angle (CA) and contact angle hysteresis (CAH) as a function of roughness, R_5 , of a hydrophobic silver surface (solid line) and a hydrophilic SiO₂ surface (dashed line). **b.** The visualization of nano-filaments under a 200 nm thick parylene, showing the nano-filaments grow underneath the parylene film (contrast). **c.** The topography profiles of the surfaces of parylene, silicon dioxide and chalcogenide solid electrolyte, from the top down, using a stylus profilometer.

Figure 4a shows the setup to evaluate the effectiveness of the nano-valve in a 25 µm deep PDMS-enclosed microfluidic channel. The fluid flow could be stopped and restarted by nano-filaments within ~25 seconds of actuation (Figure 4b). The nano-filaments are persistent and therefore no static power is required to retain the on/off states of the valve. We attempted valving in channels up to 30 um deep and up to 250 µm wide, which were all successfully regulated by the nano-valve. However, the nano-filaments failed to regulate the flow in 50 µm deep channel. This is likely due to the lack of a sufficiently strong hydrophobicity control of such a large fluid bulk, especially in the liquid located far from the nano-filament surface. The nano-valve was evaluated by flow rate measurements and visualized by suspended microspheres in the fluid (Figure 4c). The number of microspheres in individual regions was counted in different phases (Figure 4d), corresponding to the phases shown in Figure 4b. Region 1 had fewer and slower microspheres than region 4 when the valve was in the process of opening and closing. The trend was also apparent in the period of reopening the channel. This trend matched the presence of partial nano-filaments. The region where the nano-filaments existed had fewer and slower microspheres than the region where the nano-filaments retracted or did not yet reach. The behaviors of the microspheres reflected the flow rate of the liquid flow, thus this observation was consistent with the filament growthinduced hydrodynamic changes.



Figure 4: Testing of valves in standard PDMS-based microfluidic channels. a. Experimental setup for the nano-valve in a microfluidic channel. b. On/off valve operation: bias (red) and the corresponding measured flow rate (blue). c. The visualization of the microfluidic flow via monitoring of 5 μ m diameter microspheres inside the liquid. d. Counts of microspheres in the individual regions at different windows of time corresponding to the phases shown in b.

The nano-valve has much room to improve. The current growth / retraction rates are relatively low so that actuation time constants are in the order of 10s of seconds for a 250 μ m wide channel. The growth / retraction rates can be improved by optimizing the chalcogenide materials and by moving to higher bias. The electrodes shape was tapered to focus the electric field at the tip, however, multiple growth / retraction sites often became activated at the tip and the tapered sides of the electrode. One may passivate the tapered sides of the electrode by covering the electrode, except the tip, using an inert material, to allow electric field focusing at the tip to grow nano-filaments only at the tip.

The nano-valve benefits from a unique operating mechanism, deploying nano-scale rough solid to liquid interfaces, requires no pneumatic accessories to operate, and thereby may offer attractive potential in micro-/nanofluidics. Unlike existing pneumatic valves, the nano-valve demands no static power consumption to retain the on/off states of the valve. This feature becomes critical when a large array of valves needs to be integrated in a powercritical setting, such as microfluidic portable analyzers. The nano-valve is scalable. When the feature sizes are scaled down to form a nano-fluidic system, the nano-valve The growth / retraction rates are performs better. proportional to the electric field, thus smaller dimension increases the growth / retraction rates at a given actuation voltage.

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A SCALABLE ELECTRONIC SENSOR FOR MULTIPLEXED DETECTION OF CELLS IN DIFFERENT MICROFLUIDIC CHANNELS

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ABSTRACT

Microfluidic devices are extremely attractive for performing a wide variety of biomedical assays, some of which rely on accurate determination of spatial information from manipulated particles/biological cells. To obtain this spatial information, microfluidic devices are often tied to microscopic analysis leading to complex instrumentation with higher cost and reduced portability. To address these shortcomings, we introduce a sensor technology, microfluidic CODES, that combines resistive pulse sensing with code division multiple access (CDMA) to detect the particles/cells in multiple channels from a single electrical waveform. We designed and fabricated a proof-of-principle microfluidic device and characterized it by processing a simulated biological sample of human cancer cells. By post-processing, we decoded the electrical signal and successfully identified cells in different microfluidic channels, even if cell signals overlap. Microfluidic CODES has the potential to enable low-cost integrated lab-on-a-chip devices that are ideally suited for point-of-care testing of clinical samples.

INTRODUCTION

Analysis of micro-sized particles such as cells, bacteria or viruses using lab-on-a-chip devices continues to be a major area of research that is poised to revolutionize point-of-care testing of biological samples. Numerous biological assays can be performed by spatially tracking particles on lab-on-a-chip devices, where cells/functionalized particles are sorted or captured based on their biophysical [1] or biochemical [2] properties. In most devices, such spatial information is only available through microscopic analysis, limiting the practical utility of the microfluidic chip by tying it to a lab infrastructure, and also increasing the complexity of a system-level implementation. Therefore, an on-chip sensor that can spatially track manipulated particles on a microfluidic chip can potentially enable low-cost, integrated lab-on-a-chip devices that are particularly attractive for testing of biological samples in resource-limited settings.

Among various sensing modalities, resistive pulse sensing (RPS) [3] offers a simple and robust method for label-free detection of small particles and forms the basis of commercially available Coulter counters, widely used for micro-particle sizing and counting. Specifically, RPS relies on modulation of the impedance between two electrodes as a particle, suspended in an electrolyte, passes through an aperture that is placed between two electrodes. By incorporating miniaturized electrodes in microfluidic channels, microscale resistive pulse sensors were successfully used to detect particles ranging from blood cells to viruses [4-5]. With the goal of increasing the device throughput, devices with multiple microfluidic channels equipped with dedicated resistive pulse sensors have previously been demonstrated [6-8]. However, these devices are more complex as they require interfacing with a large number of electrodes and have limited scalability.

In this paper, we introduce a simple and scalable technology, named Microfluidic Coded Orthogonal Detection by Electrical Sensing (microfluidic CODES), to create a multiplexed network of resistive pulse sensors that can be used to spatially track particles on microfluidic devices. We achieve multiplexing by designing micromachined sensors such that each generates distinct electrical waveforms when a particle is detected. We specifically design orthogonal signal waveforms similar to the digital codes used in code division multiple access (CDMA) telecommunication networks, so that individual sensor signals can be uniquely recovered from a single output waveform, even if signals from different sensors interfere. Therefore, our approach allows us to achieve multiplexing of resistive pulse sensors without increasing the number of external electrode connections to the microfluidic device, keeping both device- and system-level complexity to a minimum. We created a microfluidic device with four channels as a proof of the concept. Using human ovarian cancer cells in phosphate-buffered saline (PBS) as a model biological system, we demonstrated that individual cells in different microfluidic channels could be uniquely identified.

DIGITAL CODE DESIGN

To design an orthogonal digital code set, we used the mathematical principles of CDMA telecommunication networks [9]. CDMA is a spread spectrum communication technique, where multiple users simultaneously communicate with a base station through a shared communication channel. Multiplexing of information in CDMA is accomplished by modulating individual user signals with assigned distinct digital codes, called digital spreading codes. By using a decoder that is matched to a specific user's digital spreading code, the information from any user can be retrieved with minimal effect from interference from other users. In this way, CDMA allows multiplexed transmission of information simultaneously and within the same frequency band.

A set of distinguishable codes is a critical requirement for a successful implementation of a CDMA system. Ideally, the digital code set should be perfectly orthogonal, i.e., cross-correlation between different digital codes is zero. While it is possible to design a perfectly orthogonal code set for synchronously transmitted signals, only quasi-orthogonal signal sets with bounded cross-correlation properties can be designed for asynchronous CDMA systems. Randomly arriving cells in microfluidic channels constitutes an asynchronous system and therefore a code-set that maintains its orthogonality under random phase shifts is required. To address these requirements, we used Gold sequences [10] that have both desirable autocorrelation and cross-correlation properties and therefore are commonly employed in asynchronous CDMA uplink to minimize multi-user interference.

To encode four microfluidic channels, we designed 7-bit long Gold sequences. In this process, we first generated a pair of 7-bit long preferred maximal length pseudorandom noise sequences (*m*-sequences). To achieve this, we created two linear feedback shift-registers (LFSRs) based on two primitive polynomials: x^3+x^2+1 and x^3+x+1 . We initialized the two LFSRs at 001 and 111 states, respectively and obtained a preferred pair of *m*-sequences: m_1 =1001011 and m_2 =1110100. We then added cyclic shifts of m_1 and m_2 using bitwise XOR operation to obtain a set of nine 7-bit long Gold sequences. We also tested the validity of the generated sequences by analyzing their autocorrelation and cross-correlation properties. Finally, we chose four 7-bit Gold sequences (g_1 =1010110, g_2 =0111111, g_3 =0100010, g_4 =0011000) as digital codes to encode four microfluidic channels.

MICROFLUIDIC CHIP DESIGN & FABRICATION

To demonstrate microfluidic CODES technology, we designed and fabricated a four-channel microfluidic device (Figure 1(a)). RPS signal waveforms can be created by micromachining the electrodes [11-12] or the microfluidic channels [13]. In our device, we micropatterned three coplanar electrodes to form a network of electrode finger at the bottom of each microfluidic channel. Each electrode finger is 10 μ m wide and is separated from the neighboring electrode finger by a 10 μ m gap. We designed the electrode finger pitch to be on the order of a typical cell size and this leads to localized modulation of impedance between electrode finger pairs as cells flow through microfluidic channels. This way, we use the sequential interaction of the flowing particles with the electrode finger array to generate distinct orthogonal digital codes in different microfluidic channels.



Figure 1. Image of the microfluidic CODES device (a) Full image of the four channel device composed coding surface electrodes on a glass substrate and PDMS microfluidic channels (b) A close-up image of coding electrodes producing 7-bit Gold sequences: "1010110", "0111111", "0100010", "0011000".

An important advantage of our electrode network design is its ability to produce bipolar RPS signals. Bipolar signals are less prone to interference than unipolar signals as they lead to lower cross-correlation between different codes. To generate bipolar signals, our electrode design includes a third electrode as a reference since the impedance modulation between a pair of electrodes is inherently unipolar. Using differential impedance modulation in this configuration allows us to directly generate digital codes in a bipolar form similar to the codes generated using Binary Phase Shift Keying (BPSK) modulation in wireless CDMA communication networks.

We developed a systematic approach to micropattern three electrodes to create a sensor network that can generate any digital code in an arbitrary number of microfluidic channels (Figure 1(a)). First, positive and negative electrodes are placed at the opposite sides of each microfluidic channel. Then we extend these electrodes to form electrode fingers crossing microfluidic channels. The order of positive and negative fingers along each microfluidic channel is determined based on the digital code uniquely assigned for each channel. Second, we route the reference electrode between each pair of positive and negative electrodes fingers. In this configuration, the center-to-center distance between two consecutive reference electrode fingers constitutes a single bit of the digital code. The type of the electrode finger (positive or negative) between two reference electrode fingers determines the polarity (1 or 0) of each bit. Finally, we place the positive and negative electrode traces far from the outer reference electrodes in order to minimize the conduction outside of the coding region.

Another important aspect of our electrode design is that it forms a balanced impedance network to maximize device sensitivity. To achieve this, we first design a code set that contains an equal total number of "1"s and "0"s. This enables us to use an equal number of positive and negative electrode fingers in our layout. When configured as such, the static impedances between the reference and positive/negative electrodes are balanced. This improves the sensitivity of the device since the dynamic impedance changes due to particles are not dominated by the suppressed background signal due to static impedance difference. In addition, the balanced sensor signal is more robust, i.e., less affected by the fluid or material properties.

To fabricate our device, we used a combination of surface micromachining and soft lithography. The device consists of a glass substrate with surface electrodes and a polydimethylsiloxane (PDMS) microfluidic layer. The surface electrodes were fabricated using a lift-off process. Briefly, a 1.5 µm-thick negative photoresist film on a 4-inch glass wafer was patterned using photolithography. A 100 nm-thick Cr/Au film stack was deposited using e-beam evaporation and underlying photoresist was etched in acetone under sonication. The microfluidic channel laver was fabricated using soft lithography. A 15 µm-thick SU-8 photoresist was spun on a silicon wafer and then patterned using photolithography to create the mold. The mold was subsequently coated with a 10:1 mixture of PDMS prepolymer and cross-linker. Degassed PDMS polymer was cured at 65 °C for 4 hours and cured PDMS is peeled off from the mold. PDMS layer and diced glass chips were surface activated under oxygen plasma and were bonded to form the final device (Figure 1).

EXPERIMENTAL METHODS

To characterize our device, we used cells suspended in PBS as a model biological sample. We cultured HeyA8 ovarian cancer cell line (obtained from Dr. John F. McDonald, Georgia Institute of Technology) in RPMI 1640 media (Cellgro, Herndon, VA) supplemented with 10% fetal bovine serum (FBS; Seradigm, Radnor, PA) and 1% penicillin (aMResco, Solon, OH) under a 5% CO₂ atmosphere at 37 °C until a 80% confluence is reached. Following a short trypsinization step, cells were pelleted and then suspended in PBS solution.

In our experiment, suspended cells were driven through the microfluidic device at a constant flow rate using a syringe pump.

The electrical signal from the device was acquired using a data acquisition system. The cell transit was also recorded using a microscope equipped with a high-speed camera to verify the validity of the sensor signals. The complete experimental system is shown in Figure 2.



Figure 2. The Experimental setup used for our measurements: cell suspension is driven through the microfluidic chip using a syringe pump. A bipolar electrical signal is generated using a differential amplifier and is sampled into a computer for decoding. High-speed optical microscopy is used to validate decoded electrical signals

We measured changes in the current flow between the surface electrodes as the cells pass through the microfluidic channels. Specifically, we applied a 400 kHz AC signal to the reference electrode and measured the current flow from negative and positive electrodes separately using two transimpedance amplifiers. We then obtained the differential impedance signal by subtracting the positive electrode signal from the negative electrode signal using a differential amplifier. The amplitude of the resulting bipolar signal was measured using a lock-in amplifier, and the lock-in amplifier output was sampled into the computer at 1 MHz through a data acquisition board for post-processing. During these measurements, the microfluidic chip was placed on an inverted optical microscope (Nikon Eclipse Ti-E) and imaged using a high-speed camera (Vision Research Phantom v7.3). A video of cells flowing through the microfluidic device was recorded at a rate of 8000 frames per second. Recorded videos were downloaded into a computer and compared with the simultaneously recorded electrical signals from our sensor. Frame by frame analysis of the microscopy images enabled us to test the accuracy of our results.

RESULTS AND DISSCUSSION

Distinct digital codes from each microfluidic channel can clearly be identified in the recorded sensor signal. The sensor signals match well with the designed Gold sequences (Figure 3(a)). Deviations are due to several factors such as non-uniform current flow between coplanar electrodes, spherical cell geometry and the coupling between electrodes in the network.

We processed the signal using MATLAB to analyze and decode individual digital spreading codes as follows: for each cell, we first measure the time between signal peaks to determine the bit duration, which we use to generate a template library of four square pulse sequences (each corresponding to a microfluidic channel). Correlating the signal with these four square pulse sequences, we obtain a dominant autocorrelation peak uniquely identifying the microfluidic channel the cell passed through (Figure 3(b)).



Figure 3. Decoding the electrical signal. (a) Normalized sensor signals corresponding to each microfluidic channel are shown with corresponding ideal square pulse sequences. (b) Each signal is correlated with the set of four square pulse code sequences and is decoded through an autocorrelation peak.

It should also be noted that unlike a CDMA communication network in which digital pulses are generated electronically, temporal properties of our digital spreading codes are determined by the microfluidic flow speed. In order to minimize variations in flow speed between different microfluidic channels, we designed all four microfluidic channels to have equal hydraulic resistances. We also choose the microfluidic channel cross section to be close to the cell size so that cell speed is less affected by the non-uniform flow profile across the channel.

An important feature of our technology is that it can resolve cells in microfluidic channels even when they overlap in time. This is quite important for processing samples with high density of particles, where overlapping cases are more frequently to occur. When multiple cells occupy the coding region simultaneously, the interference between signals makes it hard to identify individual cells (Figure 4(a)). Here, we demonstrate a case where we resolve two overlapping cells (Figure 4). We first estimate the bit duration for both cells using the signal peaks in the non-overlapping region. Following our decoding scheme explained before, we determined from two autocorrelation peaks that the cells were in the third and fourth microfluidic channels (Figure 4(b)). This was also confirmed from the simultaneously recorded images by high-speed optical microscopy (Figure 4(c)). In addition, this approach is also valid to identify cells overlapping within the same microfluidic channel, since it can be considered as interference of the same digital spreading code with its own time-shifted version.

In addition to detecting the presence of cells in microfluidic channels, our technology can also resolve their timing. This is because our digital codes are very sensitive to time-shifts and produce sharp autocorrelation peaks only at zero time delay. For example, we measured the time delay between the two cells in Figure 4 to be ~ 1.9 ms from the autocorrelation peaks. The same time delay was estimated to be ~ 2.1 ms from the simultaneously recorded video, validating our result.



Figure 4. Decoding of the overlapping signals. (a) Two cells occupy the coding region at the same time and sensor signals with each other. (b) Correlation calculations determine which channels cells were in and also the amount of time delay between them from autocorrelation peaks. (c) Simultaneously recorded images using high-speed optical microscopy validate results.

Finally, the peak amplitudes in autocorrelation plots should be interpreted carefully as they are affected by several factors including the size of the cell, the proximity of the cell to surface electrodes and interference in the case of overlapping events. First, larger cells lead to larger signals and therefore higher autocorrelation peaks. For example, optical image in Figure 4 shows that the cell in the fourth microfluidic channel is slightly larger and is also associated with the larger autocorrelation peak. Second, the closer the cell is to the surface electrodes, the larger the signal and hence the autocorrelation peak is. To minimize the variations due to this effect and also to utilize it in increasing the sensitivity, we designed our microfluidic channel to be 15 µm-high. Third, the peak autocorrelation value is affected by the cross-correlation properties of digital spreading codes when cells overlap. While the use of orthogonal digital codes such as Gold sequences minimizes this effect, peak autocorrelation value might still change due to constructive or destructive interference.

CONCLUSION

Combining the techniques from telecommunications and microfluidics, we have introduced a scalable electronic sensor technology, microfluidic CODES, to detect particles in multiple microfluidic channels from a single electrical output. Microfluidic CODES incorporates a multiplexed network of resistive pulse sensors into a microfluidic chip such that each sensor produces a distinct digital code when a particle is detected. These digital codes were designed using the principles of coding in CDMA telecommunication networks and could be uniquely recovered through simple mathematical calculations even when they interfere. We also demonstrated that our technology could readily be applied to detect human ovarian cancer cells on a multi-channel Microfluidic CODES offers a simple, microfluidic chip. all-electronic interface to spatially track particles on microfluidic devices, which will potentially be useful in creating integrated low-cost lab-on-a-chip assays.

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HIGH HEAT FLUX EVAPORATIVE NANOPOROUS SILICON MEMBRANE DEVICE FOR ADVANCED THERMAL MANAGEMENT

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ABSTRACT

We report the design, fabrication and characterization of a novel nanoporous membrane evaporation device to address ultrahigh heat fluxes in advanced electronics. Our design leverages high capillary pressures (~500 kPa) generated by a liquid wetting 130 nm diameter pores of an ultra-thin (600 nm) silicon membrane, suspended over a microfluidic supply network. The device was microfabricated in silicon and tested using methanol as the working fluid. We demonstrated a heat dissipation over 600 W/cm² with a heat transfer coefficient more than 15 W/cm²K, which suggests this approach is promising as a high flux thermal management strategy.

INTRODUCTION

Heat dissipation is a critical limitation in a range of electronic devices including microprocessors, solar cells, laser diodes and power amplifiers. Advanced thermal management solutions using phase change heat transfer are one of the most promising approaches to address these challenges. While liquid-vapor phase change techniques such as flow boiling in microchannels have been thoroughly investigated, significant limitations associated with flow instabilities and power consumption prohibit practical implementation [1, 2]. Meanwhile, capillary driven evaporation structures such as those used in vapor chambers and heat pipes, e.g. sintered particles, are limited by capillary dryout. This process occurs when the viscous pressure resistance overcomes the capillary pumping potential which leads to the wick drying out. Water is the highest performing fluid in traditional capillary pumped wicks due to its high surface tension and high latent heat relative to its viscosity. Since the capillary and viscous pressures are coupled to pore size, the highest critical heat flux (CHF) are for wicks with particle sizes of 250 - 355 µm using water and yield ~500 W/cm² during nucleate boiling in the wicking structure but less than 100 W/cm² in pure evaporation [3], *i.e.*, without nucleate boiling. The heat transfer coefficient in pure evaporation is low as a result of the conduction resistance through the wick which is typically 0.6-1.2 mm thick. Advancements have been made using bi-porous wicks which leverage high capillarity pores with high permeability mesh [4-7], however the thickness of the structures $(135 - 250 \mu m)$ still limits the heat transfer coefficient before the onset of boiling.

In this work, we designed a device where the wicking structure has a thickness of only 2.6 μ m and consists of a suspended nanoporous membrane in which high capillarity pores (130 nm diameter, 0.6 μ m long) are bonded to a high permeability liquid supply network of channels that are 2 μ m wide, 2 μ m high and 200 μ m long (Figure 1). Each section of the membrane is 200 μ m wide, 10 mm long and bonded to the liquid supply channels which are connected to a manifold. The liquid entering the device is distributed by the manifold which is 100 μ m wide and 10 mm long. With an alternating array of membrane and manifold channels, areas larger than 1 cm² can be cooled. Some of the liquid bypasses the device while some liquid is drawn into liquid supply microchannels by the capillarity of membrane pores where it evaporates from the nanoporous membrane surface. This configuration leverages the passive operation and stable, self-regulating nature of heat pipes while extending the heat flux in the pure evaporation regime by $10\times$ compared to traditional wicks. The capillary pressure generated by 130 nm pores is large enough to reach ~1000 W/cm² even with dielectric fluids which exhibit low surface tension and low latent heat compared to water. Despite the lower latent heat compared to water, dielectric fluids are generally highly volatile which results in a higher heat transfer coefficient. Furthermore, the use of dielectric



Figure 1: Cross-sectional schematic of supported membrane structure. Liquid flows through a series of manifold channels (shown in green) and is wicked into the liquid supply channels (shown in red) before flowing through the membrane pores and evaporating from the liquid-vapor interface.

978-1-940470-02-3/HH2016/\$25©2016TRF DOI 10.31438/trf.hh2016.102 Solid-State Sensors, Actuators and Microsystems Workshop Hilton Head Island, South Carolina, June 5-9, 2016 fluids is advantageous for cooling power amplifiers because the circulation of an electrically conducting liquid causes a magnetic field which interferes with electronic performance.

Evaporation from nanopores has been investigated experimentally in previous work. Xiao et. al. demonstrated sustained negative liquid pressures in 150 nm alumina membrane pores during evaporation with isopropyl alcohol [8]. Narayanan et. al. demonstrated heat dissipation of 600 W/cm² for hot spots using 50-100 nm alumina pores and FC-72 as the working fluid, but the heat transfer coefficient was limited by conduction through a liquid film under the membrane [9]. In the present study, the membrane is supported by a high conductivity structure which also defines the liquid microchannel supply network. Using this design, the suspended membrane structure can be scaled to areas larger than 1 cm² and achieve higher heat transfer coefficients compared to thicker wicks while dissipating even higher heat fluxes. While we have previously developed a model to predict the performance of evaporation from our suspended membranes [10-12], here we demonstrate the successful fabrication and experimental validation of our proposed design.

FABRICATION

We fabricated the device using a two-wafer stack consisting of an SOI wafer bonded to a silicon wafer (Figure 2). Pore size uniformity is critical for evaporation from nanoporous membranes because the largest pore limits the maximum capillary pressure. Meanwhile, the interfacial area for small pores is lower which decreases the heat transfer coefficient. Among lithography techniques for patterning nanopores, electron beam lithography is too slow to cover 1 cm² and block copolymers have too many defects. We chose interference lithography since it is well suited



Figure 2: a) Pores defined with interference lithography and etched into SOI wafer. b) Microchannels defined with projection photolithography and etched into the silicon wafer. c) Cross-section of suspended membrane structure after bonding wafers and deep reactive ion etching through the backside of the SOI wafer.



Figure 3: a) Top down view of sample with magnified view of suspended nanoporous membrane. The supporting ridges are faintly visible through the membrane pores. b) Back side of sample with magnified view of RTD within serpentine heater.

based on its high uniformity, low defect density, and fast exposure time. We first defined a pattern for 130 nm holes on the device layer (black), with a 200 nm pitch using a 325 nm HeCd laser for interference photolithography. The silicon device layer was selectively etched to high aspect ratio (5:1) with reactive ion etch using HBr + O₂ chemistry (Figure 2a) using the buried oxide as an etch stop. Also on the SOI wafer, the manifold was etched through the device layer, buried oxide and 140 μ m into the handle layer (green). On the silicon wafer (red), we etched trenches, which serve as liquid supply microchannels. The walls between the trenches are 0.5 µm thick and serve as structural support as well as a thermal conduction pathway for the nanoporous membrane (Figure 2b). Smooth sidewalls were essential in the microchannels to prevent nucleation, therefore, the trenches were dry etched using a continuous flow of $SF_6 + C_4F_8$ rather than alternating the chemistry which produces scalloped sidewalls. Next the SOI wafer and silicon wafer were fusion bonded together to form a monolithic evaporation structure. Once bonded, the membrane was released by etching through the backside of the SOI using the buried oxide as an etch stop (Figure 2c). The buried oxide (gray) was then dry etched with CHF3 which has high selectivity to silicon dioxide. Finally, platinum heaters and resistive temperature detectors (RTDs) were deposited 200 nm thick by electron beam evaporation and liftoff to emulate the high performance IC and measure the device temperature during experiments, respectively. Gold was deposited over electrical contact pads and metal traces to improve localization of heating. Silicon nitride was deposited using PECVD below the platinum and above the gold for electrical insulation. The liquid inlet and outlet ports were etched through the silicon wafer to interface with the liquid manifold.

The sample used for experiments has two membrane sections, which are spaced 200 μ m apart, 10 mm long and each nominally 200 μ m wide (Figure 2a) although the actual width is only 162 μ m due to etch non-uniformity through the handle layer (Figure 2c). The platinum heaters, shown in Figure 3b, are 200 μ m wide and located directly underneath each patch of membrane such that the



Figure 4: Experimental setup schematic. Degassed liquid is delivered to the device from a reservoir. Liquid is siphoned into the microchannels by capillarity of the pores, evaporates at the membrane surface and condenses on the chamber walls.

total heated area is 0.04 cm^2 . The average pore diameter is 135.7 nm and the membrane porosity is 0.378 (Figure 3a).

EXPERIMENTAL SETUP

We experimentally characterized the devices in an environmental chamber that can be pumped to vacuum (~3 mTorr) and then back-filled with pure vapor as shown in Figure 4. Methanol was chosen as a working fluid for this work because its surface tension (0.021 N/m), viscosity (443×10⁻⁶ Pa/s) and latent heat capacity (1145 kJ/kg) at 40 °C are similar to dielectric fluids such as pentane and R245fa. Although methanol is not a dielectric liquid, it is less flammable than pentane and has a lower vapor pressure than R245fa such that it is stable as a liquid at room temperature. The liquid was degassed by pulling vacuum on the reservoir then the liquid was distilled by vaporizing it in one reservoir and the condensing the vapor into a second reservoir. The liquid methanol was then delivered to the sample by heating the reservoir to pressurize it. Heat applied to the sample was measured using current and voltage from an external power supply, although not all of the applied heat was dissipated by evaporation. The time-dependent parasitic heat loss to the sample holder was estimated by heating the sample in a dry ambient. The heat loss due to sensible cooling was calculated using the flow rate and temperature change of the liquid flowing through the sample. The remainder was dissipated by evaporation and is normalized by the heater area to determine the evaporative heat flux. The evaporative heat flux accounts for between 72-91%



Figure 5: Calibration curve and linear fit for platinum RTDs deposited on the bottom of the sample. The inset shows the data deviate by less than $0.1 \,^{\circ}$ C from the linear fit.



Figure 6: Experimental results of temperature rise as a function of evaporative heat flux. Modeling predictions account for conduction resistance in the 650 μ m substrate, 2 μ m supported membrane structure and interfacial transport resistance.

of the applied heat flux depending on the substrate temperature and flow conditions.

Before experiments, the platinum RTDs were calibrated inside the environmental chamber by heating and insulating the outside of the chamber with four independent PID controllers. A T-type thermocouple in contact with the sample was used as the reference temperature and the data was fit with a linear regression as shown in Figure 5.

RESULTS

The plot of temperature rise as a function of heat flux is shown in Figure 6 where the temperature rise is the difference in RTD temperature on the back side and the ambient vapor temperature. The data are shown alongside our developed model [11, 12] which captures conduction resistance in the supported membrane structure along with the interfacial resistance. The intrinsic conductivity in the support (k=39.2 W/mK) and membrane (k=18.2 W/mK) are reduced due to phonon scattering at small length scales. The model accounts for sub-continuum and non-equilibrium transport in the Knudsen layer above the liquid-vapor interface. The model includes two bounds which represent the fraction of liquid covering the membrane surface, ξ . The red curve represents the case where the liquid meniscus is confined to the pores, *i.e.*, the liquid area fraction is equal to the porosity, $\xi=0.378$. The green curve represents liquid spreading to cover a larger area fraction up to a limit of $\xi=1$. At low heat fluxes, the radius of curvature of the meniscus is low relative to the pore radius, so the liquid spreads over the membrane surface. Since the liquid interface is a significant thermal resistance to evaporation, the temperature rise is lower when $\xi \rightarrow 1$. As the heat flux increases, the temperature rise increases as liquid recedes into the pore. Above 300 W/cm^2 the data diverged from the model as the membrane began to clog with soluble, nonvolatile contaminants. The error bars in temperature represent the uncertainty in RTD calibration and uncertainty in the reference thermocouple while the error bars in the evaporative heat flux represent uncertainty in calculating the sensible cooling and parasitic loss.

With the enhanced capillarity of sub-micron pores, we experimentally demonstrated dissipation of heat fluxes of 412±54 W/cm² using methanol when normalizing to the heater area as plotted in Figure 6. Normalizing instead by the evaporation area

and including sensible liquid cooling, the total heat flux was 612 ± 28 W/cm² with a temperature rise of 40.9 ± 2.2 K including the substrate conduction resistance. The highest heat transfer coefficient was 16.0 ± 2.3 W/cm²K at 274 ± 10 W/cm² including the substrate conduction resistance. In electronics cooling applications, the substrate may be thinned from 650 µm to less than 100 µm, significantly improving the heat transfer coefficient.

CONCLUSIONS

We designed, fabricated, and characterized a high performance suspended nanoporous membrane device. This was accomplished with an ultra-thin wicking structure which leverages the high capillarity of 130 nm pores coupled with a high permeability microfluidic network. Ultimately, the heat flux was limited by accumulation of contaminants in the working fluid, not by capillarity of the nanopores. In the future, the liquid channels can be configured to constantly flush contaminants out of the sample. This work represents a new paradigm for phase-change heat transfer and promises a new thermal management approach for high power density electronics.

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MICROFLUIDIC FLOW-THROUGH MICROBIAL FUEL CELLS

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ABSTRACT

This paper reports a flow-through (FT) miniaturized microbial fuel cell (μ MFC) using three-dimensional graphene foam (GF) as a porous 3D anode. The FT design allows enhancing nutrient use efficiency for electrochemical reaction between electron-generating bacteria and bio-convertible and minimizing to sustain energy production of μ MFC. The built-in interconnected pore network of the GF anode can not only provide 3D scaffolds for bacterial attachment and colonization, but also serve as a microfluidic porous channel for flowing nutrient solutions over the biofilm formed on the scaffolds. The FT μ MFC provides a volume power density of 745 μ W/cm³ based on the total volume of anolyte chamber.

INTRODUCTION

Microbial fuel cells (MFC) use bacteria to convert organic matter in bio-convertible substrate into electricity [1]. MFCs not only can be used to remove organic matter from wastewater and produce renewable energy [2], but also have been suggested as an in-field energy source to power sensors for environmental and process monitoring [3]. However, currently, the main applications remain confined to laboratory-scale devices. Factors limiting the application of MFCs to natural scale plants include high initial capital cost, especially for electrode construction, membranes, and limited power density that can be achieved [2, 4-9].

Miniaturized MFC (µMFC) technologies have recently received increased attention, because of their potential to deliver significant insights into green power generation and to realize high-throughput screening of different bacterial strains for conversion of substrates to electricity [2, 4-6]. Most variations in µMFCs have been to optimize anode materials, reduce substrates feeding fuel cell microbes, and optimize bacterial strains. Qian et al. developed a µMFC using gold as an anode material with a remarkable 1.5 µL anode chamber [4]. Choi et al. reported their µMFC using an optimal Geobacter-enriched mixed bacterial culture [5]. Ren et al. demonstrated another device with an increased surface-area-to-volume ratio that led to an improved power density [6]. Also, several three-dimensional (3D) micro/nanomaterials have been demonstrated as high performance anode materials of µMFCs, due to their larger surface area for bacterial attachment and colonization, and high electrochemical catalytic activity [7-9]. Inoue et al. utilized carbon nanotubes (CNTs) to demonstrate the hypothesis that the electron transport is based on direct attachment of bacteria on the surface of anode [7]. Mink et al. applied multi-walled CNTs as anode to their µMFC capable of harvesting stable and high power [8]. Our previous work resulted in another µMFC with a 12 µL anode chamber and using electrospun conducting poly(3,4-ethylenedioxythiophene) nanofibers as a 3D porous anode. This device produced a power density of 423 μ W/cm³ using S. oneidensis MR-1 as a biocatalyst [9]. Generally, these µMFCs are featured by large surface-area-to-volume ratio, low material consumption, and short start-up time. Despite these considerable efforts, it still remains challenging in obtaining high-current and high-power intensity for uMFCs, due to their small processing volume and insufficient biofilm formation.

Graphene foam (GF) is a porous conductive structure formed by vapor deposition of graphene onto a 3D mesh of metal filaments. GF has been utilized in electronic devices [10], energy storage and conversion devices [11], and neural tissue engineering. Recently, GF has been used as anode material of medium-to-large size MFCs [10, 12] since GF-based MFCs have relatively large dimensions and demonstrate that the conductive 3D scaffolds are favorable for bacteria colonization and electron transfer. In particular, the pore size of GF is approximately a few hundred micrometers, which enables efficient mass transfer of nutrients and easy access of bacteria to the interstitial space within the 3D anode in order to grow on the surface of scaffolds.

It should be noted almost all existing MFCs employs a similar device structure (with limited variations) where carbon-containing organic substrate solutions flow over the surface of a planar metal anode (e.g. gold and platinum) or 3D micro/nanomaterials based anode (e.g., carbon cloth, CNTs, and GF) laid on the bottom of anolyte chamber or attached to a proton exchange membrane (PEM). During the batch mode operation, mass transport of nutrients to the microbes colonized on the surface of anode is often implemented through a slow diffusion process from the bulk solution outside the anode to the surface or the inside of the anode. In a continuous flow mode, some of the bio convertible substrates are inevitably wasted as they directly flow out from the anolyte chamber through the freeway space outside the anode, without participating in the reaction with the colonized microbes on the surface of anode.

Thus, it is critical to improve the efficiency of electrochemical reaction between the bioconvertible substrates and the colonized microbes in miniaturized anolyte chambers. With continuing efforts in µMFCs, the volume of substrate solutions decreases dramatically, thus necessitating increasing the efficiency by which the nutrients are made available to microbes colonizing the anode. Here we propose that a porous 3D GF enabled flow-through (FT) mechanism would increase nutrient use of µMFC for microbial colonization and reduce response time of electricity generation (Fig. 1). The porous GF anode is embedded in the anolyte chamber and sandwiched between a PEM and a gold electron collector at the bottom of the anolyte chamber. With the built-in interconnected 3D pore network, the GF anode can not only provide 3D scaffolds for bacterial attachment and colonization, but more importantly serve as a natural microfluidic porous channel for flowing nutrient solutions over the biofilm formed on the scaffolds. The catholyte chamber located on the other side of the PEM contains carbon cloth, which acts as the cathode. A unique feature of this µMFC design is that the porous GF anode provides numerous passages for nutritional media to flow through the anode. By minimizing freeway space, nutrient solutions are not wasted through flowing over the anode without interacting with the bacteria colonizing the porous scaffold anode. As a result, nutrient use efficiency should be improved as a consequence of efficient mass transport driven by pressure and fast diffusion of nutrients directly inside the interstitial pores of the GF. Over the micrometer length scale, molecular diffusion is fast because the diffusion time scales as the square of the distance.



Figure 1: (a) Schematic of the proposed FT (left) and non-FT μ MFC devices (right). (b) Schematic of the device components for the FT μ MFC. (c) Optical image of the porous analyte chamber with an embedded 3D GF anode. Diverging microfluidic channels distribute the analyte solution flow in a relatively even manner across the width of the chamber. (d) Photo of a $\sim 1 \times 1 \times 3$ inch³ array of six FT μ MFCs. (e, f) Scanning electron microscopic images of the porous GF anode with a bacterial biofilm formed on the surface of the GF scaffolds. A close up view in image (f) shows the microbial colonization of the scaffolds in (e).

EXPERIMENTAL

The following materials were used for fabrication of the proposed μ MFCs: GF (multilayer graphene film on nickel foam; thickness: 1.2 mm; pore size: 580 μ m; Graphene Supermarket, Calverton, NY), PEM (Nafion 117; Fuel Cells, College Station, TX), carbon cloth (Fuel Cell Store, College Station, TX), acrylic sheets (TAP Plastics, Oakland, CA), glass slides (Corning, Oneonta, New York), a photopolymerizable precursor solution for making microfluidic channels.

Tryptic soy broth (TSB, Sigma-Aldrich, St. Louis, MO) was used as a culture medium for *S. oneidensis* strain MR-1. A lactate defined minimal medium used for electrochemical measurements. Potassium ferricyanide (Fisher Scientific, Fair Lawn, NJ) was used as catholyte solutions. For studying electrochemical activity of GF anode electrode, the phosphate buffered saline (PBS) containing 5 mM [Fe(CN)₆]^{3-/4-} (pH = 7.0) was used, where [Fe(CN)₆]^{3-/4-} acted as a redox mediator.

The μ MFCs were sterilized by filling all of compartments with a 70% ethanol in water and letting it sit for 20 min at room temperature, followed by flushing the device with sterile DI water for 5 min prior to TSB culture medium for 5 min. The TSB medium flowed into the anolyte chamber through the polyethylene tubing using a programmable syringe pump (210P, KD Scientific, Holliston, MA). To minimize possible oxygen contamination in the batch mode operation, the tubing was closed by steel clamps after the injection of bacterial suspension into the anolyte chamber. The catholyte solution of potassium ferricyanide was supplied using a syringe pump.

An external resistor (*R*) was connected between the anode and cathode to form a closed circuit. The voltage potential (*U*) between the two electrodes was measured using a data acquisition device (Model DI-245; DATAQ Instruments, Akron, OH) and recorded once a minute via DATAQ Instruments Hardware Manager software. The current (*I*) flowing through the resister was calculated *via I* = *U*/*R* and the output power was calculated *via P* = *U*×*I*. Electrochemical properties of the GF anode was measured in PBS containing 5 mM [Fe(CN)₆]^{3-/4-} by an electrochemical workstation (SP1, Zive Potentiostat, Seoul, Korea). A platinum (Pt) wire and a silver/silver chloride (Ag/AgCl) wire were used as the counter and reference electrodes, respectively. Each electrical measurement result given in this paper is representative of the typical result obtained over three independent experiments on three devices.

RESULTS AND DISCUSSION

To illustrate how the FT design affected fluid flow in the anolyte chamber, we conducted hydrodynamic simulation using a finite element method based commercial software package (COMSOL Multiphysics). Fig. 2 shows simulated flow rate distributions of the FT design with the GF anode sandwiched by the PEM and the glass slide, and two non-FT counterparts with freeway space height of three and six times the thickness of the GF anode, respectively. Free- and porous-media flow models were used for the simulations. For the porous media, the porosity, permeability, and Forchheimer coefficient were set as 0.9, 1×10^{-7} m², and 4.35×10^{3} kg/m⁴, respectively. In the settings for fluid properties, the density, dynamic viscosity, and flow rate were set as 1×10^{3} kg/m³, 1.02×10^{-3} Pa·s, and 4.6×10^{-4} cm/s (calculated by a sample volumetric flow rate of 10μ L/hr used in the experiment), respectively. The width and side length of the porous media were set to 1.2 mm and 5 mm, respectively, based on the geometric parameters used in the device.

Fig. 2a shows that as the pressure driven laminar flow was pumped through the FT porous anolyte chamber, the fluid velocity approaches zero the closer to the walls generating a parabolic velocity profile within the chamber in each case. The parabolic velocity profile has significant implications for the distribution of molecules transported within the anolyte chamber. Specifically, in the FT device all fluids would travel through the porous anode, while in the two non-FT systems (Fig. 2b, c), only a portion of the fluids directly interacted with the GF anode, with a majority of the fluids flowed through the freeway space channel as waste. The wider the freeway space, the smaller the flow rate inside the porous anode and the lower the efficiency use of the medium. Importantly, the FT design will allow for faster nutrient replenishment due to the presence of a higher flow rate inside the GF anode.



Figure 2: Simulated flow rate distributions over the vertical cross-section of the analyte chambers of three sample μ MFCs: a FT (a), and two non-FT devices with the freeway space height (above the GF anode) 3 (b) and 6 times (c) the thickness of the GF anode.

Electric current output of the FT device before and during cell inoculation was monitored using a closed circuit carrying an external resistive load of 11.5 k Ω . When TSB culture medium was injected into the anolyte chamber the background current without S. oneidensis bacteria was as low as 12±10 nA. When TSB with S. oneidensis cell suspensions was injected into the anolyte chamber the output current increased to $\sim 43 \ \mu A$ within five hours and then decreased gradually over time. Fig. 3 shows the polarization and power density curves of the FT device plotted by measuring the output voltage and current at different external resistive loads. According to the literature [1], a polarization curve can be divided into three regions reflecting activation loss, ohmic loss, and mass transfer loss. At the activation loss stage, the output current increased from zero (measured at the open circuit voltage of 1240 mV) to 18.1 µA. The voltage dropped as the external resistance decreased. In the current range of 18.1 to 44.2 µA, there was a near-linear drop in voltage with increasing electric current. As a result, an internal resistance was estimated to be 7.3 k Ω through linear fitting of the curve in the ohmic loss region. In addition, the surface power density reached a maximum of 89.8 μ W cm⁻¹ in the ohmic loss region, while the output voltage decreased in the mass transfer loss region.

The device provided 745 μ W/cm³ volume power density based on the total volume of anolyte chamber, and 89.4 μ W/cm² surface power density and surface current density of 91.8 μ A/cm² based on the planar surface area of GF anode.



Figure 3: Polarization curve (black) and power density output (red) curves of the FT μ MFC as a function of current.

To evaluate the influence of the feeding rate of culture medium on the electric current generation of the FT device, TSB medium was continuously injected into the FT anode at different flow rates after inoculating the chamber with S. onediensis. As shown in Fig. 4a, the output currents at the low flow rates were similar ($\sim 5 \mu A$) to each other. But, with increasing flow rates at or above 10 μ L/hr, there were dramatic current increases. It is interesting that the electric currents of FT and non-FT at flow rates between 10-40 µL/hr were very different which FT anode researched to the level of 50 µA, but only a small current increase as the flow rate increased on non-FT anode. Fig. 4b shows the time (t_1) that the FT device and six non-FT counterparts took to generate 80% of their corresponding peak current with respective flow rates of 20, 40, and 60 µL/hr. The result shows that the FT design allowed for significant reduction in t_1 . The larger the freeway space volume of the non-FT device, the longer the time t_1 of the device. Along the length direction of the GF anode in the FT device, mass transport of nutrients was mainly driven by pressure in the continuous flow mode. As a result, the higher the feeding flow rate the shorter the time t₁ required. Inside the interstitial pores of the GF anode, nutrients could diffuse to the surface of scaffolds over a short length of approximately 290 µm or half of the mean pore size of the GF, which also contributed to the short t_1 of the FT device. In contrast, the non-FT devices required more time because the nutrients in the freeway space outside the anode diffused over a longer distance to the scaffolds of the GF anode. Although pressure-driven mass flow also occurred in the non-FT devices, the effective amount of nutrients delivered to the colonized microbes was actually less than that in the FT device. As shown in Fig. 4b, when the medium flow rate was set at 20, 40, and 60 μ L/hr, the response time t₁ of the FT device was 4.2, 3.2, and 2.6 times, respectively, shorter than that of the non-FT device with $V_{\rm fr} = 6V_{\rm gf.}$ Fig. 4c shows the total medium volume consumed to obtain 80% of the peak current increases with increasing freeway space volume. The result demonstrates that with the FT design, the medium consumption reduces by up to 16.4 times and the response time pf the device reduces by up to 4.2 times.

Lastly, to reveal the benefit of the FT design, the electrochemical properties of the GF anode are investigated by cyclic voltammetry and electrochemical impedance spectroscopy studies (no figure is shown here) with the lactate defined minimal medium. Compared to the non-FT device, the FT device has a

higher diffusion coefficient ($6.28 \times 10^{-9} \text{ cm}^2/\text{s} \text{ vs. } 1.40 \times 10^{-9} \text{ cm}^2/\text{s}$), a desired lower charge transfer resistance R_{ct} (0.88 vs. 2.96 kohm), and a higher electron transfer rate k_0 ($1.13 \times 10^{-10} \text{ cm}^2/\text{s} \text{ vs. } 2.35 \times 10^{-11} \text{ cm}^2/\text{s}$).



Figure 4: (a) Maximum output current of the FT and non-FT devices at different TSB medium flow rates. The non-FT devices used here have the freeway space volume $V_{\rm fr}$ varying from $V_{\rm gf}$ to $6V_{\rm gf}$ ($V_{\rm gf}$ represents the volume of GF anode). (b) Time required for the FT and non-FT devices to obtain 80% of the peak output current as a function of freeway space volume of the devices. (c) Total volume of TSB medium consumed to obtain 80% of the peak output current as a function of freeway space volume of the devices.

CONCLUSION

We have demonstrated a FT μ MFC with a porous GF anode sandwiched by a PEM and an electron collector at the bottom of the anolyte chamber. Mass transport of nutrients was mainly driven by pressure. In addition, molecular diffusion of nutrients to the biofilm on the scaffolds occurred directly inside the pores of the GF over a short length scale. Therefore, the present FT design allowed reducing bioconvertible substrate consumption, enhancing nutrient use efficiency to sustain energy production of the biofilm, and shortening response time of current generation.

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RECONFIGURABLE ION SELECTIVE SENSOR ARRAY ENABLED BY DIGITAL MICROFLUIDICS

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ABSTRACT

This paper presents a proof of concept demonstration of a reconfigurable ion selective electrodes (ISE) array enabled by an electrowetting on dielectric (EWOD) digital microfluidics, for the first time. The on-chip preparation of an ISE array includes electroplating Ag followed by forming AgCl layer by chemical oxidation and forming a thin layer of arbitrary ion-selective membrane liquid on a sensor electrode. The calibration curve of fabricated ISE shows complete agreement with conventional ones. The proposed device with the capability of on-chip ISE fabrication has many advantages such as automation simplicity, minimal membrane consumption, longer lifetime and easy integration in lab-on-a-chip (LOC) platforms.

INTRODUCTION

In an EWOD digital microfluidics, liquids are manipulated in droplet form by applying electrical potential to series of electrodes with dielectric layer on top [1]. Many superior capabilities of digital microfluidics over channel-based microfluidics, such as multiplexing capability, have been demonstrated. The precise control over working liquids and reagents makes them suitable for lab on a chip and micro analysis platforms [2]. In general, lab-onchip (LOC) devices consist with two main units - the liquid handling unit and the chemical analysis unit. Over the past decades, there has been significant improvement in both areas. However, complete integration of different fluid handling and sensor technologies remains as complex tasks. There has been efforts to integrate optical sensors in digital microfluidics [3], but such integration is not IC compatible nor easy to use. In general, having two (or more) units with different working principles in one microsystem makes the system bulky and less attractive for commercial use.

With improvement in solid-state micro-electrochemical sensors and growth of bio electrochemical sensors in the past two decades [4][5][6], electrochemical analysis began to draw attentions again due to their simplicity and easy fabrication. Various methods support electrochemical sensing - cyclic voltammetry, amperometry, potentiometry and impedance spectroscopy, etc. Recently, an amperometric sensor was integrated into EWOD device to analyze ferrocene methanol and dopamine [8]. Also Liu et al reported integration of conductometric sensors on microfluidic LOC devices [9]. However, integrating potentiometric sensors, wherein the signal is not dependent on the area of the sensor, has not been popularly considered due to oxidation of electrode which results in shorter lifetime of the sensor [10].

To address the challenge of short sensor lifetime, in this paper, we report an ion selective potentiometric sensor fabricated on-chip with the help of EWOD. This new method allows users of LOC device to reconfigure the sensor array online without any interruption or the disassembling of the device while increasing the lifetime of the sensor. In addition, it will enable on-demand sensor functionality of LOC devices – type of sensors are not predesignated but user can determine it at the time of use.

This present work focuses on testing the proposed on-chip

sensor fabrication and evaluating the performance of the fabricated sensor by comparing it with that of conventional ISEs.

EXPERIMENTAL

Material

Au/Cr coated wafer was used to fabricated EWOD electrodes and the seed layer of ISEs. S1813 (MICROPOSIT) was used as the photoresist for patterning and mask layer in different stages of fabrication. SU-8 2005(Micro-Chem) was used as dielectric layer. The solution of Teflon AF1600S (DuPont) powder dissolved in Fluorinert FC-40 (Sigma-Aldrich) was spin coated to form hydrophobic layer. 1025 RTU (TECHNIC INC) was used for Ag electroplating solution. HCl analytical reagent grade was used for formation of AgCl layer. For Potassium ion selective membrane which contains 1 wt % of potassium ionophore I, 0.5 wt % potassium tetrakis(4-chloropheny) borate, 49.5 wt % bis(2ethylhexyl)sebacate and 49 wt % PVC were used; these materials were purchased from Sigma-Aldrich. Tetrahydrofuran (THF-T397-4) was used as an organic solvent and carrier liquid for potassium ion selective membrane. For acquiring the calibration curve, different molarities of KCl solution (1µM-1M) were prepared by dissolving potassium chloride (Sigma-Aldrich) powder in Deionized water.



Figure 1: two-chip configuration of EWOD DMF integrated with ISE.

Design & configuration of EWOD-ECC

Figure 1 shows the configuration of the EWOD electrochemical cell (EWOD-ECC). The top plate is coated with indium tin oxide (ITO) to provide a ground electrode for EWOD motion as well as a cathode electrode for electroplating. The bottom plate contains

patterned gold (Au) electrodes for EWOD operating electrodes as well as sensor electrodes. Although the use of Au electrodes is common to cyclic voltammetric and amperometric sensors, Au electrodes cannot deliver a stable voltage in potentiometric applications. Whereas Ag/AgCl electrodes can establish the required stability for the voltage measurement, thus Ag/AgCl electrodes are commonly used as reference electrodes in electrochemical sensors. The significant improvement in the fabrication of Ag/AgCl electrodes through electroplating and chemical anodization methods lends even higher stability for EMF measurement [11] yet the short lifetime of Ag/AgCl electrodes still exist. Therefore, in this study, we present the DMF platform where we fabricate our Ag/AgCl electrode on-demand and on-chip as demonstrating the renewal of worn-out reference electrodes surfaces. For liquid handling, we used six reservoirs with the capability of dispensing droplets (~650 nL) and 45 electrodes (2 ×2 mm² each) for EWOD driving electrodes. For sensing, we designed four electrochemical cells with sensor electrodes (400 µm diameter) integrated with EWOD electrode. The hydrophilic-tohydrophobic ratio (i.e. sensor to EWOD electrode area) was 3.1%; this ratio is critical in order not to hinder the droplet motion.



Figure 2: Fabrication process of sensor electrodes integrated in the bottom plate of EWOD digital microfluidic chip.

EWOD-ECC fabrication

Figure 2 shows the fabrication of EWOD-ECC. The electrodes of EWOD device and sensor electrode were patterned

by standard lithography followed by wet chemical etching of Au/Cr layer. Su8-2005 was then spin coated and flood-exposed to form a dielectric layer over the EWOD electrodes with the thickness of 5 μ m. Afterwards, a Teflon layer (300 μ m) was spin-coated and baked to form a hydrophobic coating. This was followed by reactive ion etching treatment of Teflon surface for 5 seconds at 140 Watts and 30 sccm Ar rate. This step makes the Teflon surface ready for the spin-coat of S1813 to form RIE etching mask. Then, after selectively etching the Teflon layer over the sensor electrodes for 45 minutes with the power of 200 Watts and 30 sccm Ar rate, the remaining PR layer was exposed to UV light (flood exposure) to get weakened and removed by dissolving in acetone solution. Finally, after annealing the chip in 200 °C oven for one hour, the Teflon surface recovered its hydrophobicity properties and got ready for operation.

On-chip ISE fabrication and calibration

To carry out the on-chip fabrication of Ag/AgCl sensor electrodes (Figure 1), a droplet (~650 nL) of Ag plating solution was dispensed from the reservoir and brought over the sensing site by EWOD microfluidic actuation (Step 1 in Figure 3). Electroplating was driven at 12 mA/cm² for 300 seconds. To avoid Ag+ depletion, plating solution droplets were replenished. Similarly, a droplet of 0.1 M HCl solution was dispensed and brought over the Ag-plated electrodes (Step 2 in Figure 3) and applied to the surface for 45 s to form AgCl layer. Lastly, a droplet was dispensed from membrane liquid reservoir and brought over a sensor electrode to form the layer of an ion selective membrane (ISM) (Figure 4(c)) and Step 3 in Figure 3). Due to the wettability contrast, the liquid membrane was pinched-off at the hydrophilic opening on the sensor electrode (Figure 5). After evaporation of THF, a thin layer of the gel-like membrane has been formed over sensor electrode.

Electromotive force (EMF) was measured with high input impedance $(10^{12}\Omega)$ using HP 34401A multimeter at room temperature (21 °C) in an electrochemical cell fabricated on the chip.



Figure 3: Fabrication on-chip and serial dilution and calibrating procedure. Step (1): Ag electroplating, Step (2): chemical oxidation of Ag layer, Step (3): forming a thin layer of ISM on a sensor electrode and step, and Step (4): serial dilution for calibrating sensor.



Figure 4: Process flow of on-chip fabrication of ISE. (a) Silver electroplating, (b) chemical oxidation of Ag layer with HCl to form AgCl layer, and (c) forming of a thin layer of potassium selective membrane on sensor electrode.



Figure 5: Ion-selective membrane (ISM) pinching-off process; (a) an ISM liquid droplet brought over a sensor electrode by EWOD motion, (b) the ISM droplet being driven away from the sensor electrode while a part of ISM wetting the hydrophilic opening over the sensor electrode (c) a thin layer of ISM on sensor electrode after the completion of pinch-off.

RESULTS & DISCUSSION

The theory of potentiometric sensors is based on Nicolskii-Eisenman formalism (1) which derived from Nernst equation:

$$E_{cell} = E_{cell}^0 \square (RT/zF) \ln(a_i(I))$$
⁽¹⁾

Where, $a_i(I)$ is the primary ion activity in the sample.

We used Ag/AgCl for both electrodes and used potassium ISM on one of the electrodes, therefore our measured electromotive force is related to the activity of potassium in the solution.

The standard calibration curve for the potassium ion selective sensor was performed on-chip. Serial dilution of standard KCl solution by DI water was done on the designated mixing area (Step 4 in Figure 3) to prepare 11 different molarity $(10^{-6} - 10^{-1} \text{ M})$ solutions. Droplets of each molarity were brought over the on-chip fabricated ISE; then electromotive force was measured (Figure 6). Figure 7 shows the Nernstian response to solutions as indicating that the on-chip fabricated ISE work with complete agreement with typical ISEs.



Figure 6: Measuring electromotive force on EWOD



Figure 7: Nernstian response to different molarity of KCl solutions

CONCLUSION

This successful demonstration promises that the proposed reconfigurable ISE can provide a sensor device where a user can define and form desirable ISEs instantaneously at the time of use. Simultaneous (and serial) detection of multiple analytes will be available. Also, ISEs will be renewable by adding dissolving membrane and washing electrode steps followed by another membrane formation step.

The proposed platform solved the main challenge of the potentiometric sensor by adding lifetime to sensor electrodes while adding a reliable and reproducible analysis unit to lab-on-a-chip devices. This method of fabrication can pave the path to low cost and compatible home-use sensors with using benefits of EWOD such as ease of use and automation and minimal consumption of reagents.

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A BIOLOGICAL FUEL CELL MICROFABRICATED WITHIN A SINGLE SHEET OF PAPER

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ABSTRACT

We report a novel technique for fabricating microbial fuel cells (MFCs) within a single sheet of paper, which greatly (i) reduces fabrication cost/processes of the fuel cell devices and (ii) revolutionizes their mass production for the potential use as a disposable battery in resource limited settings. A number of device units were simply batch-fabricated on a flexible/scalable single sheet of paper by spraying/screen-printing electrode materials and forming hydrophilic regions with hydrophobic wax. A 3-D MFC structure was created by forming four functional layers of an anode, a hydrophilic reservoir, a hydrophobic wax-based ion exchange membrane, and an air-cathode in a single sheet of chromatography paper. Upon adding one drop of bacteria-containing liquid onto the device, the hydrophilic reservoir allowed for rapid bacterial cell attachment to the electrode, where bacterial respiration then transferred electrons from the organic liquid to the electrode, generating power to an external load.

INTRODUCTION

Recently, paper-based microbial fuel cells (MFCs) have attracted a great deal of research interest as a disposable and accessible power source in resource-limited regions [1-7]. The paper-based device can generate electricity from bacteriacontaining liquid derived from renewable and sustainable wastewater which is readily accessible in the local environment [7]. Moreover, the intrinsic feature of paper allows for rapid adsorption of bacteria-containing solution through capillary force of paper without external pump/tubing and promotes immediate microbial cell attachment to the electrode, leading to a rapid power generation using a small amount of the liquid [8, 9]. However, the promise of this technology has not yet been translated as practical power applications, because their device configuration is complicated with necessary multifunctional parts such as the anode, cathode, and ion exchange membrane, demanding manual assembly of the device and hampering batch fabrication. In addition, there are potential issues during device fabrication such as misalignment of paper layers and vertical discontinuity between layers, which ultimately decrease power generation. Even newly developed origami MFCs suffer from lack of clamping methods for mechanically maintaining the stacked layers [7].

In this work, we microfabricated the MFC in a single sheet of paper, eliminating the aforementioned major technical hurdles. A 3-D MFC structure was developed by incorporating four functional layers of a nickel and polypyrrole/carbon black (Ppy/CB) anode, a hydrophilic reservoir, a hydrophobic wax-based ion exchange membrane, and an activated carbon based air-cathode in a single sheet of chromatography paper (Fig 1). Since the configuration of the microfluidic reservoir and anode has a great influence on bacterial cells' distribution through the paper matrix, their attachment over the anode, and electron transfer efficiency [10], different shapes of anodes over the reservoir were thoroughly explored in terms of device performance and sample injection efficiency. The device developed in this work greatly reduced fabrication time/cost of the fuel cells and revolutionized their mass production for the potential use as a disposable power source in remote and resource limited regions.



Figure 1. (a), (b) & (c) Photo-images of the biological fuel cell fabricated in a single sheet of paper with different anode/reservoir configurations, (d) a schematic diagram of the device and (e) a microscopic image of the cross section of the MFC.

MATERIALS AND EXPERIMENTAL SET-UP Device Fabrication

All functional layers for the MFC configuration were constructed in a single large sheet of Whatman 3MM chromatography paper (20cm x 20cm), where 25 individual devices were simultaneously batch-fabricated (Fig. 1 & 2). The hydrophobic wax melted into the paper substrate strengthened the paper fiber matrix while retaining its flexibility. The wax was also used as the ion exchange membrane as it provided hydrophobic property of the paper, separating the anode part from the aircathode and allowing protons to pass through efficiently [1]. The penetration depth of the melted wax printed on the bottom side of a paper was carefully controlled by adjusting the heating time (Fig 1e). The anode was prepared on one side of the paper, exposing the cathode to the air for MFC cathodic reactions.

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Figure 2: Schematic illustration of the process employed to create the microbial fuel cell in a single sheet of paper

Hydrophobic wax boundary and ion exchange membrane $(\text{Step }(1)\sim(5))$

The wax boundary, first printed on both sides of the paper (Xerox phaser printer), was formed into the paper substrate by baking in the oven at 130°C for 2.5min (Fig 2 (1)~(3)). Then another film of wax was printed on bottom side of the paper and subsequently baked for 45s at 130°C to form the ion exchange membrane (Fig 2 (4) & (5)). As the penetration depth of wax is proportional to the heating time, different ion exchange membrane thicknesses can be achieved to optimize the device performance.

Air-cathode preparation on paper (Step $(6) \sim (8)$)

Activated carbon (AC)-based air-cathode was constructed over the ion exchange membrane prepared on the bottom side of the paper. The air-cathode was prepared by applying the AC catalysts (CABOT Corporation) (560mg) with a blinder solution (Fig 2 (6)). The blinder solution was prepared by adding a mixture of (i) 1200 μ L of 5 wt% Nafion solution, (ii) 150 μ L of DI water, and (iii) 600 μ L of isopropanol into a beaker, followed by ultrasonication for 1 min. Then, the conductive nickel was sprayed on top of the AC-based cathode to provide stronger structural support and to function as a current collector (Fig 2 (7)), which was subsequently air-dried for 2h (Fig 2 (8)).

Anode preparation on paper (Step (9) ~ (13))

The anode consists of two parts; (i) a bottom Ppy/CB film to improve the bacterial adhesion/biofilm formation and the electron transfer efficiencies [11], and (ii) a highly conductive nickel film for efficient electron collection. The reservoir was filled with deionized (DI) water and frozen in -20 °C for 2 h (Fig 2 (9)) so that the hydrophilic reservoir region was secured from the top side of the paper during the anode preparation. The Ppy/CB solution was



Figure 3: Schematic diagram of the device operating principle and test setup (a), and the photo-image of the device measurement (b).

prepared by suspending 83.5mg of Ppy/CB mixture (Ppy 20wt%, Sigma-Aldrich) in 25mL of 70% v/v ethanol. After we sprayed the Ppy/CB liquid through the patterned mask, the device was air-dried at room temperature for 8 minutes to evaporate the excessive solvent prior to forming nickel film (Super ShieldTM Nickel Conductive Coating, MG Chemical) (Fig 2 (10) & (11)). The device was then air-dried for 24h to remove the residual moisture in the anode reservoir and fully solidify the anodic films (Fig 2 (12) & (13)).

Inoculum

Wild-type *Shewanella oneidensis* MR-1 cultivated in L-broth medium (10.0g triptone, 5.0g yeast extract and 5.0g NaCl per liter) at 35°C for 24 hours was subsequently centrifuged and suspended in fresh L-broth medium. The optical density (600 nm, OD_{600}) of the bacteria suspension was 2.606Å. Since the centrifugal process removed the shuttle-molecules for extracellular electron transfer (EET), the direct contact of bacteria to the anode is considered to be the major method for EET in this application [12, 13].

Operating principle and measurement setup

When the inoculum was added into the inlet of the device, it was transported throughout the reservoir. The rapid adsorption of the paper promoted bacterial cell attachment to the anode, where bacterial respiration transferred electrons from the organic matter to the anode and flow through an external load while the anode was connected to the cathode. The protons were transferred through the ion exchange membrane and reduced with the oxygen and electrons travelled from the anode. Data acquisition system (NI, USB-6212) with a customized LabVIEW interface were used to measure the potential between anode and cathode (Fig. 3). To determine the current generated by the device, external resistors ($10k\Omega$ and $1k\Omega$) were used to connect the anode and cathode, and current flow through the resistor was calculated by Ohm's law.

RESULTS AND DISCUSSION

 80μ L of bacterial inoculum was slowly and carefully dropped on the centers of all the device samples. Upon dropping, the liquid simultaneously spread along the microfluidic inlet channels formed by different anodic configurations and infiltrated underneath the anode. The device samples were first left open-circuit, and then $10k\Omega$ resisters and $1k\Omega$ resisters were subsequently connected to the devices (Fig 4). Once the inoculum was added to the device,



Figure 4: Voltages measured from (a) the devices with different anode coverage, and same microfluidic channel length. And (b) devices with same anode coverage, but different microfluidic channel length.

open circuit voltages (OCVs) and currents with resistors were generated. To maximize the device performance of the device, various anode/reservoir configurations with different microfluidic channel lengths and anode areas were explored in this work [14]. We prepared nine device samples with different anode shapes for two experimental sets. One experimental set had five device samples with the different anode surface areas (or exposed inlet reservoir areas) and the same microfluidic channel lengths (Fig 1b). Another set included five other samples with different channel lengths and the same anode area (Fig 1c). The sample #AP was used for both experimental sets as a reference.

For the first experimental set with the different anode areas (or exposed inlet reservoir areas), the OCVs significantly varied with samples (Fig 5a). The sample #A1 with the smallest exposed inlet area (or the largest anode area) generated the highest OCV while the values decreased with the increase of the inlet areas or the decrease of the anode areas. These results indicate that the larger anode areas provide a greater accessible area for bacteria to attach [15] while the consequently reduced inlet areas decrease the oxygen invasion into the reservoir, ultimately increasing the OCV [16]. In particular, the oxygen penetration into the anode abiotically and biotically reacts with the anode and reduces the OCVs.

When external resistors were connected to the device samples, the higher current densities with the larger anode areas (or smaller exposed inlets) were obtained in the same way as the OCVs (Fig



Figure 5: (a) Open circuit voltages (OCVs) were measured for the first 7 minutes and then all devices were connected to (b) 10 $k\Omega$ and (c) 1 $k\Omega$ external loads and the currents were calculated. (d) The current density increased with higher anode area coverage under different load conditions.

5b & 5c). The highest current density of 0.87μ A/cm² and power density of 55.2μ W/m² under the 1k Ω was obtained from the device with the largest anode coverage (#A1) (Fig 5c & 5d).

On the other hand, the second experimental set exhibited the similar current densities from the devices with the same anode area (Fig 6) even though there was a significant change in the microfluidic channel lengths. The results indicate that the anode area or exposed inlet region is a more critical factor for the device performance than the microfluidic channel architecture. However, it should be noted that the minor variations in the current generation were observed with the microfluidic geometry of the device inlet, showing that the sample #P1 provided more efficient bacteria cell transportation to the anode than the sample #P5.

The single sheet paper-based MFC showed great potential in terms of device fabrication and cost. For higher performance toward the practical applications, the device array can be readily prepared in a large sheet of paper and a series or parallel



Figure 6: Similar current densities were observed on shapes with same anode coverage under (a) 10 k Ω and (b) 1 k Ω load.

connection can be achieved by folding the sheet or by piling up the several sheets of papers.

CONCLUSION

In this work, we demonstrated a novel and practical technique for manufacturing a bio-fuel cell in a single sheet of paper. The single sheet device platform solved the intrinsic issues generated from the conventional paper-based fuel cell devices such as discontinuity between vertical layers, inconsistent connections between different layers, and complicated fabrication procedure. Furthermore, the proposed design provided an inexpensive, timesaving, and user-friendly device platform that would facilitate studies on the fundamental MFC studies such as effects of different anode materials, MFC device architecture and bacterial cells' transportation on its performance. Although further research has to be done to more accurately characterize and improve the proposed design, the current work shows the great potential of the single sheet paper-based MFC as a novel power source.

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A HIGH POWER-DENSITY, SELF-SUSTAINED HYBRID BIO-SOLAR CELL WITH CO-CULTURE OF HETEROTROPHIC AND PHOTOSYNTHETIC BACTERIA

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ABSTRACT

We demonstrated self-sustainable high power generation from a microliter-scale bio-solar cell by using syntrophic interaction between electricity-generating heterotrophic bacteria and phototrophs. The device continuously generated light-responsive electricity from the heterotrophic bacterial metabolic respiration with the organic substrates produced by the photosynthetic bacteria. Without the external input of organic fuels, the bio-solar cell utilizing the mixed culture generated self-sustained current more than 400 times greater than that of the bio-solar cell using only photosynthetic bacteria. The device featured (i) a small-scale microchamber to reduce the start-up time by increasing the probability of cell attachment/biofilm formation, (ii) a carbonbased anodic material to promote bacterial adhesion and (iii) a sandwich configuration of the anode/proton exchange membrane (PEM)/air-cathode to minimize the inter-electrode distance and significantly reduce the internal resistance.

INTRODUCTION

With increasing concerns about the energy crisis and global warming, heterotrophic microbial fuel cells (MFCs) have been a major focus for renewable energy production [1-5]. The MFCs are powered by live heterotrophic bacteria to efficiently degrade a broad range of organic substrates under natural conditions and offer clean and sustainable features. Although the MFCs are capable of generating a high power density, they require a continuous supply of organic substrate for energy, hampering the entirely self-sustainable power supply. On the other hand, bio-solar cells are another type of the MFC that can provide self-sustainable power generation by using the photosynthetic and respiratory activities of photosynthetic bacteria [1, 6-10]. The bio-solar cell can continuously generate electricity from solar energy without additional organic substrates since light energy absorbed by the photosynthetic reaction splits water and generates oxygen, protons, and electrons. Despite their promise as a self-sustainable power source, however, the bio-solar cells have not yet been successfully translated into commercial applications because of their persistent power limits. Their power densities, which are typically several orders of magnitude lower than that of even the smallest power MFCs [11, 12], remain a significant challenge.

Recently, the "Plug and Play" photosynthetic concept has been proposed by the Johnes' group at Arizona State University as a strategy to increase bio-solar cell performance; the light and dark reactions can operate independently, coupling microbial respiratory metabolism to electricity generation using photosynthetic co-cultures [13]. Also, several research teams generated a wealth of new scientific and technological results that demonstrated synergistic cooperation clearly between photosynthetic microorganisms and heterotrophic bacteria [14-16]. However, much of this work is still in its nascent stages; the evolution of this technology will require additional exploration through practical application of established techniques and comprehensive systematic integration.

In this work, we created a microfluidic bio-solar cell that integrated both heterotrophic and photosynthetic bacteria for high power density and self-sustainability. The heterotrophic bacterial



Figure 1: Conceptual illustration of the hybrid bio-solar cell based on the synergistic cooperation between photosynthetic bacteria and heterotrophic bacteria

biofilm (Shewanella oneidensis MR-1), which formed first at the bottom of the anode, oxidized organic substrates and efficiently transferred electrons to the anode while photosynthetic bacterial biofilm (Synechocystis sp. PCC 6803) formed over the heterotrophic bacteria and provided the *in-situ* organic substrates (Fig. 1). Without the external input of organic fuels, the bio-solar cell utilizing the mixed culture generated self-sustained current more than 400 times greater than that of the bio-solar cell using only photosynthetic bacteria. Furthermore, bio-solar cell miniaturization inherently produced favorable conditions for increasing power density by reducing internal resistance and improving mass transport [17, 18] while greater bacterial adhesion was promoted by using the carbon-based anode materials compared to an inefficient conventional inorganic materials [11, 17]. In addition, a sandwich configuration of the anode/proton exchange membrane (PEM)/air-cathode minimized the interelectrode distance and significantly reduced the internal resistance [12].

EXPERIMENTAL SET-UP

Operating principle

In this hybrid bio-solar cell, the main biocatalyst to generate electricity was the heterotrophic bacteria. The bacteria extracellularly transfer electrons to the anode while they oxidize organic substrates in the micro-chamber [19]. The phototrophic biofilm formed over the heterotrophic bacteria captures solar energy to convert carbon dioxide and water (generated from heterotroph's respiratory reactions) into oxygen and organic matter, which are subsequently used for both bacterial respiratory reactions (Fig 1), offering self-sustainable and self-maintainable features. During those reactions, electrons are released through extracellular electron transfer pathways and flow to the cathode through the external electrical circuit. Released protons

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Figure 2: Schematic diagram of the individual layers for the device.



Figure 3: Photo-images of the fully assembled biological solar cell: (a) the bottom view and (b) the top view

simultaneously diffuse from the anodic chamber to the cathode, where they re-combine with electrons and oxygen to re-form water. The photosynthetic bacteria themselves can also be biocatalysts to continuously harvest electrons from microbial photosynthetic and respiratory activities under day-night cycles [20-22]. However, their electron transfer efficiencies are low and not practical to be used as a practical power source [11, 12].

Device Fabrication

The bio-solar cell was assembled as shown in Fig 2 & Fig 3, where the main body of the device was comprised of four polymethly methacrylate (PMMA) substrates and the sandwiched electrode assembly. Five different functional layers were cut by the laser cutting machine (Universal Laser System, VLS3.5): (i) a top PMMA layer, (ii) a PMMA microfluidic chamber layer, (iii) a rubber gasket, (vi) an anode/PEM (Nafion 117)/air-cathode sandwiched electrode assembly, and (v) a bottom PMMA layer. All layers were carefully aligned and assembled with 10 small screws. The device utilized the air-cathode to allow freely available oxygen to act as an electron acceptor by the installation of the catalyst side of the air-cathode to face toward the chamber while the opposite side was exposed to air. The air-cathode was fabricated from 30% wet-proofed carbon cloth with four layers of polytetrafluoroethylene (PTFE) coating [23, 24]. The other side of the cathode was coated with Pt/C catalysts (0.5mg/cm2 Pt loading). The electrodes were pierced with a 0.5mm thick Ti wire as a current collector. The bio-solar cell had two holes for fluidic



Figure 4: Photo-images of (a) the bio-solar cell with cocultures and (b) the enlarged image of the chamber, showing green colored photosynthetic bacteria.

inlet/outlet. Tubes (CODAN, 0.35 mL volume) were plugged into the holes with adhesive to form a fluidic channel. The 1.6mm-thick PMMA microfluidic chamber layer (ii) and 0.508mm-thick rubber gasket layer (iii) were precisely laser-machined to define a 90μ L chamber over the electrodes. The size of a completely assembled device was 6cm x 3.5cm. The assembled device was sterilized with 70% ethanol and ultraviolet light for 24 hours.

Inoculum and Catholyte

Synechocystis sp. PCC 6803 (phototrophs) were grown from -80°C glycerol stock cultures by inoculating 15mL of BG-11 medium with gentle shaking under a 24h light cycle (12h light/dark). The BG-11 contained 1.5 g NaNO₃, 40 mg K₂HPO₄, 75 mg MgSO₄, 36 mg CaCl₂, 1 mg of EDTA, and 6 mg of citric acid and of ferric ammonium citrate per 1 L of distilled water. The continuous aeration and illumination were provided by fluorescent lamps for 2 weeks. Growth was monitored by measurement of the optical density at 600 nm (OD_{600}) and the culture we used reached an OD₆₀₀ of 1.4. Shewanella oneidensis MR-1 (heterotrophs) were grown from -80°C glycerol stock cultures by inoculating 20mL of L-broth medium with gentle shaking in air for 24h at 35°C. The Lbroth media consisted of 10.0g tryptone, 5.0g yeast extract and 5.0g NaCl per liter. The culture we used reached an OD_{600} of 2.0. Both cultures were then centrifuged at 5.000rpm for 5min to remove the supernatant. The bacterial cells were re-suspended in a new medium and used as an anolyte for the device.

Measurement Setup

We measured the potentials between the anodes and the cathodes with a data acquisition system (National instrument, USB-6212), and recorded the readings every 1 min via a customized LabView interface. An external resistor connected between the anode and the cathode closed the circuit. The current through this resistor was calculated using Ohm's law.

RESULTS AND DISCUSSION Biofilm formation

In this work, we constructed a hybrid bio-solar cell comprised of two microbes, namely *Shewanella oneidensis* MR-1



Figure 5: Photosynthetic bacterial biofilm formed on heterotrophic bacterial biofilm. The extracellular polymeric substance of the heterotrophic microbes facilitate the adhesion of phototrophic ones rapidly forming their biofilm.

(heterotrophic) and Synechocystis sp. PCC 6803 (photosynthetic). For better understanding of the biofilm formation, we used our previous microfabricated MFC designed for both optical and electrical studies of the bacteria [25, 26]. The MFC included a transparent thin gold as an anode and a protruded anode chamber, which could be directly placed under an optical microscope for observing live bacterial behavior. After the heterotrophic bacterial biofilm was first formed, photosynthetic bacterial inoculum was introduced (Fig. 4). Although further study needs to be done, we could claim that the biofilm extracellular polymeric substance of the heterotrophic microbes might facilitate adhesion of phototrophic microbes rapidly forming their biofilm. In this way, cultivating photosynthetic bacterial biofilms could be maximized by using co-cultures with heterotrophic bacteria. Fig 5 shows the rapid (< 12 hours) and densely-packed biofilm formation of the phototrophs over the heterotrophic bacterial biofilm.

Current generation

The efficiency of photosynthetic electron transfer can also be improved via co-cultures with heterotrophic bacteria. This statement is based on the fact that electron transfer rate among heterotrophic bacteria is much more efficient than that of photosynthetic bacteria, which leads to significantly increased power generation in the absence of an electron donor [4]. Although the understanding of the individual and synergistic roles for key microbial populations is still missing, some studies have already shown increased power generation from mixed microbial communities [5-7]. The bio-solar cell utilizing the mixed culture generated light-responsive current from organic matter released by the photosynthetic bacteria. Without external introduction of organic substrates, the bio-solar cell with co-cultures generated self-sustained current density (6µA/cm²) (Fig 6a), which was more than 400 times greater than that of the bio-solar cell using only photosynthetic bacteria (15nA/cm²) (Fig. 6b). During operation at 12h/12h light/dark cycles, the device with the co-cultures shows the negative light response (Fig 6b) which is consistent with the mechanism where photosynthetic bacteria produces organic fuels that feed heterotrophic bacteria, and the respiratory electrontransfer chain of the heterotrophic bacteria is the source of electrons deposited on an anode. A current decrease during



Figure 6: Current densities measured from the (a) photosynthetic bacteria only and (b) co-cultures of Shewanella oneidensis MR-1 and Synechocystis sp. PCC 6803. The white bars indicate the illuminated period and the shadow indicates the dark period.

illumination was presumably due to the negative impact of photosynthetically evolved oxygen. While the oxygen abiotically reacted with the anode decreasing the open circuit voltage of the device, it could also compete with electron transfers of the heterotrophic bacteria and, therefore, decrease the current generation. Approximately a four-fold higher current was generated during the dark than during the illumination.

On the other hand, a positive light response was observed from the bio-solar cell loaded only with photosynthetic bacteria; approximately a three-fold increase was generated during the 12h of illumination than during the dark phases. This positive light response indicates that the photosynthetic electron transfer chain is the source of the electrons harvested on the anode. During the dark periods, bacterial respiration produced energy from the organic substrates (which is the product of photosynthesis) to keep the current generation above zero.

CONCLUSION

In this work, we created a microfluidic bio-solar cell which continuously generated an entirely self-sustainable current by using syntrophic interaction between heterotrophic biocatalysts and photosynthetic bacteria. We could generate much higher current density (6μ A/cm²) than that of the bio-solar cell using only photosynthetic bacteria. Moreover, by using an innovative device architecture in a miniaturized chamber, the bacterial adhesion was improved and the internal device resistance was reduced, leading to higher current/power generation. This work could result in barrier-transcending advancements in miniature bio-solar cells that could facilitate higher performance with self-sustainability, releasing bio-solar cell technology from its restriction to research settings, and translating it to practical, real-world applications.

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A PVDF-BASED FLEXIBLE AND SHAPEABLE ACOUSTIC POWER SOURCE FOR IMPLANTABLE BIOMEDICAL DEVICES

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ABSTRACT

Piezoelectric materials have been increasingly investigated over the past decade for energy harvesting and remote acoustic/ultrasonic/mechanical powering. In particular, wireless powering of implantable devices with sound and/or mechanical waves provides certain advantages compared to the traditional inductive method, mainly increased range, omni-directionality, and increased efficiency at mm and sub-mm scales. In this paper, we present a low-cost method for fabricating polyvinylidene fluoride (PVDF) power sources in form factors that are easily insertable with a biopsy needle and can form a desired shape once inside the body via a shape-memory alloy (SMA) wire. *In-vitro* experiments using receivers with 1×2 cm² in dimensions driven by an acoustic transmitter with an output intensity of 92 dB at 250 Hz results in an electrical output power of 9.5 nW.

INTRODUCTION

The recent proliferation of low-power electronics has been one of the main driving forces behind the renewed interest in energy scavenging as a mode of powering for implantable medical devices [1]. Batteries and inductive powering have been the mainstay of power generation for decades [2-4]. Due to their superior reliability, batteries have been the power source of choice for most low-power long-term implantable systems (e.g., cardiac pacemakers). However, batteries are still large, have non-desirable form factors, and surgical intervention is necessary when depleted and needs to be replaced. As an alternative approach, inductive powering has been the method of choice in satiations where power is not needed continuously and where surgical replacement is not easily feasible. Nevertheless, inductive powering suffers from its own shortcomings such as low misalignment tolerance and short transmission range.

Acoustic/ultrasonic powering can resolve some of the abovementioned shortcomings associated with batteries and inductive methods [5-6]. These include larger tissue penetration depth, omnidirectionality, and better efficiency at smaller receiver dimensions (mm and sub-mm) [7]. Vibration and acoustic energy, as one of the most available ambient energy sources, is also a promising format for energy harvesting. Lead zirconate titanate (PZT), due to its superior electromechanical coupling coefficient (k), is the most widely used piezoelectric material in ultrasonic/acoustic systems; yet, concerns regarding possible leaching of lead have impeded its adoption for implantable applications. Polyvinylidene fluoride (PVDF) with its biocompatibility, flexibility, better acoustic matching to the soft tissue, and wide bandwidth, have become an attractive alternative candidate (at the expense of lower k). Nevertheless, issues regarding its incompatibility with many standard processing methods, 3D shaping, and difficulty of insertion into the tissue still pose challenges.

In this work, we present a low-cost benchtop method for the fabrication of an implantable PVDF power source in a size and form factor that can be easily implanted through a simple biopsy needle. In addition, by using a shape memory alloy wire, the PVDF can be automatically reshaped into a predefined geometry, once inserted.

OPERATION PRINCIPLE

Figure 1 shows the schematic of the PVDF power source with a SMA wire backbone in the bore of a biopsy needle. The device

consists of a narrow strip of PVDF with a SMA wire (here thereafter referred to as a skewer) pierced through several micro-machined slits. The PVDF skewer can be implanted into soft tissue or body cavities (including vascular system) using a biopsy needle (Figure 1, left-side). Once the rolled PVDF is properly implanted *in-situ*, the unfolding and reshaping process start immediately after the removal of biopsy needle when the surrounding temperature reaches 37 °C. When in place, the PVDF strip is driven into vibration by an outside acoustic source. The resonant frequency of the PDVF receiver depends on its shape, dimensions, and material properties. When the vibration is considered as bending mode, due to the Young's Modulus of PVDF is 8.3 GPa, the first resonant frequency for the mm-scale dimensions is expected to lie within the range of audible sound.



Figure 1: Schematic of a PVDF strip with an insertion-wire/skewer; the device inside a biopsy needle.

FABRICATION

The fabrication process of the PVDF power source is shown in Figure 2a. It starts with patterning a 110 µm-thick PVDF sheet (gold electrodes deposited, Precision Acoustics Ltd, UK) into a narrow strip with multiple transverse slits using a tabletop CAMEO® craft cutter plotter (Silhouette America Inc, Utah, USA). This technique allows for simple and inexpensive machining of PVDF, which is notoriously difficult to process using traditional micromachining technologies. At this step, a small mass can be attached to the PVDF strip to increase the displacement. Then, a shape memory alloy wire (skewer) (Kellogg's Research Labs, USA) is stitched through the slits. SMA wire can be fixed to the PVDF using silver paint (Ted Pella Inc., USA) at room temperature to protect the piezoelectric property of PVDF. Finally, the entire device is coated with 5 um parylene-C for electrical passivation. Figure 2b shows a fabricated prototype with both a 1.5 mm diameter biodegradable 3D-printed polylactic acid (PLA) (NinjaFlex, NinjaTek, USA) and SMA skewer inside a biopsy needle (inner diameter of 2.8 mm). Figure 2c shows the PVDF with a SMA skewer before and after exposure to 37 °C, illustrating the possibility of shaping the device *in-situ*, once inserted

To increase the output power of proposed design, multiple PVDF films can be stacked with the poling directions of each layer aligned in parallel [8]. In this case, flexible isolation layers will be required to prevent the interference between PVDF layers. The

978-1-940470-02-3/HH2016/\$25©2016TRF DOI 10.31438/trf.hh2016.107 Solid-State Sensors, Actuators and Microsystems Workshop Hilton Head Island, South Carolina, June 5-9, 2016 maximum number of layers depends on the thickness and rigidity of PVDF film in order to be rolled and fitted into biopsy needle.



Figure 2: (a) Fabrication process; (b) fabricated prototype with PLA and elastic skewer in a biopsy needle; (c) temperature-shaped PVDF with SMA skewer.

RESULTS AND DISCUSSION

An *in-vitro* experiment setup was used to characterize the vibration behavior as well as the output level of PVDF strip, Figure 3(a). A function generator (33220A, Agilent, USA) was connected to a power amplifier (XTi 1000, Crown Audio, USA) to drive a speaker for the generation of a controllable acoustic wave. In order to increase the deflections, PVDF, an impactor pointing at the center of the PVDF strip was attached to the speaker. To mimic the soft tissue (acoustic impedance Z=1.5 MRayls), 1.0% agarose gel was used to house the PVDF strip. A digital multi-meter was used to read the output voltage from the PVDF. Assuming a simple strip forming a cantilever beam, the resonance frequencies can be given by

$$f_n = \frac{k_i}{2\pi} \sqrt{\frac{EI}{\rho W t L^4}}$$

where *L* is the length of the beam, ρ is the density of the material, W is the width of the beam, t is the thickness of the material, *I* is momentum of inertia and *E* is Young's modulus, and k_i is 3.52 for the 1st resonance. A frequency sweep was initially conducted to locate the resonant frequency, indicating 250 Hz for a 1 × 2 cm² PVDF strip.

Once the resonant frequency was determined, the electric output characterization was studied with different boundary conditions to simulate possible implantation situations. First, a planer PVDF in $1 \times 2 \text{ cm}^2$ dimension was submerged in a water balloon with the vibration delivered to the water. Thus, water is the acoustic coupling media for conducting the acoustic vibration and can also dampen the motion of PVDF, Figure 3(b); such configuration has been described for an acoustically powered transponder [9]. To compare with the first case, by using the same

setup as shown in Figure 3(a), the PVDF of the same dimension was also tested in a piece of 1.0% agarose gel (Sigma-Aldrich, USA) with the thickness of 5 mm between the impactor and the PVDF.



Figure 3: Schematic of in-vitro experiment setup: a function generator is connected to a power amplifier for driving the speaker and impactor. Device is placed in different situations for measuring the output: (a) inside agarose gel slab; (b) bending vibration in water.



Figure 4: Electric output characterization under different conditions: (a) Output I-V curve with different load resistances; (b) Output power curve with different load resistances.

Figure 4(a) depicts the output I-V curve from single layer PVDF ($1 \times 2 \text{ cm}^2$) when different load resistors were applied. For the output characterization, a 92 dB acoustic intensity at 250 Hz was applied at the surface of the speaker to provide sufficient acoustic pressure for powering. As shown in the plot, PVDF vibrating in the agarose gel produces a higher displacement and more charges as well. A higher output power at different loads are generated than the case in water. Water has been commonly used as coupling media for most ultrasonic therapeutic applications due to its ideal acoustic characteristics [10]; however, in our experiment setup, the reflection at the water/air interface (reflection would be 99.9% [11]) causes the wave pattern inside the spherical container to become less uniform along the PVDF's poling direction (compared with the gel), which results in a lower output power. Figure 4(b) illustrates the output power with different load resistances. The maximum output power can be detected with a load resistance of 562 k Ω for the case when the PVDF is in the agarose gel and the output power is ~ 9.5 nW. The output power decreases dramatically when the load resistance is smaller than 100 k Ω . The output power reduces by 56% when the load is 48 k Ω .



Figure 5: Electric output characterization under different conditions: (a) Output I-V curve with different load resistances; (b) Output power curve with different load resistances.

In order to increase the displacement while implanted, a mass of 0.11 g was attached to the same PVDF strip. The resonant frequency was shifted to 135Hz due to the increase of the total weight of the cantilever and the difference in driving capability is compared in Figure 5. For the measurement, the same acoustic input (92dB) was chosen at different resonant frequency. The peak output power was measured to be 10.2 nW which is 7.4% higher when mass is added. The output curve of the original PVDF is only shifted upwards. Thus, the application of a small mass can increase the output power without changing the driving behavior of PVDF. A higher output and a lower resonant frequency is expected when a heavier mass is attached. It is important to note that for the simplicity of implantation, the width of the mass cannot exceed the diameter of the biopsy needle (2 mm) and a mass in smaller size can also introduce less boundary constraint on the PVDF.

Based on the characterization, local delivery of acoustic vibration and an additional mass are preferred for the optimization of output power. The output of different PVDF piezoelectric power sources are summarized in Table 1. Without sacrificing the feasibility of easy implantation, the output can also be increased further if PVDF is larger in size and stacked to form a multi-layer structure. Output impedance matching circuitry can also be integrated with powered electronic system to amplify the output power delivered to the load.

As a proof-of-concept, the capability of using the design for energy harvesting from low-frequency human motion/impact was also validated. The output from a series of soft-impacts on PVDF was measured inside water with an interval of 1 second. The softimpact was applied periodically by a single finger tapping motion. As shown in Figure 6, once the PVDF was bended, the releasing of finger and an inverse displacement create two voltage peaks in different polarizations. The vibration of PVDF in water lasts for around 0.1 second. The maximum output power at the peak is calculated to be 1.58 nW which is 25% higher when the speaker is used to generate a continuous output. The tissue motion during the normal activity can provide a higher displacement and the electric energy generated can be stored using a rectifier and capacitors.

Table 1: Output power in different PVDF energy sources [8][12-13].

Device	Dimension (mm)	Input level	Output (nW)
PVDF cantilever in Helmholtz resonator	15×10.6×0.37	118 dB (15Pa)	190
PVDF beam in sonic crystal	50×12×0.028	7 Pa	35-40
PVDF bimorph cantilever	34.8×10×0.011	1.5 g acceleration	6.05
This work	20×10×0.11	92 dB	10.2



Figure 6. Output voltage when the PVDF is receiving a series of soft-impacts in water.

CONCLUSION

We presented an acoustically-powered implantable PVDF power source for low power implantable devices. The designed configuration as well as fabrication process make it possible for lowcost manufacturing and reshaping after implantation. The device was characterized *in vitro* using a variety of acoustic and impact forces in tissue simulating environments. The designed form factor allows the PVDF to fit in a biopsy needle. A shape memory alloy wires used in a skewer configuration provides in situ shaping once the device is inside the body at 37 °C.

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VIBRATION ENERGY HARVESTER BASED ON FLOATING MAGNET FOR GENERATING POWER FROM HUMAN MOVEMENT

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ABSTRACT

This paper reports a brand new approach of forming an electromagnetic energy harvester with a pair of pyrolytic graphite substrates to float a magnet in air for generating power from human walking motion. The novel levitation mechanism allows a low resonant frequency (1 - 5 Hz) without a heavy proof mass. A subminiature energy harvester weighing 22 gram has been demonstrated to have a resonant frequency of 3.4 Hz and generate 11.7 μ W from the back of a human walking at 2 m/s.

INTRODUCTION

Technical improvement in microelectronics and MEMS, coupled with burgeoning internet of things (IoT), has brought wearable devices closer to our daily lives than ever before. These devices have changed our lifestyle in many ways. For example, a wearable activity tracker monitors and tracks fitness-related metrics (such as distance walked or run, calorie consumption, heartbeat rate, and quality of sleep), and leads us to change our lifestyle for better health. However, users of wearable or handheld devices are often inconvenienced by the need to recharge battery, and would not mind carrying (or paying for) an energy harvester to supplement or even replace battery, as long as it is small, light and affordable. Compared to a solar cell which has been widely used in consumer electronics like calculator, a vibration energy harvester that can generate electrical power from vibration or motion is better for wearable application since it can work day and night independent of availability of light energy.

Vibration-energy harvesters usually convert mechanical energy into electrical energy through capacitive [1], piezoelectric [2] or electromagnetic [3-4] transduction. Capacitive (or electrostatic) energy harvesters can easily be manufactured with a fabrication process that is compatible with CMOS integrated circuit (IC) microfabrication. However, it needs a DC bias voltage or pre-charged membrane which quickly loses its charge when ambient temperature becomes higher than 80 °C. Piezoelectric energy harvester based on PZT [5] or AIN can achieve high open circuit voltage but suffers from low current associated with large resistance of the piezoelectric material itself. Compared to these two mechanisms, electromagnetic energy harvesting based on Faraday's law offers the advantage of high current, even for a low impedance load, and is ideally suited for wearable devices [6-8].

A major obstacle of generating power from human movement such as walking is the inherent low frequency (1 - 5 Hz) associated with such motion [9]. A magnetic spring [10] based on the repelling force between the same pole is one way to suspend the proof mass (usually a magnet or magnet array) to obtain a low resonant frequency, but requires heavy magnets for a resonant frequency as low as 1 - 5 Hz. Thus, we have invented a novel suspension technique based on diamagnetic material that can induce a magnetic field which is directed opposite to an externally applied magnetic field and repels the applied magnetic field.

DESIGN

Electromagnetic vibration energy harvester uses the relative movement between magnet and coil to produce electromotive force

(EMV). The magnet also acts as a proof mass in most cases. In order to reduce the friction which is the main source of mechanical damping that reduces the transduction effectiveness, the ideal design would be to completely float the magnet in the air.

A magnetic spring based on the repulsive force between two magnets of same polarity can be used to levitate a magnet. However, according to the Earnshaw's theorem [11], for hard magnets of which the magnetic strength is independent of external fields, a stable stationary equilibrium cannot be maintained solely by the repulsive magnetostatic force. The theory was first proposed for electrostatic fields, but since it applies to any classical force of inverse-square law, it also applies to the magnetic spring. Thus, the Earnshaw's theorem forbids touch-free magnetic levitation by a magnetic spring. Braunbeck further extended Earnshaw's theorem to state that even if the materials are not hard, it is impossible to achieve static magnetic levitation or suspension with magnets of relative permeability greater than one (i.e., paramagnets) [12].

On the other hand, a diamagnetic material creates an induced magnetic field (within the material) in a direction opposite to that of an externally applied magnetic field, and thus is repelled by the applied magnetic field. Superconductors can expel all magnetic field and therefore can be considered as perfect diamagnetic material. But superconductors work below a critical temperature that is far lower than room temperature, and are impractical for energy harvesters. There are plenty of materials that are diamagnetic at room temperature although the effect is usually weak. The most common diamagnetic material is water, and if an external applied magnetic field is large enough, one can levitate a living creature like frog in air.

Pyrolytic graphite is a very strong diamagnetic material, and a thin slice of pyrolytic graphite can be stably levitated above the magnetic field provided by a permanent magnet. However, a magnet cannot be levitated by a pyrolytic graphite sheet alone, no matter how thick the graphite sheet is, due to the heavy weight of a magnet. And a stronger magnetic field (for stronger repelling force) requires a heavier magnet. This means that the gravity of a magnet needs to be reduced partially or completely balanced by other mechanism. We have used another magnet to counteract most of the gravity through an attractive force between the two magnets, while using two graphite sheets (above and below a proof-mass magnet) to stably levitate or float the proof-mass magnet in air.

In the vibration-energy harvester illustrated in Fig. 1, we use a pair of pyrolytic graphite substrates to provide the repelling forces so that a magnet may be floated and confined in air between the two diamagnetic substrates. The floating magnet moves, without contacting the graphite substrates, in response to an applied vibration, and comes back to its static equilibrium position automatically when there is no applied vibration. The relative motion between the floating magnet and the nearby coil, as the magnet moves, induces electromotive force. Since the repelling force from the bottom graphite is not strong enough to overcome the gravity of the floating magnet, an additional magnet (denoted as upper magnet in Fig. 1) has been used.

The upper magnet is chosen to provide enough attractive force to balance the gravity of the floating magnet. Though the attractive

978-1-940470-02-3/HH2016/\$25©2016TRF DOI 10.31438/trf.hh2016.108 Solid-State Sensors, Actuators and Microsystems Workshop Hilton Head Island, South Carolina, June 5-9, 2016 force may be large enough to make the floating magnet touch the top graphite, the floating magnet does not come in contact with the top graphite, because of the repelling force provided by the top graphite. As the floating magnet gets closer to the bottom graphite, the attractive force from the upper magnet is reduced, while the repelling force from the bottom graphite is increased, thus the floating magnet does not come in contact with the bottom graphite, either. Instead it stays stably between the top and bottom graphite sheets as a static equilibrium.



Figure 1: Schematic of the vibration energy harvester based on floating magnet through diamagnetic levitation. Though much of the gravity of the floating magnet is counteracted by an attractive force provided by upper magnet, the floating magnet floats in air, well balanced (without being completely pulled up to the upper magnet), because of the repelling force from the pair of pyrolytic graphite sheets surrounding the floating magnet. Relative movement between the floating magnet and coil due to in-plane vibration induces electromotive force.

With the assumption that the distance between the upper and floating magnets is much larger than the magnet thickness, the two magnets can be simplified as dipoles. When the floating magnet is at the equilibrium position as shown in Fig. 2(a), the attractive force between the upper magnet and the floating magnet should be:

$$\frac{\mu Q_T Q_B}{4\pi h^2} = mg \tag{1}$$

where μ is air permeability; Q is magnetic field intensity; h is the distance between the upper and floating magnet; m is the mass of the floating magnet; and g is gravitational acceleration.

When the floating magnet deviates from its balanced position as shown in Fig. 2(b), the forces in the vertical direction counter-balance each other, since the repelling forces provided by the graphite sheets self-adjust according to the magnetic field from the floating magnet, and there is a net force in the lateral direction equal to

$$F = \sin\theta \frac{\mu Q_T Q_B}{4\pi (h/\cos\theta)^2}$$
(2)

where θ is the angle between the line connecting the center of two magnets and the vertical direction. In fact, the force in the lateral direction is the horizontal component of attractive force between the upper and floating magnets. If we take equation (1) into equation

(2), we can derive the vertical force as a function of the deviated angle θ as follows.

$$F = mg\sin\theta\cos^2\theta \tag{3}$$

If the deviated distance x is much smaller than the distance between the upper and floating magnets (h), we have the following approximations:

$$\sin \theta \approx \frac{x}{h}, \cos \theta \approx 1, \text{ if } x \ll h$$

Then the lateral force is linearly proportional to x as expressed in the following equation (4):

$$F \approx -\frac{x}{h}mg \tag{4}$$

And the equation of motion can be expressed as $m\ddot{x} = F$

$$m\ddot{x} + \frac{x}{h}mg = 0 \tag{5}$$

Equation (5) is exactly the same as the one for a simple harmonic vibration with an equivalent spring constant of mg/h. Thus, as long as the deviated distance x is much smaller than h, the floating magnet vibrates in simple harmonic motion with the resonant frequency equal to

$$\omega = \sqrt{\frac{g}{h}} \tag{6}$$

Consequently, the resonant frequency of the energy harvester is inversely proportional to the square root of the distance between the upper and floating magnets (the larger the distance, the lower the resonant frequency). In this way, a very low resonant frequency (e.g., 1 - 5 Hz) can be achieved with relatively small size for scavenging energy from human's walking motion.



Figure 2: (a) The floating magnet is at balanced position. (b) The floating magnet deviates from its balanced position due to applied vibration. For small deviation, the restoring force in the lateral direction is linearly proportional to the deviation.

FABRICATION

Neodymium (NdFeB) permanent magnets are used since they provide high magnetic flux density. Two disk NdFeB magnets of the same size are used for the upper and floating magnets. Two pyrolytic 5cm×5cm×3mm graphite sheets of and 3.2cm×1.6cm×0.75mm are located below and above the floating magnet, respectively. The upper pyrolytic graphite is chosen to be thinner to minimize the attenuation of the magnet flux, as it is between the coil and the floating magnet. The coil is fabricated by a winding machine, and has 22 Ω resistance and 400 turns. The frame of the energy harvester is made of laser-machined acrylic slab, and the distance between the upper and floating magnets is adjusted to be 2.9 cm to make the attractive force balance out the gravity of floating magnet. The dimensions and parameters of the energy

harvester are listed in Table 1. The photos of the fabricated energy shown in Fig. 3 clearly show that the lower magnet is completely floating in air without touching the pyrolytic graphite because of the repelling force provided by the pair of the graphite sheets while the gravity is counteracted by the attractive force from the upper magnet.

(1)	Table 1. Dimensions/	parameters o	of the energy harveste	er.
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Magnet diameter	19 mm	Coil turns	400
Magnet thickness	1.58 mm	Coil resistance	22 Ω
Surface field	1,217 Gauss	Effective weight*	21.7 g

*Without the acrylic frame



Figure 3: Photos of the fabricated vibration energy harvester. The lower magnet is completely floating in air with the repelling force provided by a pair of pyrolytic graphite substrates. The floating magnet vibrates in the lateral direction without any direct contact with the graphite.

RESULTS AND DISCUSSION

The energy harvester is tested on a linear actuator (Aerotech ACT115DL) with in-plane vibration over 1 - 5 Hz. The schematic of the testing platform is shown in Fig. 4. The linear actuator is driven by a digital controller which receives the control parameters (i.e., the vibration amplitude and frequency) from a computer. The position data of the linear actuator are fed to the computer for accurate control of the vibration. The output signal from the energy harvester is observed by and stored into an oscilloscope.



Figure 4: Schematic of the testing platform. The linear actuator is controlled by a computer and calibrated with a feedback signal. The output voltage of the energy harvester is measured with an oscilloscope.

The measured output voltage as a function of frequency under various accelerations is shown in Fig. 5. Under a fixed acceleration, the voltage varies as the vibration frequency is varied, and peaks at a resonant frequency. The measured resonant frequency is around 3.4 Hz, while the theoretical prediction is 2.93 Hz (for 2.9 cm distance between the upper and floating magnets).

The root mean square (rms) output voltage and the corresponding power with a matched load are shown in Fig. 6. The power delivered to a matched load increases as the applied acceleration increases, and 68 μ W can be delivered into a load of 22 Ω from 2 g acceleration (corresponding to 43 mm vibrational amplitude) at 3.4 Hz.

The energy harvester is also tested on the back of a human walking on a treadmill as shown in Fig. 7, and 11.7 μ W is delivered to a 22 Ω load from 2 m/s walking speed. The power decreases a little beyond 2 m/s, due to (1) more of the vibration energy being at higher frequencies than the harvester's resonant frequency (3.4 Hz) and (2) more energy in the vertical direction instead of the lateral direction, as the walking becomes jogging or running.



Figure 5: Measured frequency response of the energy harvester under various applied accelerations. The resonant frequency is around 3.4 Hz, while the theoretically calculated one is 2.9 Hz.



Figure 6: Measured power and rms voltage vs input acceleration at the resonant frequency of 3.4 Hz. A 2 g acceleration produces 68 μW power (into 22 Ω load).



Figure 7: (Top) Photo of the harvester on the back of a human walking on a treadmill. (Bottom) Measured power from the harvester vs the walking speed of the one carrying the harvester on the back: $11.7 \mu W$ is delivered to a 22 Ω load from 2 m/s walking speed.

CONCLUSION

The novel idea of a floating magnet based on diamagnetic graphite with an additional magnet to counteract the gravity of the floating magnet has been implemented for electromagnetically generating electrical power from human's walking motion. A prototype energy harvester weighting 21.7 gram produces an rms voltage of 77.4 mV (in open circuit) and 68 μ W power output (into 22 Ω load) at the harvester's resonant frequency of 3.4 Hz in response to a vibration amplitude of 43 mm (i.e., 2 g at 3.4 Hz). The low resonant frequency makes the energy harvester suitable for harvesting energy from human walking motion. And 11.7 μ W is delivered to a 22 Ω load from 2 m/s walking speed from the harvester mounted on a human's back.

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130 SECOND RING-DOWN TIME AND 3.98 MILLION QUALITY FACTOR IN 10 KHZ FUSED SILICA MICRO BIRDBATH SHELL RESONATOR

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ABSTRACT

We achieved a 130.0 second ring-down time constant for the 9.736 kHz n = 2 wine glass mode of a birdbath shell microresonator, corresponding to a quality factor of 3.98 million. The resonator is a fused silica shell with an outer radius of 2.5 mm, anchor radius of 0.5 mm, height of ~2.1 mm, and thickness ranging from ~15–70 µm. This is the longest reported ring-down time and quality factor to date for any micro resonator at this frequency. We also measured an extremely low 138 mHz frequency split between wine glass modes at 10.389 kHz with a low damping mismatch of 7.1×10^{-5} Hz. This unprecedented result is a considerable achievement for microresonators and will pave the way to the development of next generation navigation-grade MEMS gyroscopes with 10e–3°/hr bias stability.

INTRODUCTION

Great advancement in the performance of MEMS gyroscopes has been achieved over the past two decades, yet they are still not suitable for dead-reckoning of drones, self-driving cars, and pedestrians, which require very high bias stability (<0.1°/h). The vibratory decay time constant (τ) is an important parameter that directly affects bias stability (bias stability $\propto \tau^{-1}$ [1]). Unfortunately, τ of silicon gyroscopes is typically limited to <5 s due to large thermoelastic damping (TED) at frequencies greater than 10 kHz. A quad mass silicon gyroscope with $\tau = 181$ s (Q =1.21 million) at 2.1 kHz has been reported [2]; however, it has potentially high vibration sensitivity since its frequency is close to that of external noise sources.

Over the past few years, our group has demonstrated a fused silica micro birdbath (BB) resonator gyroscope [3], [4]. The BB resonator is a significant step toward achieving a low-cost micro-scale navigation-grade MEMS gyroscope. Compared to other existing micro shell resonators such as [5] or [6], the BB resonator has several key advantages for achieving both very long τ and small Δf .

First, the anchor and the rest of the resonator are self-aligned. That is, the shell is defined from a single reflow-molding step using a radially symmetric mold. Misalignment between the anchor and rim is known to be a key source of anchor loss; the BB resonator achieves low anchor loss by eliminating this misalignment.

Second, the BB resonator has a long, smooth transition region that connects the rim to the anchor. This long transition region isolates the part with the most mechanical energy (rim) and the energy sink (anchor). The smooth curvature reduces the concentration of mechanical stress near the anchor. Both of these features effectively trap the mechanical energy in the rim, greatly reducing energy leakage to the anchor.

Third, the micro blowtorching process can form BB resonators out of fused silica (FS)—which has one of the lowest amounts of thermoelastic damping of any material—and results in an ultra smooth surface quality. Other researchers have demonstrated a structure similar to the birdbath resonator that achieved quality factor (Q) of 1.05 million [7], but due to its high 105 kHz frequency it only produced τ of 3.18 s. A slightly longer 4.32 s τ was reported in [6] for a cylindrical microcrystalline

diamond shell resonator, with a frequency of 23.07 kHz and Q of 313,100. A higher frequency 160 kHz hemispherical resonator gyroscope with a 9.0 s τ (Q = 4.5 million) was reported in [8].

We initially reported the micro fused silica (FS) birdbath (BB) resonator (Figure 1) and accompanying micro-blowtorch molding process (Figure 3) to form BB shells with thermal reflow-molding at ~1600 °C [9]. In [10] we first reported demonstration of the BB resonator as a gyroscope. The resonator operates in the n = 2 wine glass (WG) modes; in rate-mode, the gyroscope is operated in force-to-rebalance mode, while in rate-integrating mode the vibrations are allowed to freely precess around the rim, their orientation indicating the rotated angle. We have designed the BB resonator geometry to place the desired n = 2 wine glass modes around 10 kHz to avoid environmental noise, and as far as possible from parasitic modes to prevent interference. More detail on the BB resonator gyroscope can be found in [4].



Figure 1: Fused silica birdbath resonator resting on a silicon base. We have measured τ of 130.0 s and Q of 3.98 million at 9.736 kHz for an n = 2 wine glass mode of this type of resonator.



Figure 2: Cross sectional profile of a birdbath resonator showing relevant dimensions and approximate thickness variation due to reflow molding, as well as its high surface-to-volume (S/V) ratio.

Recently, we made significant progress in perfecting the fabrication process and achieved a record τ of 130.0 s and Q of 3.98 million at 9.736 kHz, a huge improvement from 8.3 s at ~8.2 kHz [9]. Based on the analysis on bias stability and τ in [1], we estimate our reported τ will enable an ultra-high bias stability <10e-3°/h in future gyroscopes. In this paper we describe several

978-1-940470-02-3/HH2016/\$25©2016TRF DOI 10.31438/trf.hh2016.109 Solid-State Sensors, Actuators and Microsystems Workshop Hilton Head Island, South Carolina, June 5-9, 2016 key improvements in the BB resonator fabrication process that allowed us to achieve this level of performance.

FABRICATION

An overview of the fabrication process is provided here, but for a detailed discussion on the principles of fused silica birdbath resonator fabrication, refer to [11].

Molding the Shell

Individual birdbath shells are molded by thermal reflow from ~100- μ m-thick 1.5 × 1.5 cm² fused silica substrates. The substrates are placed atop a graphite mold with a 2.5-mm-radius cylinder machined out from its middle and a 0.5-mm-radius post left standing in the center of the cylinder. To rapidly reach the high softening temperature of fused silica, ~1600 °C, a propane-oxygen blowtorch is lowered from above by a motorized Z-stage. With minimal contact-only at the anchor post and outer wall-the softened fused silica substrates reflow into the desired shape. The final BB resonator shape is therefore not explicitly defined by the mold; the outer mold wall determines the major radius and the central post defines the anchor, but the curvature and overall shape are determined by pressure and temperature during the <10 s molding process. This process is depicted in Figure 3. We have performed extensive finite element modeling to improve our understanding of the impact of each process condition on the final resonator shape [12], [13].



Figure 3: Thermal reflow of a ~100- μ m-thick fused silica substrate into a graphite mold to form a birdbath resonator. The pressure inside the mold cavity (P₂) is lower than the outside pressure (P₁) [11]. The reflow process takes <10 s.

Shell Separation and Polishing

The reflowed substrate now consists of a square frame with a molded birdbath shell protruding from its center. The square frame must be removed to isolate the shell resonator. This is accomplished by lapping and chemical mechanical polishing (CMP). A thick silicon lapping jig wafer with an array of through holes protects the shells while their frames are removed. The shells sit below the surface of the jig wafer and are encapsulated in thermoplastic polymer for protection. The square frame sits on the surface, leveling the shells to the wafer. As the wafer is lapped, the frame is removed. A CMP step completes the fabrication, polishing the rims of the shells. This process is depicted in Figure 4. The shells are then released and cleaned in a solvent bath. A photograph of a completed resonator is shown in Figure 1 and relevant dimensions are given in Figure 2.

Metallization

If the birdbath shell is to be used as a gyroscope, a conductive

film is applied to its surface to enable electrostatic drive and readout of its rim motion. This is typically accomplished by sputtering a gold film over a chromium or titanium adhesion layer.



Figure 4: After molding, birdbath shells are protected with thermoplastic polymer within a jig wafer so their square frames can be removed by lapping and their rims polished with CMP.

TESTING

To prepare a birdbath resonator for testing, its anchor is attached with glass frit to a raised pedestal on a silicon substrate. The silicon substrate is then temporarily attached to a piezoelectric actuator with a thermoplastic polymer and placed into a vacuum chamber that is evacuated to <10 μ Torr (Figure 5). The actuator drives the resonator with sinusoidal vibrations at its resonant frequency along its axis of radial symmetry; the vibrations couple into the desired n = 2 wine glass modes. Each of the two modes, in turn, are excited into resonance, then the drive signal is cut and the resonator is allowed to ring down. The motion of the rim is measured with a Polytec laser Doppler velocimeter (LDV) through a window in the vacuum chamber, as depicted in Figure 5. This technique works well for metallized or uncoated fused silica resonators.



Figure 5: A PZT transducer drives the shell into resonance before it freely rings down. A laser Doppler velocimeter measures motion of BB shell rim through a window in the vacuum chamber.

RESULTS AND DISCUSSION

Our birdbath resonators, whose natural n = 2 wine glass mode frequencies are around 10 kHz, routinely have quality factors over one million and frequency splits between the two modes in the low tens of Hz. This outstanding performance is unprecedented for shell-microresonators, and even more impressive considering their rapid and relatively simple fabrication process. For an uncoated BB resonator, we achieved an extremely long τ of 130.0 s at $f_{1,n=2}$ = 9.736 kHz (Q = 3.98 million) for one mode (Figure 6) and 115.1 s at $f_{2,n=2} = 9.776$ kHz (Q = 3.54 million) for the second mode. Implemented as a gyroscope this resonator should achieve an outstanding bias stability of <10e-3°/h, according to Challoner et al.'s analysis [1]. This marks a considerable improvement over our previous works, which most recently reported a 43 second τ at 8.8 kHz for a birdbath resonator.

The frequency split between wine glass modes, Δf , is also
important for gyroscope performance. While it is possible to cancel the frequency split by electrostatically tuning stiffness, it is preferable to address this during fabrication. We measured an extremely low as-fabricated Δf of 138 mHz for a BB resonator at 10.389 kHz. This device has relatively long ring-down times of τ_I = 76.4 s (Q_I = 2.49 million) and τ_2 = 76.0 s (Q_2 = 2.48 million) and a low damping mismatch ($\Delta \tau^{-1}$) of 7.1×10⁻⁵ Hz. This low Δf and $\Delta \tau^{-1}$ indicate a high degree of radial symmetry that should minimize anchor loss. The fact that our longest τ comes from a device with a 40 Hz Δf indicates that anchor loss may not be the dominant mechanism of energy loss for the BB resonator. In fact, we have not seen a strong correlation between Δf and τ , leading us to conclude that BB resonators are somewhat tolerant to molding variations.



Figure 6: Ring-down plot of an uncoated birdbath resonator with our longest recorded $\tau = 130.0$ s. The other mode at 9.776 kHz has $\tau = 115.1$ s (Q = 3.54 million). Results measured with LDV.

Radial Symmetry

We attribute our experimental results to improvements in our fabrication process. Maintaining a high degree of radial symmetry is essential for maximizing Q_{anchor} of the BB resonator. It is designed so that the anchor point is a vibrational node, but any deviation from symmetry causes the nodal point to shift. In some cases we have seen a warped upper anchor region. This is the area that stretches the most and becomes the thinnest during molding, so it can be challenging to maintain a smooth transition from anchor to shell. This must be controlled, because if the radial symmetry is disrupted, the resonator will likely suffer increased anchor loss and damping mismatch between WG modes. Warping and imbalance also result in a frequency split between wine glass modes, which is problematic for gyroscope operation. The thermal distribution within the mold must be uniform, which requires a flame that will uniformly heat the mold as well as ensuring that the flame and mold are aligned.

Rim Polishing

After lapping away the square frame of fused silica from which a birdbath resonator is molded, the rim of the shell is quite rough. We have experimentally determined that roughness, scratches, and chips may limit performance and that the rim must be polished using CMP in order to reach a high Q. A high quality, defect-free rim is achieved by fine-tuning the pressures, rates, and times of the lapping and CMP processes, and by selection of an appropriate thermoplastic to protect the shells.

Surface Quality

We have observed experimentally that devices with more

microscopic defects such as etch pits tend to have relatively low Q. There are several papers showing that the Qs of fused silica resonators are inversely proportional to their surface-to-volume (S/V) ratios due to energy loss mechanisms pertaining to the surface [14]–[17]. The BB resonator may be especially sensitive to the condition of its surface considering its high S/V ratio of ~33 mm⁻¹. Fortunately, the micro blowtorch process smoothes out the surface during reflow to an average roughness of 1.8 Å (Figure 7).



Figure 7: This AFM scan shows the smooth fused silica surface produced by thermal reflow molding with a blowtorch [18].

Anchor Mounting

The birdbath resonator is designed with an automatically centered anchor post that helps to minimize anchor loss. As mentioned earlier, the center of the anchor is a nodal vibration point, which helps reduce sensitivity to the anchor mounting condition. Still, a rigid anchor is required to minimize motion at the attachment point, which would lead to anchor loss. We have found that glass frit works well for this purpose.

Conductive Coating

Our 130.0 s τ was measured for a bare resonator with LDV, but to implement a BB resonator as a gyroscope we must drive and read out its motion electrostatically. This requires a conductive surface, typically sputtered gold with a chromium adhesion layer. Figure 8 shows the effect of sputtering Cr/Au on the top surface of each of two previously uncoated BB resonators. The initial deposition of 15/20 Å Cr/Au considerably decreases τ , but consecutive 40 Å Au depositions have a decreasingly strong effect. This suggests that the nature of the initial film is most important, rather than the overall thickness. A more complete discussion with additional details on this study can be found in [18].



Figure 8: Coating 15/20 Å Cr/Au on two previously uncoated BB resonators considerably decreases their τ . Adding additional 40 Å

layers of Au further decreases τ , but less severely [18].

Effect of Sputtered Gold on BB Ring-Down Time Constant

Based on this analysis, we are therefore developing techniques to maximize τ for metallized microresonators, including optimizing metal coverage and thickness and experimenting with other materials and deposition techniques. One attractive option is platinum atomic layer deposition (ALD). ALD enables conformal deposition of highly uniform thin films over corners and high aspect ratio features [19]. This should enable us to use a thinner film than would be required with sputtering to produce sufficient conductivity, thus maintaining the highest possible τ for a packaged gyroscope. We estimate from finite element modeling (FEM) that we are not limited by anchor loss or TED, and that resonators with optimized processing—even with a metal coating—will reach $Q \approx 6.8$ million ($\tau \approx 215$ s).

CONCLUSION

The results presented in this paper represent a major step forward in the development of high-performance MEMS gyroscopes. We have demonstrated the ability to precisely fabricate fused silica micro-shells and control their form to the extent necessary to produce a very high 3.98 million Q and extremely long 130 s τ near our target frequency of 10 kHz. We predict that a gyroscope with this performance will reach 10e– 3°/hr bias stability. We achieved these results by optimizing our fabrication process to improve radial symmetry, rim polishing, surface quality, and anchor attachment.

Although birdbath resonators are molded individually, the blowtorch molding process takes less than 10 s and we are able to rapidly produce devices. Resonant frequency and frequency split between wine glass modes are reproducible, especially for devices from the same mold. Furthermore, adjusting parameters such as shell depth can be easily performed during a fabrication run in order to fine-tune the shape and operational characteristics of the shells. This flexible yet controllable technique has enabled us to produce very high-performance resonators with fused silica that would not be possible with conventional micromachining.

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920 MHz ALUMINUM NITRIDE CROSS-SECTIONAL LAMÉ MODE PIEZOELECTRIC MEMS TRANSFORMER WITH HIGH OPEN-CIRCUIT VOLTAGE GAIN IN EXCESS OF 39

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ABSTRACT

In this work we present the first piezoelectric transformer based on a 920 MHz 2-port CLMR. This device, which is dubbed *crosssectional Lamé-mode transformer* (CLMT), shows a loaded quality factor of ~ 1000 and a record high k_r^2 for AlN 2-port resonators in excess of 3.9%, thus leading to a G_v value, in air, close to 39. Such record high value of G_v, which matches closely the values predicted by both Finite-Element-Methods (FEM) and circuit modelling, is attained through the use of two optimized 3-finger interdigital metal electrodes (IDTs) sandwiching a 4-µm thick AlN film. The demonstration of such a high voltage-gain in an AlN piezoelectric transformer enables the development of new strategies to achieve energy awareness and deeper degrees of miniaturization in next generation integrated RF circuits.

INTRODUCTION

The growing demand of service bands in mobile wireless platforms is strengthening the need of adaptive radio-frequency (RF) front-ends. In particular, the use of reconfigurable antennas and filtering banks is key to reduce the size of front-ends without compromising their sensitivity and, consequently, degrading the quality of their baseband operations. However, in order to enable this approach, the development of reconfigurable matching networks is highly desirable to maximize the power-transfer, from filters to antennas and vice versa, for any possible operative scenario.

The use of piezoelectric transformers (PTs) has been proposed as a compact approach to achieve power-matching without the use of lumped low-Q integrated inductors [1],[2].

PTs are 2-port devices enabling maximum power-transfer between two significantly different electrical terminations. The difference between such impedances is set by their voltage-gain (G_v). In addition, integrated PTs reaching high G_v -values are highly desirable to achieve miniaturized and high conversion efficiency AC-to-DC [3],[4] and DC-to-DC [5],[6],[7] converters.

Aluminum-Nitride (AlN) piezoelectric resonators are promising candidates to replace off-chip Surface-Acoustic-Wave (SAW) devices in commercial wireless platforms. In particular, since such devices can be built through traditional semiconductor processes, they can be monolithically integrated with the rest of the CMOScircuitry. This crucial feature enables fully-integrated RF frontends. Two main AlN-resonator technologies were previously demonstrated: contour-mode resonators (CMRs) [8] and film-bulkacoustic-resonators (FBARs) [9].

AlN CMRs rely on the transduction of longitudinal vibration along the width of an AlN plate through the use of the e_{31} piezoelectric coefficient of AlN. In contrast, AlN FBARs rely on the transduction of longitudinal vibration along the thickness of an AlN plate through the e_{33} piezoelectric coefficient of AlN. As the e_{33} is significantly larger than the e_{31} , AlN FBARs show higher electromechanical coupling coefficient (k_t^2) than CMRs, thus being preferred for the synthesis of wide-band filters for RF applications. In contrast, as the development of 2-port FBAR-based devices is only possible through a significantly more elaborate fabrication process [7], CMRs are preferred to FBARs when devices with two separate terminations are needed. Consequently, previously reported piezoelectric PTs are mostly based on CMRs. However, as the voltage-gain of PTs is proportional to their k_t^2 , PTs based on AlN CMRs can only achieve limited G_v-values.

More recently, AlN cross-sectional Lamé-mode resonators were demonstrated (CLMRs) [10],[11]. CLMRs are piezoelectric resonators formed by two metallic IDTs sandwiching an AlN plate. They rely on the transduction of a Lamé-mode in the cross-section of an AlN plate through the combined use of both the e_{31} and the e_{33} piezoelectric coefficients of AlN. Differently from other existing piezoelectric resonator technologies, CLMRs ate characterized by high- k_1^2 and lithographically determined resonance frequency (f_{res}). Therefore, they are suitable for the synthesis of lithographically defined integrated contiguous and not-contiguous pre-select filters for platforms adopting carrier-aggregation (CA).

In this work we report the performance of the first PT-based on the CLMR-technology. This device, which we labeled as *crosssectional Lamé-mode transformer* (CLMT), operates in the RFfrequency range (\sim 920 MHz) and shows maximum G_v-value in excess of 39. Such a record-high value for AlN PTs is enabled by the large input- k_r^2 (\sim 4%) achieved by 2-port AlN CLMRs.

CROSS-SECTIONAL LAMÉ-MODE RESONATORS

CLMTs exploit the unique piezoelectric and electromechanical properties of the recently developed cross-sectional Lamé-mode resonators (CLMRs).

AlN CLMRs use the coherent combination of the e_{31} and e_{33} piezoelectric coefficients of AlN to transduce a Lamé-mode in the cross-section of an AlN plate (Fig. 1). Thanks to the opposite sign of these two piezoelectric coefficients, in-phase charge components are generated by vibration along both the cross-sectional directions (thickness and width) of the plate. As a consequence, the k_r^2 attained by AlN CLMRs is function of both the e_{31} and e_{33} piezoelectric coefficients of AlN and it is higher than the k_r^2 achieved by conventional laterally vibrating AlN resonators such as CMRs.



Figure 1: schematic-view of a 3-finger CLMR. The device is formed by two IDTs sandwiching an AlN film. The pitch of the IDTs (W) is selected to be similar to the thickness of the AlN layer (T_{AlN}). Such choice enables the excitation of high- kt^2 degenerate or nondegenerate Lamé-modes in plates. The mode-shape relative to the total displacement of the same device, when exciting a nondegenerate Lamé mode, is also reported.

Similarly to CMRs, CLMRs can be excited through either a Lateral-Field-Excitation (LFE) [12] or a thickness-field-excitation (TFE) approach [6]. LFE CLMRs are formed by one set of IDT patterned on either the top or the bottom surface of an AlN layer. In contrast, TFE CLMRs are formed by two interdigital-metal electrodes sandwiching an AlN film. The IDTs, in both TFE and LFE CLMRs, are needed to produce the excitation electric field in the ross-section of the AlN layer.

As demonstrated in [5], CLMRs achieve maximum k_t^2 -value when the pitch of the IDTs (W) is set to a specific value (W^{opt}) similar to thickness (T_{AIN}) of the AlN plate. In fact, in this scenario, a nondegenerate Lamé-mode is excited in the cross-section of the AlN plate. However, due to the capability of exciting high- k_t^2 *degenerate* cross-sectional Lamé modes [14] in plates, CLMRs can attain high k_t^2 also when W is slightly different from W^{opt}. In addition, since f_{res} depends on W, the transduction of such degenerate modes also enables a significant lithographic tunability of the device operating frequency. This feature is crucial for the implementation of multi-frequency resonators filters and PTs monolithically integrated on the same chip with minimal fabrication complexity.

CROSS-SECTIONAL LAMÉ-MODE PTs

The high transduction coefficient of AlN CLMRs enables the implementation of 2-port devices with k_i^2 values much higher than the ones of conventional 2-port AlN CMRs [15]. This special feature is key to achieve high performance *Cross-Sectional Lamé-Mode transformers* (CLMTs). Similarly to CLMRs, CLMTs are formed by two-IDTs sandwiching an AlN-plate. However, differently from CLMRs, only a portion of the IDTs, labeled as *input*, is connected to the device input-voltage (V_{in}). Such portion is responsible for the excitation of the mechanical vibration in the AlN plate, whereas the remaining portion, labeled as *output*, is adopted to probe the piezoelectrically generated device output-voltage (V_{out}).

A schematic representation of the 920 MHz CLMT of this work is shown in Figure 2.



Figure 2: Left) Schematic representations of the CLMT demonstrated in this work. In this device the input- and output-ports use a common ground.

The most important parameter of any piezoelectric transformer is the voltage gain (G_v), which is defined as the ratio between the voltage levels at the output and input ports respectively. In particular, to compare the performance of different PT-technologies, the open-circuit *voltage-gain* (G_v ^(max)) is often used. This parameter expresses the magnitude of the ratio between V_{out} and V_{in}, when the output port is kept open. The expression of G_v ^(max) is reported in Eq. (1).

$$G_{v}^{(\max)} = \frac{k_t^2 Q_{load} C_{in} \eta \pi^2}{8C_{out}}$$
(1)

It is important to point out that $G_v^{(max)}$ (Eq. (1)) is proportional to the value of the resonator figure-of-merit (FoM $\propto Q \cdot k_t^2$), which further highlights the advantage of CLMTs over PTs based on any other laterally vibrating piezoelectric resonator technology.

Fabrication Process

The CLMT of this work is formed by a 4 μ m thick AlN layer and two 0.1 μ m thick platinum IDTs placed on the top and bottom surfaces of the AlN film. The choice of using platinum for the bottom IDT was dictated by the need of growing a high quality AlN film. Platinum was also used for the top IDT in order to preserve high acoustic symmetry in the device cross-section.



Figure 3: Microfabrication process of the CLMT fabricated in this work: (a) Pt film was deposited on top of Silicon, through a 10 nm thick Ti layer used as adhesion layer, and patterned through lift-off process; (b) AlN film was deposited on top of the Pt film and vias in the AlN were formed; (c) Pt film was deposited on top of Si and patterned through lift-off process; (d) AlN film was etched through the use of a SiO₂ hard mask that was preferred to traditional photoresist mask to attain steeper AlN sidewalls; (e) Si substrate was released by XeF_2 isotropic etching.

The devices were fabricated using a four-mask micro-fabrication process (Figure 3): 10/100 nm of Ti/Pt was deposited and patterned on top of a high resistivity silicon wafer to form the bottom IDT. Next, a 4 μ m thick AlN film was sputter-deposited.



Figure 4: Scanned-Electron-Microscope picture of a fabricated CLMT.

Then, we etched AlN through wet etching to form the vias. Next, 10/100 nm of Ti/Pt was deposited and patterned to form the top IDT. Then, the AlN film was etched by ICP in Cl₂ based chemistry to define the width of the AlN plate.

This was done through the use of a hard mask made out of 2 μ m of SiO₂ so as to attain steep AlN sidewall angles (>75°). Finally, the Silicon substrate underneath the resonator was released through XeF₂ isotropic etching. A SEM picture of a fabricated CLMT is shows in Figure 4.

Simulated Performance of 920 MHz 3-fingers CLMTs

The design of the CLMT proposed in this work was aided by 2D-FEA. In particular, we investigated the dependence of $G_v^{(max)}$ from the pitch of the IDT (*i.e.* W). The maximum simulated $G_v^{(max)}$ -values were attained when W was set to be around $\sim 5 \,\mu\text{m}$. Such W-value $(W^{(tx)})$ permits to maximize the input- k_t^2 when the device output port is left floating. In addition, we detected a small sensitivity of Gv(max) to the width of the metal-strip $(W_{el}^{(out)})$ forming the output portion of the IDTs. So, in the attempt of attaining the highest G_v^(max)-value, $W_{el}^{(out)}$ was chosen to be almost half ($W_{el}^{(out)}=2 \mu m$) of the width of the metal strips forming the input portion of the IDTs (Wel⁽ⁱⁿ⁾=3.5 μ m). As verified by FEA, such choice enables a $\sim 10\%$ improvement in the G_v^(max)-value with respect to the scenario in which W_{el}^(out) were be the same than Wel⁽ⁱⁿ⁾. Such improvement is originated from a slight increase of the device k_t^2 . The 2D-FEA simulated G_v- and input-admittance (Yin) distributions as functions of frequency are reported in Figure 5. As evident, a k_t^2 in excess of 4.1% was estimated by 2D-FEA. Such a high k_t^2 -value enables a $G_v^{(max)}$ -value in excess of 40 when assuming a Q_{load} -value equal to 1000.



Figure 5: 2D-FEA simulated Y_{in} -(in purple) and G_{v} - (in green) as functions of the frequency (f). The simulated responses were computed by assuming a Q_{load} equal to 1000. The resonant modeshape of the total displacement is also shown. As expected, the device displaces less underneath the output-port than underneath its input-port. This is due to the transformation mechanism explaining the origin of the voltage amplification in piezoelectric transformers.



Figure 6: In red) measured Y_{in} relative of the fabricated CLMT; in black) analytically predicted Y_{in} using the Q_{load} , C_{in} , C_{out} , k_t^2 and η values measured experimentally. In orange) measured G_v of the fabricated CLMT; in blue) analytically predicted G_v (Eq. 3) using the Q_{load} , C_{in} , C_{out} , k_t^2 and η values measured experimentally.

Measured Performance of 920 MHz 3-finger CLMTs

We experimentally characterized the performance of a fabricated 3-finger 920 MHz TFE CLMT (Figure 4). The device was tested, in

air, in a RF probe station using GSG-probes. Its performance are reported in Figure 6. As evident, values of k_t^2 , Q_{load} and $G_v^{(max)}$ in excess of 4%, 1000 and 39 were directly extracted from the measured Y-matrix. However, a small variation of f_{res} was measured with respect to its predicted value through FEA. This discrepancy is due to fabrication uncertainties associated with the actual thickness of the IDTs with respect to the value considered in the finite-element simulation (Figure 5).

CONCLUSION

We report the performance of the first piezoelectric transformer (PT) based on the cross-sectional Lamé-mode resonator technology. The device, which we labeled as a cross-sectional Lamé-mode transformer (CLMT), operates around 920 MHz and shows a record-high open-circuit voltage-gain ($G_v^{(max)}$) in excess of 39. Such a high $G_v^{(max)}$ -value demonstrates the ability of CLMTs to form low-loss narrow-band matching network and voltage-amplification stages in next generation miniaturized AC-to-DC and DC-to-DC converters.

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MULTIMODE BLACK PHOSPHORUS NANOMECHANICAL RESONATORS WITH INTRINSIC MECHANICAL ANISOTROPY AND ELECTRICAL TUNABILITY

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ABSTRACT

We report on the design and experimental demonstration of first electrically tunable multimode resonant nanothe electromechanical systems (NEMS) enabled by thin black phosphorus (P) crystals with intrinsic strong mechanical anisotropy. Combined with finite element modeling, we show that mechanical anisotropy in the black P crystal is clearly manifested in the mode sequence, frequency spacing, and mode shapes in black P NEMS resonators, which differ from those of isotropic resonators. We further experimentally demonstrate a multimode black P NEMS resonator. The multimode resonances measured and spatially mapped in black P nanoresonators, show good agreement with the design, and provide experimental evidences for the effects of the crystal's intrinsic mechanical anisotropy on the multimode resonances. We also demonstrate the ability of tuning the multimode responses by electrical gating. The findings in this study shall help open new possibilities for future realization of onchip electromechanical functions that can efficiently utilize the mechanical anisotropy at truly nanoscale.

INTRODUCTION

Black P has rapidly emerged as a new elemental, directbandgap, atomic-layer semiconducting crystal. In addition to its attractive electronic and optical properties, it is also particularly interesting for enabling new two-dimensional (2D)NEMS devices owing to its unique in-plane mechanical anisotropy (Fig. 1) that is unavailable in other atomic-layer crystals demonstrated to date [1]. In particular, it is predicted that its in-plane elastic moduli in orthogonal directions can differ by nearly 500% [2]. In comparison, the Young's modulus of silicon, the most-studied anisotropic MEMS material, only varies up to 50% with crystal orientation [3].



Figure 1: Crystal structure and mechanical anisotropy of black phosphorus (P), a new atomic-layer material. (a) Illustration of black P crystal consisting of vertically-stacked corrugated layers of P atoms. (b) Anisotropic in-plane elastic moduli of black P along armchair (E_{Yx}) and zigzag (E_{Yy}) directions.

To date, elastic properties of 2D materials are measured using nanoindentation method, in which a sharp indentation tip deflects a suspended 2D flake of known geometry and clamping conditions, and the Young's modulus is inferred from the load-deflection curves [4]. For black P, however, the strong anisotropy in elastic properties makes it less straightforward to directly apply the nanoindentation method. Theoretical predictions for its Young's moduli [2,5-7] (summarized in Table 1) range from 15.1GPa to 44.0GPa in armchair axis (E_{Yx}) and from 49.9GPa to 166.0GPa along zigzag axis (E_{Yy}); which present new complications and challenges toward deterministically designing devices with specific resonance responses.

Table 1:	Theoretical	calculations of	of Young	's moduli c	of black P.
			., .,		./

Work	$E_{\rm Yy}$ (GPa)	$E_{\rm Yx}$ (GPa)	$E_{\rm Yy}/E_{\rm Yx}$
Appl. Phys. Lett. 2014 [2]	166.0	44.0	3.77
arXiv 2014 [5]	92.7	44.0	2.11
Sci. Rep. 2014 [6]	49.9	15.1	3.30
J. Phys. D. 2014 [7]	106.4	41.3	2.58

DESIGN AND EXPERIMENT PROCEDURES Design of Black Phosphorus (P) NEMS Resonators

A close examination of the results in the literature shows that while the absolute values in E_{Yx} and E_{Yy} differ significantly from study to study, the ratio of the elastic moduli in the two crystal orientations (E_{Yy}/E_{Yx}) , a measure of the degree of anisotropy, differs by a factor of less than 2 among these studies (from 2.11 to 3.77). We therefore use E_{Yy}/E_{Yx} as a design parameter in this study instead of sweeping two independent parameters (E_{Yy} and E_{Yx}). In addition, we present the simulated frequency spacing results in form of frequency ratios (between a particular mode and the fundamental), which only depends on the ratio E_{Yy}/E_{Yx} and is insensitive to device diameter (d), thickness (t) and the absolute values of E_{Yy} and E_{Yx} .

We use finite element modeling (FEM, via COMSOL) to calculate the expected multimode responses of circular black P drumhead resonators. To focus on the effects of mechanical anisotropy, we fix most device parameters (which do not affect the frequency ratios), and only vary E_{Yy} (while fixing E_{Yx} =44.0GPa) in order to sweep E_{Yy}/E_{Yx} values. Table 2 shows the simulation parameters used in the device design. For comparison, we also show results from an isotropic case in which E_{Yy} =44.0GPa.

Description	Expression	Value
Device Diameter	d	10µm
Flake Thickness	t	95nm
Material Density	ρ	2690kg/m ³
Poisson's Ratio	ν	0.17 [2]
Elastic Modulus along	E	44.0GPa
Armchair Axis	$L_{\rm Yx}$	[2,5]
Range of Elastic Modulus		44.0GPa
along Zigzag Axis	$E_{\rm Yy}$	to
along Zigzag Axis		176.0GPa
Range of Elastic Moduli	E/E	1.2 to 4
Ratio	E_{Yy}/E_{Yx}	1, 2 10 4

 Table 2: FEM (COMSOL) simulation parameters of multimode black phosphorus (P) drumhead resonators.

Figure 2 shows the results from the FEM simulations. In presenting the results, we label every mode by using (m,n) to describe its mode index and shape, where *m* and *n* are the numbers

of antinodes (*i.e.*, 1 + number of nodal lines) along the armchair and zigzag crystal orientations, respectively.

There are two important reasons for using this naming system, which are associated with the simulation results: (i) the mode sequence and frequency spacing clearly depends on the elastic moduli ratio E_{Yy}/E_{Yx} , thus the modes cannot be simply identified using their sequence in the frequency spectrum or frequency ratios; instead, for each mode the mode shape itself is a good identifier; (ii) the mode shapes of a black P NEMS resonator clearly differ from those of an isotropic crystalline counterpart, therefore the convention used in describing the mode shapes for a circular disk resonator cannot be applied to such anisotropic resonators.



Figure 2: Effect of mechanical anisotropy on the multimode resonances in black P resonators (FEM simulation results). The frequency ratios of every mode to the fundamental f(m,n)/f(1,1) are plotted as functions of E_{Yy}/E_{Yx} , together with the simulated mode shapes and their evolutions with E_{Yy}/E_{Yx} . Note that the mode sequence depends on the E_{Yy}/E_{Yx} value.

Based on the above observations and considerations, it is important that one measures both the resonance frequencies and mode shapes in black P multimode resonators in order to correctly characterize such devices and thus verify the device designs. Therefore the device structures and experimental systems shall facilitate spectral and spatially resolved multimode resonance measurements.

Device Fabrication

Black P NEMS resonators are fabricated using a dry-transfer method that minimizes device exposure to the ambient or contaminations [8] to preserve the high quality of the black P crystals, as illustrated in Fig. 3. AFM measurements (for determining flake thickness) are performed after all electrical and optical measurements, again to minimize the exposure to the ambient prior to the resonance measurements. The resulting device (Fig. 4b) has a 95nm-thick (Fig. 4c) black P crystal suspended over a 10µm-diameter, 290nm-deep circular microtrench (fully clamped), and is contacted with Au/Cr electrodes. We choose the circular drumhead structure in order to best reveal the effect of intrinsic mechanical anisotropy of black P crystal on the multimode resonances [1].



Figure 3: Schematic illustration of device fabrication. (a) Exfoliation of black P onto PDMS stamp. (b) Dry transfer on to pre-patterned substrate. (c) The suspended device structure.

Measurement System

We drive the resonator electrostatically and detect the device motion with a laser interferometry, which facilitates spatial mapping of the resonance mode shapes through scanning spectromicroscopy measurements [9].



Figure 4: Measurement system and device structure. (a) Schematic illustration of custom-built laser interferometry system. Flexural vibrations of the Black P NEMS resonator are electrically

excited and interferometrically detected using a He-Ne laser. The chip is positioned in a vacuum chamber on a motorized XYZ stage (not shown). PD: Photodetector. BS: Beam splitter. (b) Microscopic image showing structural details of a 10µm-diameter circular black P drumhead resonator with electrodes. Scale bar: 10µm. (c) AFM height trace is measured along the dotted line in (b), showing a ~95nm flake thickness in the suspended region. (d) Schematic of the microspectroscopy measurement (a complete spectrum is obtained from one location), and spectromicroscopy measurement, where the laser scans across the device surface and the measured responses from all the locations are collected to reconstruct the mode shape.

Figure 4 schematically illustrates the laser interferometry system with high displacement sensitivity that is capable of detecting high-order/multiple mechanical resonances of 2D NEMS resonators. The flexural vibrations are actuated by applying an AC drive on top of a DC polarization $(V_g + \delta V_g)$ at a back gate, and detected by a He-Ne laser focused on the suspended area. The multiple reflections from the flake/vacuum and vacuum/Si interfaces create optical interference that is modulated by the device motion and read out by a low-noise photodetector. The device is measured in moderate vacuum (~10mTorr, vacuum chamber not shown) and the on-device laser power is ~600 μ W to minimize heating or degradation of the black P crystal.



Figure 5: Measured multimode resonance characteristics of the $10\mu m$ black P drumhead resonator. A total of 6 flexural modes up to 45MHz (HF/VHF bands) are detected in vacuum at room temperature, and the insets show zoom-in measurements of each mode and fitting to a finite-Q simple harmonic resonator model to extract both resonance frequency (f) and quality (Q) factor. The bottom plot shows Q vs. f for all the 6 modes.

The mode shapes are spatially mapped by employing the scanning spectromicroscopy technique [9], as illustrated in Fig. 4d. The vacuum chamber is mounted on a programed XYZ translational stage (not shown). We first define a 16μ m×16 μ m mapping area $X_i \in (-8\mu$ m, 8μ m), $Y_j \in (-8\mu$ m, 8μ m) that encloses the 10 μ m-diameter circular suspended region, with the origin (0,0)

located at the device center. The steps along both X and Y directions are set to be 1µm. At each position (X_i, Y_j) , we excite one particular mechanical resonance f(m,n) and measure the frequency response. The resulting signal amplitude (A_{ij}) is used to determine the device displacement at position (X_i, Y_j) for the given mode, and is then plotted in a 2D color map $(A_{ij}$ as a function of X_i , Y_j) for visualizing the corresponding mode shape.

RESULTS AND DISCUSSIONS Multimode Resonances

The measured frequency response over a wide frequency range (from 1MHz to 50MHz) is shown in Fig. 5. We identify 6 resonant modes which we investigate in detail. Measurement with a zoomed-in narrower frequency range is then performed for each mode; and the data are fitted to a damped driven harmonic resonator model (Fig. 5 insets). From the fitting results we extract the resonance frequencies (f) and qualify (Q) factors for each mode. These modes have frequency range of 10 to 45MHz (in HF/VHF bands), and Q factors from 15 to 75.

Table 3: Comparison between spatially-mapped and simulated mode shapes of the multimode responses.

Mode (<i>m</i> , <i>n</i>)	(1,1)	(2,1)	(1,2)
Experiment		Ö	
Simulation			
Mode (<i>m</i> , <i>n</i>)	(3,1)	(2,2)	(1,3)
Experiment			
Simulation	e		

These 6 modes are then carefully mapped using the spectromicroscopy techniques to resolve the mode shapes. The results are summarized in Table 3. From the measurements we clearly identify each measured mode shape with (m,n), finding very good agreement with simulations. Specifically, we observe clear difference between the measured mode shapes and those expected from a circular drumhead resonator made of isotropic material, which is characterized by nodal circles and nodal diameters. In contrast, in black P resonators, except for the few lowest modes, nodal lines do not take the form of circles or diameters, but as multiple curves that does not necessarily cross the center of the circle. The spatially resolved measurements clearly show that the intrinsic mechanical anisotropy is manifested in the multimode resonant responses in black P NEMS.

Frequency Spacing and Scaling

We further examine the frequency spacing and scaling for multimode black P resonators, with the results from both measurement and simulation shown in Table 4. We represent the frequency spacing by using the f(m,n)/f(1,1) ratio, the frequency of each mode normalized to the fundamental resonance.

The experimental values show good agreement with the predictions from the FEM simulations. Specifically, the frequency ratios from all the 6 modes (in the form of f(m,n)/f(1,1)) all fall within the expected range as in the device design (see Table 4). This shows that the measured elastic moduli ratio in black P crystal, E_{Yy}/E_{Yx} , resides between the values 2 and 4, as predicted by the theoretical calculations (listed in Table 1).

Table 4: Comparison between spatially-mapped and simulated multimode frequency ratios.

f(m,n)/f(1,1)	$E_{\rm Yy}/E_{\rm Yx}=2$	Experiment	$E_{\rm Yy}/E_{\rm Yx}=4$
<i>f</i> (1,1)/ <i>f</i> (1,1)	1.000	1.000	1.000
f(2,1)/f(1,1)	1.922	1.896	1.739
f(1,2)/f(1,1)	2.231	2.271	2.371
<i>f</i> (3,1)/ <i>f</i> (1,1)	3.109	2.999	2.749
f(2,2)/f(1,1)	3.512	3.488	3.399
<i>f</i> (1,3)/ <i>f</i> (1,1)	4.071	4.213	4.384

Tuning of Multimode Resonances

We further explore electrostatic gate tuning of the multimode resonances in the black P NEMS resonator. We measure the multimode resonances of the device between 1MHz and 50MHz, and perform the frequency sweep for each DC gate voltage (V_g) from -30V to 30V (with 1V step). Figure 6 shows that all the resonant modes exhibit clear gate tuning effect. Note that the measurement is taken with the laser position optimized for detecting the fundamental mode (1,1), and thus the signal for the higher modes are less visible using the same scale.



Figure 6: Measured gate tuning of the black P drumhead NEMS resonator, shown in a 3D-plot of multimode frequency response curves under V_{g} from -30V to+30V.

From the data in Fig. 6, we clearly observe two competing effects. In low V_g range ($|V_g| < 15V$), frequency decreases as V_g increases, as the device is dominated by capacitive softening effect [10]; at larger $|V_g|$ values, electrostatic tensioning effect [11] becomes clearly visible and dominant as the resonance frequency increases with the magnitude of V_g . We find that f can be tuned up by 163% (from 9.45 MHz to 24.9MHz for the fundamental mode) in this gate voltage range.

CONCLUSIONS

In summary, we have designed and fabricated the first multimode circular drumhead-shaped black P NEMS resonator. Using spatial mapping techniques in addition to the conventional spectral measurements, we successfully identify 6 resonant modes by directly visualizing their mode shapes. The measurements confirm that the black P resonator exhibit clear effects from mechanical anisotropy upon its multimode resonances, and show very good agreement with the expected device performance from the design. By varying the gate voltage, we observe strong tuning of the multimode resonances. The results demonstrate the power of using spatially-resolved multimode measurements to determine the device's material properties, and elucidate the mode sequence in black P NEMS resonators, which can lead to new resonant sensors or transducers that leverage the unique mechanical anisotropy in black P.

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FERROMAGNETIC RESONANCE IN BULK-ACOUSTIC WAVE MULTIFERROIC DEVICES

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ABSTRACT

The effect of ferromagnetic resonance (FMR) on the magnetomechanical coupling in thin-film multiferroic bulk-acoustic wave (BAW) devices is demonstrated. Devices consisting of piezoelectric and magneto-elastic materials are fabricated, and their electrical response is measured as a function of applied magnetic field magnitude and angle. As the applied magnetic field aligns the FMR frequency of the magneto-elastic layer with that of the BAW resonance of the structure, peaks in device conductance are observed. Approximate micro-magnetic simulations reproduce the trends seen experimentally. This represents the first investigation in the magneto-elastic coupling in thin-film BAW devices for potential applications for radio frequency (RF) miniature multiferroic devices.

INTRODUCTION

Recent interest in multiferroics, materials with coupled magnetic and electrical properties, has stimulated breakthroughs in a wide range of nanoscale magnetic systems, such as spin wave devices, non-volatile memory, and RF devices [1]. Multiferroics allow for the control of magnetization via electric fields, escaping the Ohmic losses of the typical electrical current control in miniature magnetic devices. However, single phase materials that exhibit these effects naturally are rare and have weak coupling. Strain-coupled multiferroic composites, laminates of piezoelectric and magnetostrictive materials which couple the electric and magnetic energy domains via mechanical strain, have demonstrated strong multiferroic coupling and offer a route for the realization of multiferroic devices.

To date, most multiferroic devices utilize a static magnetoelectric effect, where the DC magnetization is tuned using a DC voltage via strain-coupling [2]. This type of multiferroic coupling has been demonstrated for applications in spintronics, phase shifters, and memory devices. Relatively little work has been done on dynamic multiferroic coupling, where a time-varying voltage dynamically controls the magnetization state of a system via acoustic waves. Dynamic multiferroic coupling can also take advantage of FMR, greatly enhancing the coupling by allowing acoustic excitation of the magnetization at its resonance frequency. This enhancement of the coupling allows for RF applications in the GHz range.

Surface-acoustic wave (SAW) and BAW piezoelectric devices have already shown to be commercially successful at RF frequencies, finding applications in filters, duplexers, and oscillators [3]. This success make these devices very attractive candidates for dynamic multiferroics, where acoustic waves are necessary to mediate the magneto-electric coupling.

Previous work in this field has demonstrated SAW excitation of FMR in thin-film Ni [4, 5]. In these experiments, a set of interdigitated transducers (IDT's) excite a SAW on a LiNbO₃ substrate. This SAW then travels across the surface through a Ni thin-film to be received by a second set of IDT's. The power received by the second set of IDT's is then measured as a function of an applied magnetic field magnitude and angle, showing maximum absorption at the FMR frequency and with a bias angle



Figure 1: Structure of BAW multiferroic resonator. An applied electric field (\vec{E}) generates a standing acoustic wave. The standing acoustic wave leads to a dynamic excitation of the magnetic moment (\vec{M}) in the magnetostrictive layer. The total thickness (d) determines the resonant frequencies.

approximately 40° to the propagation direction of the SAW.

FMR has also been shown to allow for tuning in high-overtone BAW devices [6]. In these devices zinc oxide (ZnO) with a tilted caxis is deposited on an epitaxial yttrium iron garnet (YIG) magnetic film on a gadolinium gallium garnet (GGG) substrate. When electrically excited, the piezoelectric ZnO creates both shear and longitudinal standing acoustic waves throughout the GGG substrate, YIG, and ZnO. The effect of an applied bias on the reflection coefficient, S₁₁, was then measured. It was found that the resonance frequency could be tuned by 0.25 MHz around 2 GHz due to the magneto-elastic interaction.

In this work we investigate the effect of FMR in thin-film BAW resonant devices (Figure 1). Unlike the prior work, these devices do not require thick magnetic or piezoelectric substrates and can be fabricated directly on Si. As the resonance is completely contained within the thin-film structure, substrate isolation and attenuation of the acoustic wave does not need to be considered. In addition to the fundamental thickness-extensional mode, these devices can take advantage of any available low order harmonic modes to excite FMR in the magnetoelastic layer, avoiding the complications involved when using higher order harmonic devices. Such a structure has previously been proposed as a miniature antenna, potentially allowing for highly efficient radiation [7]. We also present the first experimental study on the dependence of outof-plane angular dependence of the magnetic bias on the magnetoelastic response in thin-films.

THEORY

The energy density due to the magneto-elastic interaction is given by

$$u_{me} = -\frac{3}{2}\lambda_s\sigma(\cos^2\theta - \frac{1}{3}) \tag{1}$$

where σ is the applied stress, λ_s is the saturation magnetostriction, θ is the angle between the applied stress and the net magnetization.



Figure 2: a) Colorized scanning electron microscope (SEM) image of a completed device. b) Manufacturing process for device fabrication.

If it is assumed that a normal stress is applied to the z-axis, the effective magnetic field due to an applied stress can be calculated as follows:

$$\vec{h}_{me} = -\frac{1}{\mu_0} \frac{du_{me}}{dM_z} \hat{z} = \frac{1}{\mu_0} \frac{3\lambda_s}{M_s^2} M_z \sigma \hat{z}$$
(2)

Here M_s is the saturation magnetization and $M_z = M_s cos\theta$ is the magnetization along the axis of the applied stress.

In the static case, the magnetization of the magnetic material will rotate in the direction of the effective field until it minimizes the total energy that it experiences. In the dynamic case however, the precession of the magnetization around the DC magnetic bias field cannot be ignored and the Landau-Lifshitz-Gilbert equation must be used to describe the magnetization dynamics. In the absence of dissipation, this equation is

$$\frac{d}{dt}\left(\vec{M}+\vec{m}\right) = -\mu_0 \gamma \left(\vec{M}+\vec{m}\right) \times \left(\vec{H}_0 + \vec{H}_d + \vec{h}_d + \vec{h}_{me}\right) \quad (3)$$

where \vec{M} is the DC magnetization, \vec{m} is the oscillating magnetization, γ is the gyromagnetic ratio, \vec{H}_0 is the applied bias field, \vec{H}_d is the demagnetization field due to DC magnetization, and \vec{h}_d is the demagnetization field due to the oscillating magnetization.

This equation can be solved to give the small-signal magnetic susceptibility tensor (χ) in the frequency domain, and is found to be a function of the angle of DC magnetization and the magnitude of the applied field [8]. It can be shown using this calculation that the magnitude of the susceptibility exhibits resonant behavior, increasing dramatically at frequencies near its resonant frequency. The resonant frequency, known as the FMR frequency, can be calculated from the Kittel equation, which in the case of a thin-film with an in plane magnetic bias is

$$\omega_r = \mu_0 \gamma \sqrt{(H_0 + M_s)H_0} \tag{4}$$

The susceptibility tensor can be used with the effective magnetoelastic magnetic field to find piezomagnetic coefficients analogous to those of piezoelectric materials [7].

$$\vec{m} = \chi \vec{h}_{me} = \left(\chi \cdot \frac{1}{\mu_0} \frac{3\lambda_s}{M_s^2} M_z \hat{z}\right) \sigma_{33} = d_{i3}\sigma_{33} \tag{5}$$

Just as piezomagnetic coefficients can be defined, a magnetomechanical coupling coefficient can similarly be defined.

$$K_{33}^{2} = \frac{d_{3i}^{t} \mu_{ij}^{-1} \cdot d_{j3}}{c_{33}} = \frac{\left(\left(\chi_{3i} \frac{1 \ 3\lambda_{5}}{\mu_{0} \ M_{5}^{2}} M_{2} \right)^{t} \cdot (1 + \chi)_{ij}^{-1} \cdot \left(\chi_{j3} \frac{1 \ 3\lambda_{5}}{\mu_{0} \ M_{5}^{2}} M_{2} \right) \right)}{\mu_{0} c_{33}} \propto \chi \quad (6)$$

Here c_{33} is the out-of-plane stiffness of the magneto-elastic material, t is the transpose operator, $\mu = \mu_0(1 + \chi)$ is the permeability, and i and j are the tensor indices. Since the susceptibility is a function of frequency, this equation shows that the magneto-elastic coupling is highly dependent on frequency, with the magneto-mechanical coupling coefficient approaching its peak near FMR. The magnetization naturally wants to oscillate at the FMR frequency, which means that the magnetization couples best with stimuli that oscillate at the FMR frequency (regardless of whether it's a magnetic field or strain). This effect has previously been demonstrated both numerically and experimentally [5, 9].

EXPERIMENTAL DETAIL

Design and Fabrication

To experimentally investigate this coupling, a tri-layer structure made of a piezoelectric layer (720 nm AlN), a magnetoelastic layer (735 nm Ni), and an acoustic buffer layer (300 nm Si_3N_4) were fabricated (Figure 2). An AC voltage applied across the piezoelectric generates an acoustic standing wave throughout the structure. The strain from this standing wave then drives the oscillation of the magnetization in the magneto-elastic layer. The acoustic buffer ensures that the peak of the strain wave is in the



Figure 3: Diagram measurement system for magneto-mechanical coupling experiments.



Figure 4: Colormaps of the percent change of device conductance as a function of frequency and magnetic bias. Red indicates an increase of conductance and blue indicates a decrease in conductance. Dashed lines are of the estimated FMR response found through simulation. a) Effect of magnetic field at the first resonant mode (1.8 GHz) and 0° bias angle. b) Effect of magnetic field at the second resonant mode (3.2 GHz) and 0° bias angle. c) Effect of second resonant mode at 45° bias angle. d) Effect of second resonant mode at 85° bias angle.

magnetostrictive layer to maximize the coupling of mechanical energy to magnetic energy. Thicknesses were chosen such that the peak of the acoustic wave would lie in the center of the Ni layer. Gaps were included in the magneto-elastic layer in an attempt to minimize the eddy current loss associated with a continuous film.

First, 10 nm Ti / 100 nm Pt is evaporated on high resistivity Si (>10 k Ω ·cm) to serve as the bottom electrode for the device and patterned via a lift-off process. The choice of Pt as the bottom electrode is integral for device operation as it promotes growth of high quality c-axis oriented AlN. Next, 720 nm of AlN blanket film is reactively sputtered on the wafer. 10 nm Ti / 100 nm Pt is then evaporated and lifted-off to pattern the top electrode laver. Sputter deposition of 10 nm Ti / 735 nm Ni follows and is also patterned by a lift-off process. The pressure for the Ni deposition was tuned to minimize residual film stress and to avoid the "winging" effects typical of sputter lift-off process by keeping the mean free path of the sputtered Ni long. Following Ni deposition, 530 nm of Si₃N₄ is deposited via PECVD and then etched in CHF3 plasma. The AlN layer is then etched in a mixture of CF4 and Ar plasma for access to the substrate and bottom electrode. This etch has high selectivity to Si with respect to Pt, meaning that over-etching will etch into the substrate and allow for an easier release without damaging the Pt bottom electrode. The devices are then released from the substrate using XeF₂ gas phase etching of the Si. During this step, the Si₃N₄ was unintentionally etched to a thickness of approximately 300 nm.

Measurement

To facilitate measurement, a fabricated device was packaged

as the terminating load of a coplanar waveguide (CPW) transmission line, which connects to a vector-network analyzer (VNA) via a flange mount SMA connector (Figure 3). The packaged device was then placed in an electromagnet where its reflection coefficient, S₁₁, could be measured as a function of the applied magnetic field magnitude and angle. Control of the applied angle was made possible through the use of 3D printed mounts. Measurements were made at angles of 0° , 45° , and 85° relative to the plane of the device. From the measured S₁₁ values, the admittance of the packaged system was then calculated to study the electrical response of the system as a function of the applied magnetic field.

RESULTS

From the measured S_{11} response, the complex admittance of the system was calculated. The system conductance, which is related to the amount of power dissipated by the system, is then found by taking the real part of the admittance. As the magnetic field increases, the ferromagnetic resonance frequency of the Ni film also increases, as seen by the Kittel equation (Equation 4). The oscillation of the magnetization is maximized when the FMR frequency aligns with an acoustic resonance mode, causing the most power to be absorbed by the magneto-elastic layer. This manifests itself as a peak in the measured conductance.

Figure 4 shows colormaps of the percent change in conductance of the packaged system as a function of magnetic bias magnitude and frequency at the first two resonant modes. The percent change is measured relative to the frequency response at

approximately zero magnetic bias, perfect zero bias being unachievable due to nonzero magnetic remanence of the electromagnet poles. At 1.8 GHz, the fundamental resonant mode, no field dependence of the conductance is seen as the bias needed for FMR is too low to align the magnetic moments, so FMR is not possible. The changes in conductance seen here at high bias were determined to be due to trace magnetic material in the SMA connectors and cables. Effects can be seen at 3.2 GHz, the second harmonic of mechanical resonance, under the same bias fields, confirming the frequency dependence of the magneto-mechanical coupling. Here the highest change was found at an applied magnetic bias of 237 Oe, indicating that this bias stimulates FMR at 3.2 GHz.

When the bias moves out-of-plane a higher magnetic field is needed to stimulate FMR due to the increase in demagnetization energy. At 45° , the peak conductance at 3.2 GHz was found to be at 361 Oe. At 85° a much higher field is needed to magnetize the film in the mostly out-of-plane orientation, with the peak conductance occurring at a bias of 2320 Oe.

To confirm that this effect is due to FMR, micro-magnetic simulations were done in the Object Oriented MicroMagnetic Framework (OOMMF) developed by NIST to estimate the magnetic susceptibility of the film [10]. A 735 nm Ni thin film is stimulated by an RF magnetic field at the resonant frequency of the mode of interest. The magnitude of the applied DC magnetic bias field is then stepped through values within the range applied during measurement. The amplitude of the steady-state magnetization oscillation is calculated as a function of magnetic field. This corresponds to the dashed lines in Figure 4. Maxima in these curves indicate a bias that stimulates FMR. The bias fields corresponding to the maximum change in conductance follow the same trends of the FMR response estimated by the OOMMF model and match the simulated values within 30% or less. Discrepancies between measurement and modelling are expected as crystallographic, domain, and exchange effects are not included in the model. Exchange effects, in particular, have been found to be important when investigating the magneto-elastic response near FMR [11]. However, the matching trends of maximum conductance and simulated susceptibility with respect to bias angle strongly suggests that this effect is due to FMR within the magneto-elastic layer.

CONCLUSION

In this work we investigate the effect of FMR on the magnetoelastic coupling of thin-film BAW devices. We find that the device conductance peaks at particular magnetic bias magnitudes and the trends of the angular dependence match that of FMR simulated with OOMMF. With further advancements of the experimental system, full analysis of power dissipation due to FMR will enable the possibility of quantitative measurements of dynamic multiferroic coupling for applications to RF devices.

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HIGH SENSITIVITY BIDIRECTIONAL PRESSURE SENSOR BASED ON FORCE FREQUENCY EFFECT IN MICROMACHINED AT-CUT QUARTZ RESONATORS

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ABSTRACT

In this paper, we report an AT-cut quartz diaphragm pressure sensor based on force frequency effect. Commercially available 100 μ m thick AT-cut quartz blanks were thinned down using micromachining processes to achieve high pressure sensitivity. The quartz sensor is resonated in thickness shear mode (TSM) and change in resonance frequency is monitored with change in pressure. Based on the signal-to-noise performance, we report a resolution of 2.39 mTorr for the 83.56 MHz resonator and 11.14 mTorr for the 45.5 MHz resonator. The range of linear operation for 45.5 MHz and 83.56 MHz was comparable to up to 80 Torr with the latter having higher sensitivity.

INTRODUCTION

Vacuum pressure sensors are widely used by industries like semiconductor equipment manufacturing and biomedical instrumentation in systems like metal deposition, plasma processing, process control, etc. These processes require a high vacuum environment and thus require sensors that can sense pressures in the range of 10^3 Torr to 10^{-8} Torr. Since there is no single technology that can operate throughout this regime, a combination of multiple pressure sensors, like Pirani gage (for $\sim 760 - 10^{-3}$ Torr) and cold cathode gage (for $\sim 10^{-2} - 10^{-8}$ Torr), is employed.

Micro electro mechanical systems (MEMS) based vacuum sensors have been extensively studied, but so far have been limited by the range of operation, reliability in various environments or bulkier integration with systems [1]. Micromachined Pirani gages are available commercially, but they suffer from small range of operation (\sim 10 - 100mTorr) [2]–[4]. Miniaturized ionization gages are good for the high vacuum regime but are not suitable for pressures > 1 mbar absolute pressure [5]–[7].

Both Pirani and cold cathode sensors rely on indirect inference of pressure based upon heat transfer and ionization currents that depend not only on the pressure but also on the gas composition and various other factors relating to a vacuum chamber. Capacitance diaphragm gages (CDG) measure pressure by sensing the deflection of a diaphragm to applied pressure. These devices can provide highly accurate measurements of vacuum from $10^3 - 10^{-3}$ Torr. However, several of these sensors with varying full scale ranges are required to cover such a large pressure range with optimized resolution. Further extension of the range of the CDGs is extremely challenging since the change in capacitance for pressure signals of less than 10⁻⁴ Torr results in extremely small diaphragm deflections. Under such inputs, the corresponding capacitances changes are in the $10^{-15} - 10^{-18}$ F range and therefore difficult to resolve under standard operating ambient. Extensive research was done on micromachined pressure sensors in the early 1990's showing the potential of such devices for high vacuum measurements [8]-[10].

Force frequency effect in quartz crystal has been known since the 1960's, where researchers were trying to understand the stability of crystal oscillators under various applied load conditions [11]. Specifically, the case of response of a quartz plate resonator under transverse loading conditions was theoretically studied by Markenscoff and Lee [12]. The fractional frequency change $\Delta f/f_0$ was determined to be a function of the zeroth and first order strains and first order strain gradients, and is related through second and third order elastic stiffness. This effect is used extensively for pressure sensing using quartz resonators in oil exploration for pressure measurements >1 bar [13]. However, due to the lack of effective micromachining techniques for quartz, the use of this effect for sensing vacuum pressures has not been explored.

With the motivation to span a large pressure range with high resolution, we present a micromachined AT-cut quartz diaphragm pressure sensor utilizing the force frequency effect. Unlike a CDG, the quartz pressure sensor does not require a closely placed (sub-micron) counter electrode and hence places less stringent constraints in the pressure sensor design. Thinned inverted mesa quartz resonators can be readily fabricated using deep reactive ion etching (DRIE) of quartz. Differential pressure applied across such a high-O thickness shear mode resonator diaphragm results in a uniform transverse load and consequently bending of the quartz diaphragm. The resulting change in resonance frequency is monitored by constantly measuring the at-resonance impedance characteristics of the resonator as function of applied pressure. This design allows for the realization of differential, gage, or absolute pressure sensing depending upon the pressure used on one of the faces (reference pressure) of the diaphragm. Sensitivity and range of operation with different thickness of resonators has also been studied. We also observed bi-directionality in the response of the device.

THEORETICAL BACKGROUND

Application of a differential pressure across the quartz diaphragm of circular shape will result in a peak deflection w_0 at the center of the diaphragm. In the absence of any residual stress in the diaphragm, the peak deflection of the quartz diaphragm can be related to the differential pressure Δp as [14]

$$w_0 = \left(\frac{3}{8} \frac{r_q^4}{t_q} \frac{1 - v_q^2}{E_q} \Delta p\right)^{1/3}$$
(1)

where t_q is the thickness and r_q is the radius of the quartz resonator diaphragm, E_q is the Young's modulus and v_q is the Poisson's ratio of quartz respectively.

For a rectangular plate resonator, clamped on one end and with an applied point load at the free end of the beam, the model for frequency shift of the quartz resonator with applied load in terms of second and third order elastic constants and zeroth and first order strains can be given by [12]:

$$\frac{\Delta f}{f_0} = \frac{1}{2c_{66}} \left(2C_{66} \varepsilon_1^{(0)} + C_{661} \varepsilon_1^{(0)} + C_{662} \varepsilon_2^{(0)} + C_{663} \varepsilon_3^{(0)} + \right.$$

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$$C_{664}\varepsilon_4^{(0)} - \left(\frac{t_q^2/\sqrt{48}}{\pi C_{66}} \left(C_{165} \frac{\partial \varepsilon_5^{(1)}}{\partial x_1} + C_{561} \frac{\partial \varepsilon_1^{(1)}}{\partial x_3} + C_{563} \frac{\partial \varepsilon_3^{(1)}}{\partial x_3} \right) \right)$$
(2)

where C_{66} is the second order and C_{pqr} is the third order elastic stiffness coefficients, respectively, $\varepsilon_m^{(0)}$ and $\varepsilon_m^{(1)}$ are the zeroth and first order strains, t_q is the thickness of quartz, and x_1, x_2 , and x_3 are the three principal axes in the Cartesian coordinate system.

EXPERIMENT SETUP Sensor Fabrication

100 µm thick, 1 inch diameter AT-cut quartz wafers were cleaned in Nanostrip® for 30 minutes and then 15 nm chromium (Cr) and 150 nm gold (Au) was deposited as seed metal layers using an e-beam evaporator. First lithography was done using photoresist SPR 220 to define the etch regions. 12-15 µm Nickel was electroplated that acts as a hard mask for plasma etching. Patterned quartz wafers were then etched in Alcatel AMS 100 inductively coupled plasma reactive ion etcher (ICP-RIE) using Argon and SF₆ gases. After completion of etching, the remaining metal mask was removed in agua regia (HCl: HNO₃:: 3:1), 20 nm/150 nm of Cr/Au was deposited using an evaporator, second lithography was done and Cr/Au was wet-etched to define an electrode on one side of the resonator. A third lithography was done on the other side of the resonator using LOR5A and SPR 3012. Again, 20 nm/ 150 nm of Cr/Au was deposited using an evaporator and finally lift off was done using Remover PG to define electrodes. Figure 1 schematically illustrates the process flow. The fabricated quartz sensors were cut into smaller pieces and mounted on a modified ceramic dual in-line package having a 5 X 5 mm² square hole machined in the center. RTV 157 silicone epoxy was applied all around the quartz piece to hermetically seal the two sides of the sensor while minimizing any possibility of stressing the quartz as shown in Fig. 2.



Figure 1: Sensor fabrication process



Figure 2: Finished device mounted on the package

Sensor Characterization Setup

The packaged device in the ceramic package was mounted on a custom made package fixture. This fixture hermetically seals the two sides of the quartz diaphragm with the help of a gasket on one side and an O-ring on the other. Figure 3 shows the mounting of the sensor on the custom fixture in detail. Finally, this fixture is connected to a vacuum system with the help of Swagelok fittings. The two sides of the diaphragm are isolated with the help of an isolation valve and a pressure difference is created by introducing nitrogen on one side of the diaphragm. The pressure is monitored using an MKS Baratron[®] capacitance manometer and an MKS Cold Cathode Gage. The resonance characteristics of the sensor were monitored with the help of 4294A Agilent Precision Impedance Analyzer. Figure 4 shows a schematic of the test setup.



Figure 3: Sensor assembly process



Figure 4: Schematic of test setup

RESULTS AND DISSCUSSIONS

All pressure sensing experiments were done after bringing both sides of the diaphragm into the high vacuum regime (~ 10 µTorr) and then isolating the two sides with the help of the isolation valve. Thus, one side of the diaphragm is constantly under high vacuum whereas the other side is subjected to variable pressure by the introduction N2 gas through a needle valve. The quartz diaphragm is constantly resonated in thickness shear mode with the help of impedance analyzer. Its conductance and susceptance properties were monitored as a function of the applied pressure on one side of the quartz diaphragm. For high resolution and small pressure range of 1 - 2 mTorr, the susceptibility of the resonator was monitored at a constant frequency f_0 , set by the unloaded resonance frequency, which was determined by the peak of the conductance maximum as seen in the typical resonance characteristic of an 83.56 MHz resonator in Fig. 5. This method is limited to the frequency range between the two inflexion points in the susceptance curve shown in Fig. 5. Thus for measurements involving larger pressure range of > 1 Torr, the resonance frequency shift of conductance was monitored as a function of pressure.



Figure 5: Conductance and susceptance characteristics of an 83.56 MHz AT cut quartz resonator excited in thickness shear mode.

Pressure Sensitivity with Thickness

Figure 6 shows the dependence of the susceptance of the resonator as a function of applied pressure for two resonators. Both resonators have a diaphragm diameter of 1 mm. However, the thickness of the two resonators is 20 μ m, corresponding to $f_0 =$ 83.56 MHz and 37 μ m, corresponding to $f_0 = 45.5$ MHz. From eq. (1) it is clearly evident that as the thickness of the diaphragm is reduced, the peak deflection will increase proportionally. Thus, the strain and strain gradients in the diaphragm are also expected to increase, resulting in a fractional change in the frequency as given by eq. (2). Figure 6 shows the at-resonance susceptibility sensitivity of two devices as a function of the applied pressure. Clearly, a linear dependence of the frequency on applied pressure is observed for both the resonators. The straight lines are the least square fits to the experimentally obtained data and are used to calculate the pressure sensitivity for the two resonators. At the respective resonance frequencies, the slope of the 83.56 MHz resonator was 10.65 µS/mTorr, whereas for the 45.5 MHz resonator the slope is 3.46 μ S/mTorr. Thus, the slope for ~20 μ m (83.56 MHz) thick resonator is about three times higher than the slope of ~37 μm (45.5 MHz) resonator.

Noise Characteristics and Range of the Resonator

Resonator noise was experimentally measured to be 38.5 µS for the 45 MHz resonator and 25 µS for the 83.56 MHz resonator for a bandwidth of 1.35 Hz (i.e. for 0.74 sec time constant). Based on the sensitivity measurements shown in Fig. 6, this corresponds to a SNR resolution of 11.14 mTorr for the 45.5 MHz resonator and 2.39 mTorr for the 83.56 MHz resonator. The biggest source of noise could be attributed to the vibrations on the measurement set-up from various sources such as the vacuum pump and floor vibrations and thus could be improved by better damping and isolation of the sensors. The range of linear response for both the 45.5 MHz and 83.56 MHz resonators was 0 - 80 Torr. However, in this larger pressure range, the 83.56 MHz resonator showed only two times higher sensitivity than the 45.5 MHz resonator. The burst differential pressure for the sensor diaphragm for the 83.56 MHz resonator was determined to be around 350 Torr leading to a catastrophic failure of the sensor.



Figure 6: Comparison of at-resonance susceptance versus change in pressure for 20 μ m (83.56 MHz) and 37 μ m (45.5 MHz) thick resonators.

Pressure Sensitivity to Direction of Pressure Application

Figure 7 shows that the quartz pressure sensor response also depends on the side of the diaphragm on which the pressure was

applied. Similar observations have been reported for in-plane applied stresses, whereby changing the sign of diametric force application from tensile to compressive or vice-versa, resulted in the sign of the change in resonance frequency to reverse [11]. The cause of this behavior is currently under investigation.



Figure 7: Susceptibility v/s pressure for 83.56MHz resonator showing bi-directionality (Inset: Showing etch side)

CONCLUSION

In this report, we have demonstrated a micromachined quartz diaphragm based pressure sensor with a range of operation from 1 mTorr to 80 Torr. A resolution of 11.14 mTorr and 2.39 mTorr for 45.5 MHz (~37 μ m thick) and 83.56 MHz (~20 μ m thick) resonator has been demonstrated. The range of operation has also been explored where sensors show a linear response in the 0 – 80 Torr pressure range with the 83.56 MHz resonator. Bi-directionality of the pressure sensor has also been shown. The sensitivity is shown to be a function of thickness and thus can be tuned by changing the thickness of the quartz diaphragm. A theoretical model to explain the performance of the quartz resonator as a function of applied pressure is currently under development and will be presented in future.

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MODE- AND DIRECTION-DEPENDENT AKHIEZER DAMPING IN SINGLE-CRYSTAL SILICON RESONATORS

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ABSTRACT

In this work, we determine the intrinsic mechanical energy dissipation limit for single-crystal resonators due to anharmonic phonon-phonon scattering in the Akhiezer ($\Omega \tau \ll l$) regime and evaluate the quality factor limit for intrinsic silicon devices. The energy loss is derived using perturbation theory and the linearized Boltzmann transport equation for phonons, and includes the direction- and polarization-dependent mode-Grüneisen parameters in order to capture the strain-induced anharmonicity among phonon branches. Our new theoretical framework reveals that Akhiezer damping, previously thought to depend only on material properties, has a strong dependence on resonant mode shape. Critically, we show that shear mode devices do, in fact, experience Akhiezer damping, which is in direct contrast to the result produced when applying existing models. Our expression satisfies the pressing need for a reliable analytical model that can predict the Akhiezer damping limits for modern resonant microelectromechanical systems, where precise manufacturing techniques and accurate finite-element methods can be used to select particular vibrational mode shapes and crystal orientations.

INTRODUCTION

The performance of mechanical resonators is governed by dissipation of energy stored in the resonant vibrational mode to other acoustic modes or the environment [1]. Despite the prevalence of resonant microelectromechanical systems (MEMS) as high-performance inertial sensors, mass-based chemical sensors, timing references and frequency filters, the dissipation in these structures is not well understood. The loss can be difficult to determine because there is no single, predictive theory to evaluate the quality factor (Q), defined as $2\pi [(energy stored)/(energy loss per cycle)]$. Accurate prediction of Q has tremendous design implications because it is directly related to device performance metrics including sensitivity for resonant sensors, bandwidth for radio-frequency filters, and phase noise for timing references.

This work focuses on determining intrinsic dissipation limits in dielectric and semiconductor crystals, which are governed by the interaction between the elastic wave and thermal phonons. This interaction has two components: spatial phonon transport and local phonon scattering. We focus on the local effect, Akhiezer damping, because it is present in all vibration modes and serves as an ultimate upper bound on the performance of MEMS resonators. The Akhiezer damping limit is particularly relevant for highfrequency and bulk-mode resonators, where the spatial effect (thermoelastic dissipation) is negligible [2]. Here, we derive an expression for Akhiezer loss that captures the effect of anharmonic phonon-phonon scattering as well as crystalline anisotropy.

In Akhiezer damping, strain produced by the mechanical wave modulates the phonon frequencies and, consequently, the local equilibrium phonon distribution. The phonon populations cannot change instantaneously and will relax towards the modulated equilibrium distribution via phonon-phonon scattering when the thermal relaxation time (τ) is significantly less than the period of the mechanical wave. Time varying strain and finite τ mean the time-dependent phonon populations lag behind their (perturbed) equilibrium value. This relaxation towards equilibrium

is an entropy-producing process that consumes energy from the elastic wave. It is important to note that the Akhiezer damping model applies only when the scattering rate $(1/\tau)$ is significantly larger than the frequency (Ω) of the mechanical vibration $(\Omega \tau \ll I)$, which is the case at room temperature for commonly used acoustic materials such as silicon, germanium, and quartz [3].

This limit on mechanical energy dissipation was first described by Akhiezer [4] and later solved by Woodruff using the linearized Boltzmann transport equation (BTE) and the Debye approximation to arrive at a simplified, isotropic expression for internal friction (Q^{-l}) involving only classical, bulk parameters [5].

$$Q^{-1} = \frac{\gamma_0^2 C_v T}{\rho c^2} \Omega \tau \tag{1}$$

Here, *T* is the ambient temperature, ρ is the material density, *c* is the Debye average sound velocity, C_{ν} is the specific heat per unit volume, and γ_0 is the average Grüneisen parameter associated with thermal expansion. Woodruff derives this result by assuming that all phonon modes are perturbed identically by the strain wave and neglecting the perturbation of the internal temperature of the solid. This expression is often used to make an order of magnitude prediction of the internal friction limit for a given material [6]. Modern devices, however, approach and even exceed Woodruff's oversimplified limit [7-10], indicating a need for a more predictive, analytical model that can be evaluated in a straightforward manner using known material constants and reliably compared with experimental results.

In this work, we rigorously derive an expression for the internal friction limit due to anharmonic phonon-phonon scattering. We solve for the energy loss using the analytically sound Boltzmann transport method, but rather than assuming all phonon modes are perturbed equally by strain, we include the directional- and polarization-dependent mode-Grüneisen parameters. The resulting expression for Akhiezer damping can still be evaluated using bulk parameters, but depends on the vibrational mode shape and crystal orientation of the resonator.

ANHARMONIC ENERGY LOSS

The derivation of the anharmonic phonon-phonon dissipation begins with the assumption that the strain wave is time harmonic with wave vector **K** and angular frequency Ω so that $\vec{\epsilon}(t) \propto \exp[i(\mathbf{K} \cdot \mathbf{r} - \Omega \tau)]$, where **r** is the position vector. The strain perturbs the frequencies (ω) of all thermal phonons,

$$\omega_i = \omega_{i0} [1 + \vec{\gamma}_i \cdot \vec{\epsilon}(t)] = \omega_{i0} + \Delta \omega_i \tag{2}$$

where the index *i* denotes a pure-mode branch characterized by a direction and polarization along a crystal axis so that ω_i is the instantaneous phonon frequency of the *i*th branch and ω_{i0} is the unperturbed equilibrium phonon frequency. Each high-symmetry direction in the Wigner-Seitz cell, the primitive Brillouin zone (BZ), has a longitudinal mode that is polarized along the direction of phonon propagation and two transverse modes that are polarized perpendicularly to the propagation direction. Fig. 1 shows the 13 principal crystallographic directions in the Wigner-Seitz cell for the diamond lattice. Strain, even in just a single direction, deforms the entire BZ, resulting in a perturbation of all phonon branches.

For cubic crystals, the strain tensor is assumed to be symmetric, so we can express it compactly as a vector $\vec{\epsilon}(t)$ with six

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Figure 1. Wigner-Seitz cell for the diamond lattice centered about the Γ point. (a) Three <100> directions (blue). (b) Six <110> directions (red). (c) Four <111> directions (green). There are 13 high-symmetry crystal directions and each contributes three pure modes, one longitudinal and two transverse, resulting in 39 distinct phonon branches.

components, denoted ϵ_j , where the index *j* indicates one of the six strain directions: three normal and three shear. Consequently, $\vec{\gamma}_i$ also has six components that are the mode-Grüneisen parameters $\gamma_{i,j}$, corresponding to the anharmonic perturbation of the *i*th branch frequency due to strain in the *j*th direction. Here, we assume that the $\gamma_{i,j}$'s are independent of the phonon frequency and wavenumber. As a result of the phonon frequency perturbation, the instantaneous phonon population in each branch N_i deviates from its thermal equilibrium Bose-Einstein distribution $N_{i0} = (e^{\hbar\omega_{i0}/k_BT} - 1)^{-1}$, so that $N_i = N_{i0} + \Delta n_i$.

We use the phonon BTE to describe the relaxation of the branch distributions, N_i . Akhiezer damping is a local effect, so we assume uniform strain and eliminate all spatial terms in the BTE. Thus, the relaxation towards equilibrium is solely determined by the scattering term. Following the approach of Woodruff and others, we employ the relaxation time approximation to describe the scattering term as the decay of N_i towards a Bose-Einstein distribution N'_{i0} at a modulated local temperature $T' = T + \Delta T$ [11]. We can reduce the phonon BTE to

$$\frac{\partial N_i}{\partial t} = \frac{N_i - N_{i0}'}{\tau} \tag{3}$$

where we have characterized the relaxation process using the average phonon lifetime, τ . Fig. 2 outlines the theoretical framework showing the perturbation of phonon frequencies and the corresponding dissipative relaxation.

The energy loss per cycle is simply the time average of the rate at which energy is lost via phonon-phonon scattering,

$$U_{loss \, per \, cycle} = -\sum_{k,i} \langle H_i \cdot \left(\frac{\partial N_i}{\partial t}\right)_{scatt} \rangle_{cycle} \tag{4}$$

where $H_i = \hbar \omega_i$ is the phonon Hamiltonian, $\langle \bullet \rangle_{\text{cycle}}$ denotes the time average over one period of the mechanical vibration, and the summation is over all possible wavenumbers (k) and phonon branches (i). Solving the BTE assuming plane wave solutions, $\Delta \omega_{i_L} \Delta n_{i_L} \Delta T \propto \exp[i(\mathbf{K} \cdot \mathbf{r} - \Omega \tau)]$, yields

$$U_{loss \, per \, cycle} = \pi (\langle \gamma_{i,j}^2 \rangle - \langle \gamma_{i,j} \rangle^2) C_v T \frac{\Omega \tau}{1 + \Omega^2 \tau^2} \tag{5}$$

for uniaxial strain, where ϵ_i is the only nonzero strain component [12]. In the above expression, the angle brackets indicate an average of all phonon branches, and we have eliminated the cumbersome summation by assuming each branch contributes equally to the total phonon specific heat. Our energy loss expression depends not on the average Grüneisen parameter (γ_0), but the difference between the mean-squared mode-Grüneisen parameter squared, which reflects the fact that the phonon modes tend to relax towards the modified Bose-Einstein distribution determined by the perturbation of local temperature due to applied strain.

If the second- and third-order elastic coefficients are known, theoretical values for $\gamma_{i,j}$ can be obtained [3] and the energy-loss

expression can be calculated using only bulk parameters. Ultimately, as we will show, the total energy loss can be expressed as a superposition of uniaxial losses.



Figure 2. Overview of the energy loss due to anharmonic phononphonon scattering. Normal strains ε_1 - ε_3 lead to a perturbation of phonon frequencies in each branch, characterized by the mode-Grüneisen parameter $\gamma_{i,j}$. The modulation of phonon frequencies (and energies) means the respective branch populations (N_i) are out of equilibrium. Each branch distribution relaxes towards the perturbed equilibrium distribution (N'_{i0}) via phonon-phonon scattering, characterized by the phonon lifetime, τ . The energy loss that occurs during this irreversible relaxation process is assumed to come from the acoustic wave, because that is the source of the perturbation. Shear strains ε_4 - ε_6 , however, tend to result in reduced dissipation because only a fraction of the phonon branches are involved in the relaxation process, due to the restriction that volume is preserved under shear deformation.

ENERGY STORAGE

The quality factor is a ratio of energy stored to energy lost per cycle, so it is important to account for the anisotropy and mode dependence in both. For cubic crystals, the energy storage is anisotropic and depends on the deformation profile. The elasticity of a cubic crystal can be described by relating stress ($\vec{\sigma}$) and strain ($\vec{\epsilon}$) using the second-order elastic tensor.

For a given vibration mode and orientation, an effective Young's modulus or storage modulus E_{eff} can be defined so that the energy storage per unit volume is $E_{stored} = \frac{1}{2}E_{eff}\epsilon_0^2$ [13]. The modes of interest are width extensional (WE), square extensional (SE), cubic extensional (CE), and Lamé; their deformation profiles and expressions for effective storage moduli are given in Table I. These are commonly used vibration modes for single-crystal silicon resonators oriented along [100] axes. The WE, SE, and CE modes are modes where the deformation of the solid is primarily due to extension (and contraction) along one, two, or three principal axes, respectively. The Lamé mode is a pure-shear mode with only one nonzero strain component.

Table I. Displacement profiles and elastic storage moduli for common vibration modes of a single-crystal rectangular parallelepiped with edges oriented along the [100] directions.

Width- Extensional	Square- Extensional	Cubic- Extensional	Lamé (shear)
<	-		
$\frac{(c_{11}-c_{12})(c_{11}+2c_{12})}{c_{11}+c_{12}}$	$c_{11} + c_{12} - \frac{2c_{12}^2}{c_{11}}$	$\frac{c_{11}+2c_{12}}{3}$	c_{44}

QUALITY FACTOR

Using the definition of the quality factor, the energy storage expression in the previous section, and the energy loss in Eq. (5), we can write the quality factor as

$$Q = \frac{E_{eff}}{\left(\langle \gamma_{i,j}^2 \rangle - \langle \gamma_{i,j} \rangle^2\right) C_{\nu} \tau} \frac{1 + \Omega^2 \tau^2}{\Omega \tau}$$
(6)

for the uniaxial case. If we employ Woodruff's simplifications that the material is isotropic so all $\gamma_{i,j}=\gamma_0$ and that $\Delta T=0$, and assume the storage modulus is simply the bulk modulus $B=\rho c^2$, this expression reduces identically to Eq. (1) in the low-frequency limit. Woodruff's assumptions allow for simple estimation of Qusing bulk material data, but it is important to note that these assumptions are not self-consistent. If the material is assumed to be isotropic and $\gamma_{i,j}=\gamma_0$, then the average $\langle \gamma_{i,j} \rangle = \gamma_0$, which implies that $\Delta T \neq 0$. In fact, when this assumption is applied rigorously, $\langle \gamma_{i,j}^2 \rangle = \langle \gamma_{i,j} \rangle^2 = \gamma_0^2$ and the dissipation in Eq. (5) is zero. We note that the result derived here matches Zener's phenomenological form for quality factor due to anelastic relaxations in a solid [14], as expected, because the frequency dependence of the dissipation arises from a phase delay between the applied strain wave and the corresponding relaxation of the ensemble of thermal phonons.

MODE-DEPENDENT ENERGY LOSS

The expression in Eq. (6) accounts for the anisotropic and mode-dependent energy storage, but only includes loss due to uniaxial strain. In order to more accurately determine the losses, we define an effective mode-Grüneisen parameter $\gamma_{i,eff}$ as the weighted average of the components of $\vec{\gamma}_i$ by their corresponding strain component ϵ_j . Thus, we can capture the perturbation of the phonon branch frequency due to strain in more than one direction.

Symmetry conditions in cubic crystals allow us to write $\langle \gamma_{i,eff} \rangle = \alpha \langle \gamma_{i,1} \rangle$ for pure extensional modes and $\langle \gamma_{i,eff} \rangle = \alpha \langle \gamma_{i,1} \rangle$ for pure shear modes, where α is a coefficient determined by the relative axial strain in the x, y, and z directions. The quality factor is reduced by a factor of l/α^2 [12]. In order to retain the simplicity of Eq. (1), we express the quality factor as

$$Q = \frac{E_{eff}}{\Gamma_a^2 C_v T} \frac{1 + \Omega^2 \tau^2}{\Omega \tau}$$
(7)

and define the anharmonic Grüneisen parameter as

 Γ_{a}^{2}

$$c^{2} = \alpha^{2} \left(\langle \gamma_{i,j}^{2} \rangle - \langle \gamma_{i,j} \rangle^{2} \right)$$
(8)

Our expression shows that the quality factor depends distinctly on the resonant frequency, due to a mismatch between the period of the elastic wave and the phonon lifetime, and the strain profile, due to fundamental differences in the strength of the phonon frequency perturbation, which we quantify using α^2 .

Mason and Bateman establish that the mode-Grüneisen parameters can be determined from second- and third-order elastic moduli and calculate $\gamma_{i,1}$ and $\gamma_{i,5}$ for silicon and germanium [3]. Critically, they show that $\langle \gamma_{i,1} \rangle \approx \gamma_0$ for extensional modes and $\langle \gamma_{i,5} \rangle = 0$ for shear modes, as expected. The final parameter, the phonon lifetime (τ), is determined using the definition of bulk thermal conductivity $\kappa \equiv \frac{1}{3}C_{\nu}c^2\tau$. This is, in effect, an average time constant over all phonon branches.

RESULTS AND DISCUSSION

We evaluate Q for single-crystal, intrinsic silicon resonators and compare the Akhiezer-limited performance for common vibration modes. Fig. 3 shows the room temperature $f \times Q$ product as a function of the mechanical resonant frequency for the WE, SE, CE, and Lamé modes of a [100]-oriented intrinsic silicon resonator evaluated using the expression in Eq. (7) along with Woodruff's result for reference and a number of experimental results from silicon resonators in the literature [7-10, 15-24]. Due to the quadratic dependence of Q on resonant frequency, the curves remain constant up to ~20.5 GHz, corresponding to the condition $\Omega \tau = 1$. Again, we note that the Akhiezer damping model applies only when $\Omega \ll 1/\tau$, so the results should only be interpreted below this value; at higher frequencies, the strain varies faster than the phonon scattering rate, so an alternate model, often called Landau-Rumer dissipation, should be used instead [25].



Figure 3. Anharmonic and anisotropic $f \times Q$ product limits vs. resonant frequency at room temperature for silicon. The theory curves (solid lines) indicate that Lamé mode resonators have the highest Q limit, followed by the WE, SE and CE modes. The points are experimental results from high-Q resonators in the literature [7-10, 15-24], a number of which exceed Woodruff's limit (dashed line), indicating that the simplified, isotropic expression does not provide sufficient accuracy. The ungrouped points are measurements of higher-order harmonics, so the assumption in this work of uniform strain is not directly applicable.

The (solid) theory curves show that the upper limit on Q for the WE mode is greater than that of the SE mode, which is, in turn, larger than the CE mode. In the WE mode, Poisson contraction reduces the net phonon frequency perturbation compared to the uniaxial case. The SE and CE modes oppose the natural Poisson contraction leading to increased dissipation. Our results indicate that the quality factor limits for silicon at a specified resonant frequency can vary by more than an order of magnitude. The Lamé mode has the highest upper bound on O for the modes considered in this work, despite having the smallest energy storage modulus. For shear waves, phonon scattering is limited to volume-preserving phonon branches, leading to reduced energy dissipation because fewer branches, and less phonon energy, are subject to the relaxation process. We note that Woodruff's isotropic formula actually predicts infinite Q (zero dissipation) for shear modes because the average Grüneisen parameter for volume-preserving modes is zero. The limitations of Woodruff's expression have been acknowledged in the past [11,26], but we provide a viable alternative expression that shows that shear-mode vibrations do in fact lead to anharmonic phonon-phonon dissipation.

The evaluation of the anharmonic and anisotropic expression derived in this work indicates that Woodruff's order-of-magnitude result (dashed line) fails to provide an upper bound on the quality factor due to Akhiezer damping. In fact, several silicon resonators with quality factors that exceed Woodruff's limit have already been fabricated and measured in the literature [7-10], indicating the important need for our more accurate damping model that provides a robust upper bound on the performance of modern micromechanical resonators.

The experimental points are broadly categorized by geometry and mode type. The highest Q resonators of a given type are grouped horizontally, reinforcing the assertion that the $f \times Q$ product is constant for a particular mode shape, in accordance with Eq. (7). The evaluation of Eq. (7) provides an upper bound on the quality factor, so it only predicts the performance of devices that are limited by anharmonic phonon-phonon dissipation, meaning other loss mechanisms including TED, air-damping, and anchor loss must have insignificant contributions. We also note that the theory lines in Fig. 3 use the idealized mode profiles in Table I, and, do not necessarily predict the exact behavior of the devices, because the actual vibrational modes are complicated functions of the geometry and boundary conditions of the structure. Additionally, the results here are for intrinsic silicon, and do not account for variations due to dopant species and density. The most accurate results can be obtained if the doping dependencies of second- and third-order elastic coefficients and thermal conductivity, which determines τ , are known.

CONCLUSION

In this work, we provide an analytical expression for the quality factor due to anharmonic phonon-phonon dissipation that explicitly includes the anisotropic energy storage and loss in a cubic semiconductor or dielectric crystal. We provide a rigorous derivation of the anharmonic loss using the phonon BTE and introduce the important simplifications that must be made in order to facilitate quality factor calculation using known material parameters. These simplifications are presented and justified and evaluated for the most common vibration modes for [100] silicon. Our advanced model combined with relatively straightforward evaluation allows for meaningful comparisons between theory and experimental results and provides insight into the efficiency of different vibrational modes in the Akhiezer dissipation limit. Critically, we show explicitly that shear-mode vibrations do, in fact, experience Akhiezer damping, but have reduced dissipation in this limit because only a fraction of phonon branches are involved in the dissipative relaxation process. The formulations introduced in this work can be extended to account for doping dependence (when appropriate material data are available) and integrated into a finite-element solver to provide the most accurate predictions of phonon-phonon dissipation for arbitrary vibration profiles.

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RING-SHAPED PIEZOELECTRIC MICROMACHINED ULTRASONIC TRANSDUCERS (PMUT) WITH INCREASED PRESSURE GENERATION

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ABSTRACT

As the application space of piezoelectric micromachined ultrasonic transducers (pMUT) expands and enters the industrial realm, it is necessary to continue improving the performance of individual devices, two of the most important metrics of which are generated acoustic pressure magnitude and directivity. Here, we present the first ring-shaped pMUT (r-pMUT), which have been fabricated using CMOS-compatible aluminum nitride (AlN) as both the transduction and elastic material. The r-pMUT exhibit newfound design freedom compared to standard circular pMUT (cpMUT), as their resonance frequency is found to be insensitive to mean radius r_0 . The frequency responses of fabricated devices were measured and shown to match well with finite element model (FEM) simulations. By comparing r- and c-pMUT designed to operate at 1.5 MHz with the same film stack, we show that rpMUT are capable of generating over 11.8× the pressure and with over 9× the directionality of their circular counterpart. As such, this device architecture could allow for highly efficient pMUT in applications that benefit from high pressures and directionality, such as rangefinding, fingerprint scanning, and therapeutics.

INTRODUCTION

Piezoelectric micromachined ultrasonic transducers (pMUT) are receiving great levels of interest as a technology that can be integrated with consumer electronics or in the medical field, with a growing application space that includes fingerprint scanning, imaging, gesture recognition, and therapeutics [1]-[4]. Typical pMUT operation includes both the launching and receiving of ultrasonic acoustic waves, making the pressure generated per volt one of the most critical performance metrics for pMUT. Therefore, improving the response of pMUT has been the topic of a myriad of recently reported design innovations [4]-[8]. For example, the dual-electrode bimorph pMUT shows a response that is 4× that of the state of the art [4]. Curved aluminum nitride (AlN) diaphragms show a static displacement that is $50 \times$ higher than flat devices with the same diameter [5]. An impedance matching tube has been used to increase the sound pressure level (SPL) by 350% [6], and concentric venting rings are shown to increase the SPL by 67% [7]. Conversely, a forward-looking array of concentric ring-shaped capacitive MUT (cMUT) was used to generate large pressures at an acoustic focal point while minimizing the number of drive channels [8], however, a thorough study of the individual transducers that comprise the array has not been reported.

In this work, we introduce the first ring-shaped pMUT (rpMUT) and study the ring-shaped MUT as a standalone structure. At the cost of very little added fabrication complexity, r-pMUT are shown to have significantly increased design freedom compared to conventional circular pMUT (c-pMUT) by allowing for the same resonance frequency to be achieved with different topographic geometries. Furthermore, finite element method (FEM) simulations are developed and used to evaluate the performance of the r-pMUT by way of comparison with a c-pMUT designed with the same film stack and resonance frequency. The r-pMUT shows a 1080% greater SPL, which is the largest reported improvement over the state of the art. Additionally, the r-pMUT displays a directivity factor that is $8.3 \times$ larger than that of the c-pMUT.



Figure 1: Cross-section schematic drawing of a ring-shaped pMUT being driven by an applied voltage.

RING-SHAPED PMUT DESIGN

Concept

A conceptual cross-sectional schematic of the (deformed) rpMUT is shown in Fig. 1, along with the design variables which define the diaphragm: mean radius r_0 and width w. Conversely, diaphragms in c-pMUT are defined only by the clamped boundary radius a. The operating principle is similar to that of a standard cpMUT: stress is induced in the active AlN layer when a voltage is applied between the top and bottom electrodes due to the d_{31} piezoelectric effect, which in turn generates a bending moment and deforms the diaphragm in an axisymmetric flexural mode as shown. In contrast to the c-pMUT which has only one clamped boundary, the r-pMUT has two clamped boundaries at $r_0\pm w/2$, since both the center post and substrate ideally act as mechanical anchors. As a reference to the reader, it was found throughout this study that in order to design r- and c-pMUT with the same film stack and equal resonance frequencies, $w\approx 1.48a$.

Fabrication

As the fabrication of the r-pMUT has no added complexity compared to the c-pMUT, the process used in this work nearly exactly follows the CMOS- compatible process used previously by our lab for c-pMUT [9], and is shown as a process flow in Fig. 2. The fabrication begins with (a), the sputter deposition of the AlN (1000 nm)/Mo (130 nm)/AlN (800 nm)/Mo (130 nm) film stack, where the $t_{AIN1} > t_{AIN2}$ to ensure that the neutral axis of the diaphragm is not inside of the active layer to prevent performance reduction [10]. In (b), the top electrode is patterned by a SF_6 plasma etch, then the ground vias are patterned by first depositing 500 nm of PECVD SiO₂ as a hardmask, followed by RIE of SiO₂ and a two-step etch of AlN using first a Chlorine-based plasma and a subsequent wet etch in MF-319. The top electrode is designed to have a 55% areal diaphragm coverage and mean radius of r_0 (i.e. is centered on the released diaphragm) as preliminary simulations indicate that this results in good coupling to the fundamental vibration mode of the diaphragm. The diaphragms are released (c) with a backside DRIE, and the devices are diced and mounted on a custom PCB (d) using thermal release tape and silicone adhesive to ensure a full seal.

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Figure 2: Fabrication process flow for the r-pMUT: (a) sputter film stack deposition, (b) top electrode patterning, hardmask deposition, and via etching (c) backside DRIE release etch (d) mounting to custom PCB for testing



Figure 3: (a) optical micrograph of a r-pMUT before mounting, (b) and (c) oblique and cross-sectional SEM micrographs, respectively, (d) optical image of the r-pMUT mounted on a custom PCB for testing.

Fig. 3a and 3b respectively show optical-top and SEMoblique views of the released r-pMUT (after completion of step (c) in Fig. 2), showing that the ground vias are fully exposed without



Figure 4: (a) measured displacement frequency responses of four r-pMUT showing the small dependence of resonance frequency on r_0 , and strong dependence of resonance frequency on w. (b) Measured, simulated, and analytical vibration mode shape, displaying excellent agreement and ensuring that the r-pMUT ($w_{des}=90 \ \mu m$ and $r_0=300 \ \mu m$) vibrates in the intended mode.

any residual AlN or damage to the Mo ground plane. Fig. 3c shows a cross-section SEM of a r-pMUT that broke while mounting for imaging. During the DRIE backside release process, issues with silicon undercutting result in larger fabricated diaphragm widths than the designed values. This effect is most pronounced along the center post, and was found to result in a 20-30 μ m discrepancy between the designed and actual width by using confocal microscopy (LEXT OLS3000, Olympus) for sidewall profile measurements. For this reason the designed and effective widths will henceforth be referred to using the variables w_{des} and w_{eff} , respectively. Methods of mitigating this phenomenon are currently being explored. Fig. 3d presents an optical image of the fabricated devices attached and wire-bonded to a custom PCB for testing.

RESULTS

Device Characterization

The fabricated r-pMUT were characterized with a laser Doppler vibrometer (LDV, OFV-5000, Polytec Inc.) in air. Fig. 4a displays the results of four typical displacement frequency responses for r-pMUT with center radii r_0 =150 (blue) and 300 (green) µm, and designed widths w_{des} =90 and 100 µm. While high frequency spurious resonances are observed and are likely due to fabrication imperfections, the fundamental resonance frequencies for r-pMUT with w_{des} =90 and 100 µm varied from 1.45-1.47 MHz and 1.13-1.14 MHz respectively, both of which represent a less than 1.5% deviation as r_0 is doubled. As there is only one possible geometry for a given film stack and operational frequency with c-pMUT, this insensitivity of resonance frequency to r_0 allows for an extra degree of design freedom in r-pMUT and can be explained by considering the equation for resonance frequency:

$$f_0 = \frac{l}{2\pi} \sqrt{\frac{k}{m}} \tag{1}$$

where k and m are the modal mass and stiffness of the structure. It is intuitive that mass is proportional to surface area A, and we can approximately say that $m \propto r_0$ after considering that $A \approx 2\pi r_0 w$, however, the relationship between k and r_0 is less intuitive. A firstorder approximation can be derived for the case of $r_0 \gg w$ by conceptually "unwrapping" the r-pMUT into a long rectangular strip with length $L=2\pi r_0$ and width w, anchored along the two long boundaries and free along the two short boundaries. By definition k=F/x, where x is the static deflection, and it follows that

$$k = \frac{F}{x} = \frac{PA}{x} = \frac{PLw}{x} \tag{2}$$

where *P* is a uniform pressure applied on the diaphragm. Since the static deflection of a rectangular diaphragm with these boundary conditions does not depend on the length of the diaphragm, the only variable in Eq. 2 that depends on r_0 is *L*, so it can be approximated that $k \propto r_0$. Therefore, r_0 cancels out of Eq. 1, and f_0 should show no dependence on the mean radius.

In order to ensure that the devices operate in the assumed mode, the vibration mode shape was additionally measured by recording the vibration amplitude around the resonance frequency and at different radial locations of the device. The results of such measurements taken at 1.4 MHz for an r-pMUT with w_{des} =90 µm and r_0 =300 µm are displayed Fig. 4b, alongside comparisons with a simplified finite element method (FEM) simulation by COMSOL Multiphysics, and an analytical approximation which is given by

$$\phi(r) = \left(1 - \overline{r}^2\right)^2 \tag{3}$$

where $\phi(r)$ is the mode shape, and $\overline{r} = 2(r-r_0)/w_{eff}$ is a normalized radial coordinate that varies from -1 to 1 at the inner and outer



Figure 5: Comparison of measured and simulated displacement frequency responses for r-pMUT, showing good agreement between the low-frequency displacement and resonance frequency.

clamped boundaries, respectively. All three methods show excellent agreement, and as expected, the center post and bulksubstrate act as anchors and the peak displacement occurs near r_0 . Furthermore, an effective width of w_{eff} =115µm is observed, which is consistent with the aforementioned fabrication errors.

In order to further validate the FEM model and ensure its ability to correctly predict device performance, frequency response simulations were also compared with LDV measurements. Fig. 5 shows one such comparison for an r-pMUT with w_{des} =90 µm, w_{eff} =115 µm, and r_0 =225 µm where an excellent agreement between model and experiment is displayed by a close match between measured and simulated values for both (1) low-frequency displacement (0.57 and 0.73 nm, respectively) and (2) resonance frequencies (1.47 and 1.48 MHz, respectively).

Acoustic Performance

To compare the acoustic performance of r- and c-pMUT, FEM simulations of two prototype devices (ring: $w=115 \mu m$, r_0 =225 µm, 55% electrode coverage, and circle: a=77.25 µm, 70% electrode coverage) designed for 1.5 MHz operation with 800/1000 nm thick AlN film stacks were performed. Fig. 6a and 6b respectively show the peak displacement and far-field pressure for the prototype transducers. Despite having a peak displacement of only 186 nm/V compared to 298 nm/V for the c-pMUT, the rpMUT displays a peak pressure of 201 Pa, which is more than $7.7 \times$ greater than the 23 Pa generated by its circular counterpart. The r-pMUT is able to generate such high acoustic pressures because $P \propto V_{v}$, where $V_v = v_{avg}A$ is the volume velocity and v_{avg} is the average diaphragm velocity, and the radiating area of an rpMUT may be made much larger than that of the c-pMUT by choice of r_0 . Indeed, as shown in Fig. 6c, the pressure generated by the r-pMUT shows a strong dependence on r_0 , and reaches a value of 272 Pa when r_0 =325 µm, over 11.8× that of the prototype c-pMUT. The simulated 1080% increase in SPL represents the largest reported improvement resulting from a design change. At very large r_0 the pressure begins to decrease due to increased mass loading from the acoustic medium and the effects of sidelobes. Fig. 6d shows the normalized far-field pressure from the ring and circle prototypes. Owing to its larger acoustic aperture, the prototype ring has a more directional acoustic output, displaying a directivity factor, which is the ratio of peak to average acoustic intensity, of 19 compared to 2 for the circle and thereby improving efficiency.

CONCLUSION

We have introduced, experimentally characterized, and performed FEM simulations on the first-ever ring-shaped pMUT (r-pMUT). An insensitivity of the resonance frequency on the mean radius has been observed, allowing r-pMUT an extra degree of design freedom compared to circular pMUT (c-pMUT), as the topographic geometry is no longer constrained by the resonance frequency. Owing to this fact, r-pMUT can be designed with a larger volume velocity than c-pMUT, thereby increasing their pressure generation. In fact, by comparing 1.5 MHz devices made of the same film stack, r-pMUT show an estimated 1080% increase in SPL and 830% increase in directivity over c-pMUT. These factors indicate that r-pMUT may be ideal for forward-looking applications that require large acoustic pressures, such as rangefinding, fingerprint scanning, and therapeutics. Further benefits may also be observed by creating concentric arrays of rpMUT, or even incorporating a concentric c-pMUT, and should be explored in the future.



Normalized pressure amplitude

Figure 6: Simulated (a) displacement and (b) far-field pressure frequency response near resonance of prototype transducers, (c) far-field pressure for r-pMUT with $w=115\mu m$ and various r_{0} , (d) normalized pressure amplitude of prototype transducers as a function of angle.

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SCANDIUM DOPED ALUMINUM NITRIDE BASED PIEZOELECTRIC MICROMACHINED ULTRASOUND TRANSDUCERS

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ABSTRACT

This paper presents the design, fabrication and characterization of piezoelectric micromachined ultrasound transducers (PMUTs) based on scandium doped aluminum nitride (Sc_xAl_{1-x}N) thin films (x = 20%). ScAlN thin film was prepared with a dual magnetron system and patterned by a reactive ion etching system utilizing chlorine-based chemistry with an etching rate of 160 nm/min. The film was characterized by X-ray diffraction (XRD) which indicated a crystalline structure expansion change compared to pure AlN and a well-aligned ScAlN film. ScAlN PMUTs were fabricated by a 2mask process based on cavity SOI wafers. ScAlN PMUTs with 50 µm diameter had a large dynamic displacement sensitivity of 25 nm/V at 17 MHz in air. twice that of AlN PMUTs with the same dimensions. Electrical impedance measurements indicated that the ScAIN PMUTs had 20% greater electromechanical coupling coefficient (k_t^2) compared to AlN PMUTs. The output pressure of a 7x7 ScAlN PMUT array was 0.7 kPa/V at ~1.7mm away from the array, which is ~3 times greater that of an 8x8 AlN PMUT array with the same element geometry and fill factor measured at the same distance.

INTRODUCTION

Micromachined ultrasonic transducers (MUTs) have been developed for many applications in recent years, such as medical imaging [1-2], gesture sensors [3], ultrasonic fingerprint sensors [4], and body-composition sensors [5]. Compared with conventional bulk ultrasonic transducers, MUTs have a better acoustic coupling, lower cost for two-dimensional array fabrication, and lower power consumption. Recently, piezoelectric micromachined ultrasonic transducers (PMUTs) have been rapidly developed due to the progress of piezoelectric thin films. Aluminum nitride (AlN) has been widely used for PMUT fabrication because it is available from a number of MEMS foundries and is compatible with CMOS manufacturing. However, compared to lead zirconate titanate (PZT), a piezoelectric material which requires high annealing temperature and is not process-compatible with CMOS, AlN has relatively low piezoelectric coefficient (e_{31,f}), which leads to low sensitivity and low electromechanical coupling (k_t^2) [6].

Recently scandium (Sc) doping has been proposed as a means to increase the $e_{31,f}$ of AlN, while maintaining process compatibility with existing AlN-based manufacturing [7]. Studies also found that with the increase of Sc doping concentrations, the stiffness of the thin film decreased and the dielectric constant increased [8]. Most of the previously-reported work on ScAlN focused on bulk acoustic wave (BAW) resonators or surface acoustic wave (SAW) devices which utilize the longitudinal piezoelectric mode and require high stiffness to achieve high frequency operation and high quality factor (*Q*) [9-10]. In this paper, we present flexural PMUT devices which use the transverse piezoelectric mode and where the reduced stiffness of ScAlN may provide a benefit over conventional AlN.

The schematic of each PMUT is shown in Figure 1. The PMUT was composed of a 1 μ m thick ScAIN piezoelectric layer, a 200nm Mo layer as bottom electrode and a 2.5 μ m thick silicon membrane. The vacuum cavity underneath the silicon membrane eliminates



Figure 1: Schematic of ScAlN PMUT.

possible squeeze-film damping between the PMUT membrane and the Si substrate.

FABRICATION

ScAlN thin film deposition

Sc_xAl_{1-x}N thin film (x = 20%) of 1 µm thickness was sputtered on 6 inch cavity SOI wafers (IceMOS Technology) in an Advanced Modular Systems (AMS) cluster tool with AlN deposition chambers and ion beam trimming module. The system used a standard dual conical magnetron with an AC deposition source. The ScAlN deposition process was in deep poison mode using targets composed of Al and Sc pieces. Locally adjusted magnetic field for target pieces of both Al and Sc guaranteed a constant thin film composition over the entire target life. Substrate rotation was utilized to compensate for the variation of the sputtering yield for different materials and composition non-uniformity across the substrate.

A 30 nm thick ScAlN was firstly deposited on the cavity SOI as a seed layer. Then a 200 nm thick molybdenum (Mo) layer was sputtered as the bottom electrode in a different chamber in the system without breaking the vacuum. Finally, 1 μ m thick ScAlN was sputtered on the Mo layer.

ScAIN PMUT fabrication

ScAlN PMUTs were fabricated based on a simple 2-mask process with cavity SOI wafers as reported in [6]. The process requires etching the ScAlN film to open vias to the Mo bottom electrode. An initial study of wet etching the ScAlN thin film using heated positive photoresist developer (Microposit MF-319) mainly composed of tetramethylammonium hydroxide (TMAH) showed that the ScAlN etch rate was ~50 nm/min at 60°C to 70°C, approximately 4 times slower than that of AlN thin films with the same thickness and at the same etching temperature. Then reactive ion etching (RIE) was studied with a combination of Cl2, BCl3 and He gases in a transformer coupled plasma (TCP) etcher. He gas is used as diluent to improve etch uniformity. A 6.5 µm thick g-line photoresist (OCG 825 35S, Fujifilm) was spin coated, patterned, and hard baked for 16 hours to be used as a mask. An etch rate of 160 nm/min was achieved with the recipe shown in the Table 1 with an etching selectivity of 0.4 to the mask. A 200 nm thick aluminum

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Table 1: Parameters for RIE of ScAlN thin film

Parameters	Values
Cl ₂ flow rate (sccm)	90
BCl ₃ flow rate (sccm)	30
He flow rate (sccm)	100
TCP RF Power (W)	550
RF Bias Power (W)	150



Figure 2: An optical microscope image of a $7x\overline{49PMUT}$ array. The individual PMUTs are 50 µm diameter and the array pitch is 70 µm.

(Al) layer was evaporated and patterned by a lift-off process to form the top electrode and contact pad for the bottom electrode. A PMUT array composed of 7×49 single PMUTs with 50 µm diameter and 70 µm pitch is shown in Figure 2. A cross-section scanning electron microscope (SEM) image of a PMUT, Figure 3, shows the dense columnar structure of the ScAlN film and Mo bottom electrode.

CHARACTERIZATION

ScAlN thin film characterization

The ScAIN crystalline structure was studied by X-ray diffraction (XRD). Figure 4(a) shows a comparison of the XRD peaks of pure AlN and ScAIN thin film with 1 µm thickness. The (002) peak of ScAIN was slightly shifted to a lower angle compared with that of AlN, indicating an expansion of the crystalline lattice according to Bragg's law. The rocking curve of the ScAIN (002) peak was also measured and is shown in Figure 4(b). The full-width-half-maximum (FWHM) of the peak is 1.7°, indicating that the c-axis of the ScAIN thin film is well aligned and predicting good



Figure 3: Cross-sectional SEM image of a ScAlN PMUT.



Figure 4: (a) Normal coupled XRD measurement of ScAlN and AlN films; (b) Rocking curve measurement of ScAlN (002) peak.

piezoelectric properties.

Dynamic characterization

The frequency response of ScAlN PMUTs and AlN PMUTs with the same geometry were tested in air using a laser Doppler vibrometer (LDV) in conjunction with a network analyzer. LDV measurements were collected on 16 ScAlN PMUTs across the wafer, resulting in a 17.5 +/- 1.5 MHz natural frequency and 22 +/- 4 nm/V dynamic displacement sensitivity at resonance. The results are shown in Figure 5. Cross-section SEM image showed that the Si thickness varied from 2.40 μ m to 2.93 μ m. Using equation (1), the observed variation in natural frequency is consistent with the measured thickness variation. Figure 6 compares the LDV results of ScAlN and AlN PMUTs. The dynamic displacement sensitivity of



Figure 5: Measured resonance frequency and dynamic displacement at resonance for ScAlN PMUTs with 50 µm diameter and 2.5 µm nominal Si thickness.



Figure 6: LDV measurement results for 50 µm diameter ScAlN and AlN PMUTs

the ScAIN PMUT is two times as large as that of the AlN device. The difference in the resonance frequency of ScAIN and AlN PMUTs is due to the stiffness reduction from Sc doping as mentioned earlier. The Young's modulus of ScAIN was estimated to be 200GPa via equation (1), which is consistent with the reported values obtained from ScAIN BAW devices [11],

$$f = \frac{1.63}{r^2} \sqrt{\frac{D}{\sum \rho_i t_i}} \tag{1}$$

where r is the radius, D is rigidity, ρ and t are the density and thickness of the PMUT layers. The rigidity D can be expressed as

$$D = \int_{bottom \, layer}^{top \, layer} \frac{E_z^2 z^2}{1 - v_z^2} dz \qquad (2)$$

where E_z is the Young's modulus and v_z is the Poisson's ratio of the material at a distance z from the neutral axis. The average quality factor of the PMUTs is Q = 140. The static displacement sensitivity, which is the peak displacement normalized by Q, $d_s = d_y/Q$, is ~180 pm/V. d_s is related to the transverse piezoelectric coefficient $e_{31.f}$ via:

$$d_s \propto \frac{e_{31,f} Z_n}{D} \tag{3}$$

where Z_n is the distance from the middle of the piezoelectric layer to neutral axis. The estimated $e_{31,f}$ is ~1.6 C/m² which is ~60% higher than that of AlN.

Electrical characterization

Impedance measurements of ScAlN and AlN PMUTs, Fig. 7, were performed in air using a GSG RF probe calibrated with an impedance substrate standard (Cascade Microtech). The electromechanical coupling factor k_t^2 was calculated by:

$$k_t^2 = \frac{\pi^2}{4} \frac{f_r}{f_a} \frac{f_a - f_r}{f_a}$$
(4)

where f_a and f_r are the anti-resonant and resonant frequency respectively. k_t^2 was 1.8% for ScAlN PMUTs, 20% higher compared to the AlN PMUT's 1.47% which was close to the value expected for a pure AlN device operating in transverse mode. The relative dielectric permittivity (ε_{ScAlN}) of ScAlN was also estimated from the impedance test as ~12 which is around 20% higher than that of pure AlN.

Acoustic characterization

An array of ScAlN PMUTs was immersed in Fluorinert (FC-70, 3M) and the output acoustic pressure was measured with a 70 μ m diameter needle hydrophone (Precision Acoustic, Inc.). The results are shown in Figure 8. A 7x7 ScAlN PMUT array was driven by 4 9 MHz 11 Vpp pulses from a function generator (Rigol, DG-4102). The measured pressure generated by the ScAlN PMUT array was detected at ~2.5 µs after the pulse generation, which



Figure 7: Impedance measurement results for 50 µm diameter (a) ScAlN PMUT and (b) AlN PMUT.

corresponds to ~1.7 mm from the PMUT surface to the hydrophone. The peak-to-peak pressure detected was ~8 kPa, which was 30% larger than ~6 kPa pressure generated from a 8x8 AlN PMUT array driven at 25 Vpp, suggesting 3x greater transmit efficiency from the ScAlN array. The dynamic displacement of a 17x17 ScAlN PMUT array driven with 11 Vpp was measured via LDV, Fig. 9. A ~11nm displacement of the center ScAlN PMUT was measured during the transmit (TX) excitation. The plot shows that the PMUT displacement from the returning echo is also visible 10 μ s after the TX pulse is sent. The 6.8 mm round-trip distance calculated from this pulse echo measurement is consistent with ~3.4 mm height of the Fluorinert above PMUT array. The echo generated deformation of PMUT was ~1 nm indicating a ~20 dB insertion loss from TX displacement to the RX echo displacement. This result is consistent with 16 dB spreading loss (which was verified using hydrophone



Figure 8: Pressure measurement results for 7x7 ScAlN PMUTs array



Figure 9, LDV measurement of pulse-echo from a ScAlN PMUT in the center of 17x17 Array

Material	$e_{31,f}$ (C/m ²)	833	E (GPa)	$\frac{e_{31,f}^2}{\varepsilon_{33}\varepsilon_0}(\text{GPa})$
PZT [12]	-14.0	1200	65	18.5
AlN [12]	-1.05	10.5	390	10.8
ScAlN (this work)	-1.6	12	200	24.1

Table 2, A comparison of piezoelectric materials properties

measurements at different distances) and 4 dB of loss due to the 40% fill-factor of the array.

CONCLUSION

ScAlN PMUTs appear to have important performance advantages over AlN PMUTs. The manufacturing process of the ScAlN PMUT is nearly identical to that of the earlier AlN device. A summary of the measured properties of the ScAlN film used here is presented in Table 2. Importantly, the figure of merit shown in the table's last column shows that ScAlN is 1.3 times better than PZT for PMUTs.

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SINGLE-CRYSTAL DIAMOND NEMS DEVICES FOR THE STUDY OF COLOR CENTERS

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ABSTRACT

We demonstrate fabrication methods for micro-/nanoelectromechanical systems (MEMS/NEMS) in single-crystal diamond chip. Such devices can be used to study embedded color centers, such as the nitrogen vacancy (NV) or the silicon vacancy (SiV). Color centers have discrete electronic and spin energy levels, which can be controlled by engineering the amount of strain in the diamond lattice. These strain fields can be effectively controlled using conventional MEMS/NEMS devices. As a specific demonstration, we show the tuning of SiV electronic energy levels via static actuation. The device design process accounting for various fundamental and experimental constraints is discussed.

INTRODUCTION

Solid-state qubits have been extensively studied for the last few decades in the field of quantum information science and technology (QIST). Recently, color centers in single-crystal diamond (SCD), have attracted attention as solid-state qubit candidates. The most extensively studied color center is the nitrogen-vacancy (NV) center, whose ground state spin sublevels have long coherence time (on the order of milliseconds) at room temperature. Another color center, which has received renewed attention recently, is the silicon vacancy (SiV) center, due to its excellent optical properties, such as the large zero-phonon line at room temperature (approximately 70%) and small inhomogeneous distribution.

These color centers possess discrete energy levels corresponding to electronic orbital states. The spin sublevels of these orbitals can be split to have different energies by the application of an external magnetic field. Such spin sublevels can be used to define a qubit. A qubit can be typically manipulated with external control fields such as a magnetic field (Zeeman effect) or an electric field (Stark effect).

Recently, it has been proposed that strain fields in the host lattice can be used to manipulate the quantum state of qubit [1]. Therefore, controlled generation of strain fields at the site of a target qubit, with a specific frequency and direction, has been of keen interest. The simplest approach is to use an external actuator, e.g. piezoelectric transducer, to induce static or dynamic motion [2-4]. External actuators, however, are typically bulky so that they are not compatible with solid-state qubit experiments in certain types of experiments. For example, in diamond color center studies optical control and read-out is necessary, and the sample needs to be placed under an (within a millimeter) objective lens. In the case of dynamic actuation, it can be conveniently achieved by attaching a piezo actuator externally and applying AC voltage [2, 4]. However, most commercially available piezo actuators for dynamic actuation have cut-off frequency near 1 MHz, limited by their mass

To date, high-overtone bulk acoustic resonator (HBAR) [5] and surface acoustic wave (SAW) devices [6] operating at few GHz frequency have been fabricated on single-crystal diamond and used to manipulate quantum states of NV center. The two platforms mentioned above demonstrated resonant coupling of spin states and parametric coupling of excited orbital states to strain, respectively. These devices are built on the flat surface of bulk sample and typically have large mechanical mode volume. However, many quantum information applications require the strong interaction of single mechanical quanta (phonons) and qubits, and this can only be achieved with small mechanical mode volumes. The realization of MEMS/NEMS devices in single crystal diamond can help achieve very small mode volumes, and allow actuation and detection of mechanical modes with electrical signals. However, there are several challenges in the fabrication of these devices, particularly since they should be compatible with optics experiments on color centers. In this work, we demonstrate MEMS/NEMS devices fabricated on SCD, which can be applied to color center experiments. In particular, we discuss the application of one such device to SiV electronic level tuning via application of static strain.



Figure 1: (a) Illustration of angle-etching, through steps (i)-(iv). (b,c) Fabrication steps of diamond nanobeams with (b) dielectrophoretic and (c) electrostatic actuation. Standard bilayer lift-off process is used. Fabrications steps are mostly the same between (b) and (c) except for the subtle difference in the pattern in (iii).

RESULTS

Fabrication

Currently, SCD can only be synthesized in the bulk form, which impedes the fabrication of free-standing nanostructures on them. To overcome such a challenge, in our earlier work, we reported a fabrication method for free-standing structures in SCD [7]. A Faraday cage is placed in the chamber during reactive-ion etching, which allows etching at an oblique angle. Illustration of our etching scheme (referred hereafter "angled-etching") is shown in Fig. 1(a). First, the mask pattern is formed by spinning e-beam resist (hydrogen silsesquioxane, (HSQ)) and writing with an ebeam lithography tool. The device is vertically etched down by reactive-ion etching (RIE), where oxygen plasma chemistry is applied [8]. The next step is angled-etching, in which a Faraday cage is placed inside the RIE chamber, and it alters the electrical

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potential and the sheath. The environment in the chamber accelerates ions in the plasma at a range of oblique angles, eventually shaping free-standing structures with a triangular cross-section. The physics of angled-etching has been systematically studied with three different materials [9]. With proper design of the mask pattern, various free-standing structures can be made at the micro- and nano-scale. Devices demonstrated by this technique includes nanomechanical resonators [10], as well as more sophisticated devices like photonic crystal optical cavities [11].

For the fabrication of an on-chip actuator, a standard bi-laver lift-off process is performed to place electrodes around the In this work, we present two complementary resonator approaches that result in different device configurations and driving mechanisms, suitable for color center experiments. In the first approach, based on dielectrophoretic actuation, electrodes can be placed on either side of diamond nanobeam in close proximity without physically contacting it. In the second approach, the electrodes are placed on the cantilever itself, and electrostatic actuation is performed. The fabrication process is illustrated in Fig. First, diamond sample is spin-coated with polymethyl 1(h)methacrylate (PMMA) multiple times, to ensure that the entire chip is covered with the resist. As a next step, pre-fabricated alignment markers are revealed by exposing the nearby area and developing the sample subsequently. (This step is not shown in Fig. 1 for simplicity, but it is important to achieve high precision alignment in the next step.) After the markers are cleared, a conductive layer (metal or ESPACERTM [12]) is formed on top of the PMMA layer to enhance the quality of the electron-beam lithography (EBL). Then electrode patterns are written and developed. After that, a quick oxygen plasma clean (20 sccm, 250 mTorr, 100W, 1 min) can be performed (optional) for a cleaner pattern and surface quality. Finally, desired metals are evaporated on the patterned device and lift-off is performed at 80°C in Remover PG to finalize the device. Depending on the dimensions of the nanobeam, critical point drying may be used to prevent the device from snapping down to the substrate.

Characterization

Devices made by the process shown in Fig. 1(b) can be dynamically actuated through dielectrophoretic force [13]. SEM images of the devices are shown in Fig. 2(a) and (b). We have reported dielectrophoretic actuation of devices previously [14]. Resonantly driven motion of such devices is shown in Fig. 3(a) and (b), when RF voltage was applied across two electrodes on either side. The motion of the cantilever is optically read out by using optical interferometry [15]. Displacement of the nanobeam motion can be thermomechanically calibrated by measuring thermal fluctuations [16]. Fig. 3(a) and (b) correspond to the cantilever and doubly clamped beam in Fig. 2(a) and (b), respectively.

Dielectrophoretic actuation can be useful for resonant driving when high mechanical quality factor (Q) is desired. However, the



Figure 2: SEM images of diamond nanobeams with (a, b) dielectrophoretic and (c) electrostatic actuation.



Figure 3: Resonantly driven motion of (a) cantilever and (b) doubly clamped beam with dielectrophoretic actuation.

force (deflection) generated for given voltage applied is typically smaller than in the case of the simple electrostatic actuation. For large static actuation, metals are placed near nanobeam in a slightly different fashion from the previous method as shown in Fig. 1(c). Most of the steps are similar except for the positioning of the electrode patterns. By making PMMA width slightly narrower than nanobeam width, evaporated metal is deposited both on the nanobeam and the bottom substrate. SEM image of the final device is shown in Fig. 2(c). Electrodes, one on the bottom, and the other on the nanobeam serve as driving electrodes. On the nanobeam, thin wire electrodes formed on either edge, so as to leave a large window for optical access to the color center. When a DC voltage is applied, the nanobeam is pulled down by electrostatic force, thereby applying strain to color centers embedded in diamond.

By using such an electrostatic MEMS device, we perform photoluminescence excitation (PLE) measurements on SiVs in the cantilever while deflecting the beam by applying DC voltage. The SiV is known to have two orbital states each in the ground and excited manifolds, and therefore its emission spectrum consists of four distinct lines [17]. Fig. 4 shows how the emission wavelengths of these four transitions depend on the applied voltage, in the case of the device shown in Fig. 2(c). The shifts in the emission wavelengths are entirely due to the applied strain since, due to the inversion symmetry, SiV does not show a linear Stark effect. Detailed analysis of electronic energy level shifts and the exact effect of strain are beyond the scope of the current paper, and will be presented elsewhere.

DISCUSSION



Figure 4: PLE spectra with varying applied voltage to the device shown in Fig. 2(c). Four distinct peaks reveal the electronic structure of a single SiV.

In this section, we discuss the design process for dielectrophoretic and electrostatic devices in the context of color center experiments. The dielectrophoretic scheme maintains high resonator Q factor, which is desirable for applications relying on dynamic actuation of mechanical modes. In particular, this is important to achieve strong coupling between the qubit and the mechanical resonator. This condition can be quantified by a figure called the co-operativity, which needs to exceed 1 [2, 4].

$$C = \frac{g^2}{n_{th}\kappa\gamma} > 1 \tag{1}$$

Here, g is the coupling rate between the qubit levels due to a single phonon in the mechanical mode, n_{th} is the thermal phonon occupation of the mechanical mode of interest, κ is the intrinsic mechanical damping rate (inversely proportional to Q), and γ is the qubit-dephasing rate. A large coupling rate requires the smaller scale of the device in general $(g \propto \sqrt{\frac{1}{l^3 w}}, l \text{ is the length and } w \text{ is}$ the width of the cantilever of rectangular cross section). Therefore, it is of great importance to achieve high Q for small-scale devices, which is known to be difficult [18]. Due to the premature stage of diamond fabrication compared to silicon fabrication, options for on-chip SCD mechanical actuator are limited. The most obvious approach is to deposit a conducting film and use it for electrostatic actuation as in Fig. 2(c). However, it is well known that film deposition causes degradation in Q [18]. Pursuing this goal is effectively similar to achieving high fQ product, which is the fundamental figure of merit in MEMS. In contrast to the electrostatically actuated device, dielectrophoretic actuation scheme leaves the mechanical resonator intact from any foreign material, which helps to preserve Q. As a result, fQ product of 6.8 $\times 10^{12}$ has been demonstrated, for 260nm wide, 4 μm long cantilever (Fig. 2(a)) whose resonance frequency is approximately 18.3 MHz [14].

Although dielectrophoretic actuation has the above benefits, it is not ideal for static actuation due to its small force for a given voltage compared to electrostatic actuation. Therefore, electrostatic actuation is appealing for applications where high static strain is desired. To apply significant strain, application of high voltage and large deflection is required. However, a few fundamental and practical limitations need to be considered. First of all, as is always the case for the electrostatic actuation, pull-in instability occurs when the distance between two electrodes reaches a certain threshold. Pull-in instability can be simulated by



Figure 5: (a) Illustrations of the device with electrostatic actuation when 120 V is applied to $30\mu m \log 1.4\mu m$ wide diamond cantilever. (b) Simulated strain applied to color centers near the clamp of the cantilever. Turnaround points in the graph represent pull-in instabilities.

using finite element method (FEM) and solving the inverse problem - i.e. for the given structure, voltage required to reach a target displacement is calculated. In Fig. 5(a), a 3D cartoon of the simulation is shown to illustrate the concept of the device. To have 150nm tip displacement of 1.4µm wide, 830nm thick and 30µm long diamond cantilever with the triangular cross section, approximately 120V needs to be applied to this system. By sweeping target displacement and calculating corresponding voltage, displacement as a function of voltage can be plotted. Typically, pull-in instability manifests itself as a turnaround point in the plot. The FEM simulation gives strain tensor inside the cantilever as well, and it is a monotonically increasing function of the tip displacement. Such a plot is shown in Fig. 5(b). The device cross-section is the same as the one in Fig. 5(a), and the three different lengths are simulated. Before pull-in instability, longer (floppier) cantilevers give larger strain than shorter (stiffer) ones for a given voltage. When the voltage is increased, however, longer beams reach the pull-in instability at a smaller applied voltage. Thus, assuming that high enough dc voltage can be applied, shorter beams are desirable as they result in much larger achievable strain.

In reality, however, the large applied voltage and resulting electric field can lead to breakdown and a surge in current. This phenomenon is well characterized by the Paschen curve which expresses breakdown voltage as a function of pressure and the gap product [19]. It was explained that breakdown is a result of avalanche ionization of atoms between the gap caused by accelerated electrons [20]. However, in nanoscale devices, where the mean free path of the electron is larger than the gap, avalanche ionization is likely not the breakdown mechanism. Under this condition, if the electric field is strong enough to overcome the work function of the metal, it can directly pull out electrons through a process called field emission. In practice, the deposited film is not entirely smooth and hence field emission induced current can locally vaporize the metal, due to intensified heat [21].

In addition to breakdown due to the above phenomena, nonzero surface current on the diamond and resultant destruction of the metal need to be considered. Although diamond is an excellent insulator, a non-zero current through this device (ranging from < nA to hundreds of μ A depending on the conditions) is always measured in our experiment. Typically, for many insulators, moisture adsorption on the surface and resulting electrochemical migration causes a small current [22]. At an applied voltage of 50 V or below, this current is relatively small (on the order of pA) and does not destroy nano-scale electrodes. However, at a higher voltage, on the order of hundreds of volts, current can grow fast and eventually cause fusing or bursting of small electrodes [23]. These two limiting factors, namely dielectric breakdown, and surface currents dictate the practical upper limit on the dc voltage used in these devices. Within these limits, one can use pull-in instability simulation results to design the dimensions of the cantilever that maximizes the applied strain.

Another point worthy of the discussion is the stress caused by metal film deposition. In principle, when a thin film is deposited on the substrate, it may have compressive or tensile residual stress depending on various factors such as film thickness, substrate temperature. Although the residual stress from the thin film can complicate the strain-tuning experiments with color centers and compromise the strength of the applied strain, its adverse effects can be minimized by using ultra-low film thickness at the cost of additional processing layers. In contrast, it can even aid the experiment if the system is cleverly designed [24].

CONCLUSIONS

Summary

In summary, we have demonstrated a SCD fabrication scheme for MEMS/NEMS devices by combining angled-etching and the conventional lift-off processes. MEMS/NEMS devices compatible with color center experiments with different requirements are realized with careful design of electrode patterns. In particular, dynamic actuation is demonstrated via dielectrophoretic forces, and static actuation is demonstrated via electrostatic forces. Dielectrophoretic actuation is useful to maintain high fQ product in small scale devices, while electrostatic actuation is suitable for generation of high static strain. We show the application of static strain generation in SiV electronic level tuning, and discuss the design of this MEMS device within fundamental and practical limits.

Outlook

Our fabrications method can be potentially applied to make nanodevices in materials that lack high-quality thin film technology. For example, we have demonstrated angled-etching in quartz substrate as a proof of the concept, since its piezoelectric and electro-optical properties can be useful [9]. Furthermore, it may be applied to other popular materials with high-quality color centers, such as 4H-SiC.

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SYSTEMATIC DESIGN OF MEMS RESONATORS FOR OPTIMAL NONLINEAR DYNAMIC RESPONSE

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ABSTRACT

We report on the first experimental demonstration of an effective and systematic control over the mechanical contribution to the Duffing nonlinearity in microelectromechanical (MEMS) resonators using nonlinear shape optimization methods. A set of microbeams with selected variable geometry profiles optimized for in-plane vibrations was designed and characterized. Experimental results have demonstrated that these shape changes result in more than a three-fold increase and a two-fold reduction in the Duffing nonlinearity due to resonator mid-line stretching. The minimization of the cubic term in the resonator stiffness resulted in a substantial increase of the resonator linear range.

INTRODUCTION

Resonant micro-electromechanical-systems (MEMS) have grown in popularity over the past several decades due to their numerous applications including stable frequency generation and timing [1-2], robust mass and angular rate sensing [3-4], precise signal filtering [5-6], and energy harvesting [7]. While the majority of MEMS resonators are designed to operate in their linear dynamic range, research has shown that utilizing nonlinearity in resonant MEMS sensors can significantly improve the performance of these systems in some of the applications mentioned above [1,8]. Nonlinear stiffness effects in MEMS resonators commonly arise from finite deformations that lead to nonlinear strain-displacement relationships [9], and/or the nonlinear nature of electrostatic forces in capacitive MEMS [4-6, 10-11].

The ability to control the nonlinearity in resonant MEMS allows one to design devices based on their applications with greater control over their dynamical responses. It has been shown that one can independently tune the linear and/or cubic stiffness coefficients due to the electrostatic potential of the capacitive drive in order to relax the constraints in mode mismatch in MEMS gyroscopes due to fabrications errors [4,12], to achieve optimal drive conditions for micro resonators [13], and to enhance their dynamic range [14]. On the other hand, Dou et al. manipulated geometric contributions in the nonlinear resonator stiffness parameters by performing the shape optimization of the thickness profile in different MEMS resonators to achieve targeted nonlinear coefficients using a gradient-based optimization method [15-16]. The numerical results in [15,17] showed that removing/adding the material (by changing the cross-sectional area) from the points where the slope of the resonator mode shape is maximal results in continuous decrease/increase of the geometric contribution to the resonator cubic nonlinearity. This effect becomes more

pronounced as the difference in the beam thickness increases. We followed the shape optimization approach described in [15] in order to manipulate the nonlinearity in a set of clampedclamped microbeams, and selected a few designs with geometries corresponding to different stages of the resonator shape optimization process. The beams were fabricated in single-crystal silicon using standard SOI (silicon-on-insulator) processing techniques with deep reactive ion etching and released with HF vapor. Figure 1 shows SEM images and COMSOL models of three of representative designs of the clamped-clamped beams under study: a beam with uniform thickness (Beam initial) and beams designed to minimize/maximize the cubic nonlinearity (Beam min/Beam max). The resonant frequencies of the beams range from 80 to 200 kHz.



Figure 1: SEM images and COMSOL models of three representative clamped-clamped microbeams under study. "Beam_initial" is the beam with uniform thickness, while "Beam_min"/"Beam_max" are designed to minimize/maximize the resonator Duffing nonlinearity. Beam dimensions are 500 um (length) by 20 um (out-of-plane thickness) by $2\sim6 \mu m$ (inplane thickness).

THEORY

The dynamic response of a clamped-clamped microbeam performing in-plane flexural vibrations can be modeled by the standard Duffing equation with positive cubic stiffness coefficient [18-19] as follows:

$$\ddot{x} + Q^{-1}\omega_0 \dot{x} + \omega_0^2 x (1 + \gamma x^2) = f \cos(\omega t) \tag{1}$$

where ω_0 is the natural frequency of the beam, Q is the quality factor, γ is the coefficient of the cubic nonlinear stiffness term (Duffing nonlinearity), and f and ω are the drive amplitude and frequency. The critical vibration amplitude (a_{nl}) at which

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nonlinear effects result in a qualitative change of the resonator response, specifically, at which the frequency response transitions from being single-valued to multi-valued, is given by:

$$a_{nl} = \left(\frac{64}{27}\right)^{1/4} \frac{1}{\sqrt{Q\gamma}},\tag{2}$$

which shows that one can maximize the resonator critical vibration amplitude by reducing the Duffing nonlinearity γ [18]. Once the system response enters its nonlinear region, the relationship between the nonlinear resonance amplitude (a_p) and frequency (ω_p) can be written as:

$$\omega_p(a_p) = \omega_0 (1 + \frac{3}{8}\gamma a_p^2) \tag{3}$$

Equation (3) provides the so-called Duffing backbone curve [19] that characterizes the system nonlinearity in the form of amplitude-frequency dependence. In order to compare the results of the shape optimization method on the Duffing nonlinearity of the system, we use the effective Duffing nonlinearity $\gamma_{eff} = \frac{3}{8}\gamma$ as the objective function to be minimized/maximized.

EXPERIMENTS

In the experiment, the beams were externally actuated by a shear piezoelectric stack actuator to achieve in-plane excitation. The in-plane motion of each beam was detected using a Laser Doppler Vibrometer by tilting the microbeams at 45° angle with the laser focused on the sidewalls. The actual in-plane velocity of the microbeam can be calculated from simple geometry. This



Figure 3: Amplitude-frequency responses of microbeams exhibit hardening behavior. The drive voltages for the three beams from top to bottom are: 0.2:0.1:1.4 V, 0.7:0.5:4.7 V and 0.2:0.4:4.0 V respectively.

arrangement allowed characterization of the mechanical nonlinearity isolated from electrostatic effects. Amplitudefrequency responses for each beam were obtained at multiple drive amplitudes in atmospheric pressure from a spectrum analyzer. The schematic in Figure 2 depicts the experimental setup.



Figure 2: Experimental setup schematic. Microbeams are actuated externally by a shear piezoelectric stack. The in-plane responses are measured with a Laser Doppler Vibrometer (LDV, Polytec MSA-400) by tilting the beams at 45° with the laser focused on the sidewalls. The amplitude-frequency response curves are obtained from the spectrum analyzer (HP 88410A).

RESULTS

The amplitude-frequency responses for the three representative beams shown in Figure 1 are plotted in Figure 3. As the driving amplitude increases, the amplitude-frequency response of each microbeam exhibits hardening behavior, as expected from a positive Duffing nonlinearity due to the midline stretching effects. Once the system response enters its



Figure 4: Amplitude-frequency characteristics of three representative microbeams. Solid, dotted and dashed curves represent least-square fitting of experimental results for "beam_initial", "beam_min" (final), and "beam_max" (final) respectively.
Table 1: Characterization results for microbeams under study. Data shows clear trends of the Duffing nonlinearity towards its minimum/maximum values depending on the beam geometry profile. The discrepancy between numerical and experimental results for the beams designed to maximize γ_{eff} is expected due to abrupt changes in the beam thickness profile, which causes errors in the numerical algorithm utilizing simple beam elements. In contrast, the relative error between numerical and experimental results for the beams designed to minimize γ_{eff} is 8% or less due to comparatively smooth beam profiles.

D. I	COMSOL Results	Numerical Results (following techniques in [15])			Experimental Results (from least-square curve fits)		
Beam type	$\frac{\omega_o}{2\pi}$, kHz	$\frac{\omega_0}{2\pi}$, kHz	$\gamma_{eff}, \mu m^{-2}$	$\frac{\gamma_{eff}}{\gamma_{eff,0}}$	$\frac{\omega_o}{2\pi}$, kHz	$\gamma_{eff}, \mu m^{-2}$	$\frac{\gamma_{eff}}{\gamma_{eff,0}}$
Beam_initial	135.0	135.1	0.0165	1	134.5	0.0188	1
Beam_min (5 th iteration)	145.7	148.7	0.0117	0.712	145.0	0.0129	0.699
Beam_min (Final)	191.9	189.4	0.0059	0.357	190.9	0.0072	0.388
Beam_max (20 th iteration)	86.8	85.8	0.0844	5.111	91.8	0.0472	2.558
Beam_max (Final)	79.9	70.5	0.1248	7.559	86.1	0.0569	3.309

bistable regime, the maximum nonlinear response amplitude is only a function of its corresponding frequency, as described by equation (3). Using this result, we extracted the peak amplitude from Figure 3 and plotted the frequency as a function of the peak amplitude (a_p) , followed by curve fitting to obtain the natural frequency and cubic nonlinearity. Quadratic curve fitting is performed to the data using $\omega(a_p) = a(1 + ba_p^2)$, where the fitting coefficients "a" and "b" correspond to the beam natural frequency ω_0 and the effective Duffing nonlinearity γ_{eff} After the coefficients are determined, we respectively. normalized the frequency of each beam design to its natural frequency for direct comparison of the three representative beams (Figure 4). We further compared the experimental results to the numerical simulations following the process in [15]. Table 1 summarizes the comparisons between the beam designs, including the intermediate ones.

DISCUSSION

We compared the values of γ_{eff} for beams at different stages of the optimization iteration process with the corresponding Duffing nonlinearity of the nominal (uniform) beam, $\gamma_{eff,0}$; the results are summarized in Table 1. These characterization results show that the resonator shape optimization results in the natural frequency varying from its nominal, uniform beam, value. They also demonstrate that the designs to minimize nonlinearity reduce the Duffing nonlinearity by a factor of 2.6 when compared to the initial uniform beam, which corresponds to a 1.6 times increase in the critical vibration amplitude at which the resonator becomes nonlinear [18]. Meanwhile, the beam design with maximum nonlinearity achieves a 3.3 times increase in the Duffing nonlinearity compared to the uniform beam. Further, we performed numerical simulations on the same beam geometries using the optimization techniques described in [15]. When compared the experimental results to the numerical predictions, the minimization of γ_{eff} agrees with the simulations within 8%. A larger discrepancy occurs in the maximization of γ_{eff} , however, the trend qualitatively agrees with the numerical results. The difference between numerical and experimental results for the beams designed to maximize γ_{eff} is due to the abrupt changes in the beam thickness profile, which causes errors in the numerical algorithm, which utilizes

simple beam elements. In contrast, beams designed to minimize γ_{eff} results in relatively small errors due to the relatively smooth beam profiles.

CONCLUSION

The experimental results shown in this work are in good agreement with numerical predictions [15] for the important case of minimizing the nonlinearity, and they demonstrate that one can successfully utilize shape optimization methods for adjusting the resonator nonlinearity in a well-controlled manner. The ability of reducing the Duffing nonlinearity is especially useful in increasing the linear dynamical range of resonators, which plays a pivotal role in reducing phase noise in MEMS oscillators and for increasing the signal to noise ratio in resonant sensors [20]. Finally, our results provide strong confidence that shape optimization methods can be applied in the design of nonlinear MEMS resonators with more complicated geometries, with multi-physics effects, and for different nonlinear parameters, including hard-to-control modal coupling coefficients [15].

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THE EFFECT OF CRYSTALLINE ORIENTATION ON VIBRATION SENSITIVITY OF SILICON MICRO-RESONATORS

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ABSTRACT

In this work the effect of crystalline orientation on the acceleration sensitivity of Silicon-based MEMS oscillators is experimentally studied for the first time. The thin-film piezoelectric-on-Silicon (TPoS) platform is utilized to implement the oscillators as it enables resonators with low-motional resistance and high O. A single lateral-extensional-mode resonator design is fabricated in <100> and <110> orientations on a <100> Silicon wafer. The resonators are then used to assemble two oscillators operating at ~25MHz and ~27MHz respectively. The average acceleration sensitivity of the oscillator containing the <110> resonator is measured to be $\sim 4 \times 10^{-10}$ at vibration frequencies up to 2700 Hz; an astonishing two orders of magnitude lower than that of the oscillator utilizing the <100> resonator. The acceleration sensitivity in these Silicon-based resonators is believed to stem from nonlinear elastic properties of Silicon, which is dependent on crystalline orientation as well as doping type/concentration. The Silicon substrate used in this work is Phosphorous-doped at ~5e19 cm⁻³ concentration.

INTRODUCTION

High-performance ultra-stable oscillators are of great interest in wireless communication applications. Frequency stability of the oscillators that utilize mechanical resonators can be affected by the environment vibrations especially in harsh mobile systems like radars and cell-phone towers. Therefore, acceleration sensitivity is an important characteristic of the resonator chosen for such applications.

The acceleration sensitivity of quartz resonators and their dependency on the crystalline orientation have been extensively studied [1]. With the emergence of micromachined Silicon resonators as a viable alternative to quartz in applications traditionally dominated by quartz, a thorough study of vibration sensitivity in Silicon resonators is in order. Some attention have been recently directed toward studying the acceleration sensitivity of the Silicon-based MEMS resonators [2-6] but the role crystalline orientation could play in the outcome has yet to be considered.

In general, the level of acceleration sensitivity in a resonator depends on several factors such as the material chosen for the resonant body, dimensions of the resonator, resonance mode, and anchor arrangement [7]. Most studies exploring the effect in microresonators have focused on strategies to reduce the vibration sensitivity in capacitively-transduced resonators. The dominant source of acceleration sensitivity in such resonators appear to be the variation of electrostatic stiffness (i.e. gap size) as a result of vibration. The reduction of sensitivity have been explored through strategically located anchors [2-5] and increasing the capacitive gap size [6].

Although capacitive resonators offer excellent quality factor, thin-film piezoelectric-on-Silicon (TPoS) resonators have been shown to exhibit significantly lower motional resistance and nonlinearity without excessively sacrificing the Q [8]. Therefore, TPoS resonators are believed to offer competitive advantages compared to capacitive resonators for oscillator applications. In TPoS resonators and any class of MEMS resonators that don't operate based on capacitive transduction the acceleration sensitivity is yet to be carefully characterized and understood.

In this work, the effect of the Silicon crystalline orientation on the resonator vibration sensitivity is investigated. For this purpose TPOS resonators are fabricated in the two commonly accessible Silicon crystalline orientations: <100> and <110>. The measured results of this work suggest that the acceleration sensitivity of bulk mode resonators oriented in <110> direction is about two orders of magnitude lower than that of resonators oriented in the <100> direction. It should be noted that the acceleration sensitivity in such resonators is believed to be a function of the nonlinear elastic properties of Silicon. As shown by other work presented in the past including one published by our group [9], nonlinearity in Silicon is a sensitive function of doping type/concentration. Consequently the results of this work cannot be generalized for all Silicon resonators, and rather similar studies need to be performed for a specific doping type/concertation. The Silicon substrate used in this work is doped with Phosphorous at ~5e19 cm⁻³ concertation in order to improve the temperature stability as shown by our group in earlier studies [10].

The paper is organized as follows. First, the acceleration sensitivity of the resonator is mathematically described. Then, the resonators used in this study are characterized using a network analyzer before the experimental set up for measuring the resonators vibration sensitivity is presented. Finally, the measured acceleration sensitivity results are presented and discussed.

ACCELERATION SENSITIVITY

The resonance frequency of a resonator can change due to the strain perturbation induced by external acceleration. The value of this shift depends on the vibration magnitude, its direction and acceleration sensitivity of the resonator. Thus, the resonance frequency during vibration is given by [1]

$$f(a) = f_0(1 + \Gamma . a)$$
(1)

where f_0 is the natural resonance frequency of the resonator, Γ is the acceleration sensitivity vector and a is the applied acceleration. Based on (1), Γ can be found by

$$\Gamma = \Delta f / f_0 a \tag{2}$$

where Δf is the resonance frequency shift, which is usually a small value for resonators with low vibration sensitivity complicating the characterization efforts. One reasonable approach for measuring Γ is to employ the resonator in an oscillator circuit and monitor the output frequency spectrum while an alternating external acceleration is applied to the oscillator. Let's assume the applied acceleration is:

$$\vec{a}(t) = A\cos(2\pi f_{v}t) \tag{3}$$

where A is the maximum magnitude of the acceleration and f_v is the vibration frequency. In this case the resonance frequency of the resonator should vary as follows:

$$\vec{f(a)} = f_0(1 + (\vec{\Gamma}.\vec{A})\cos(2\pi f_v))$$
 (4)

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Solid-State Sensors, Actuators and Microsystems Workshop Hilton Head Island, South Carolina, June 5-9, 2016 On the other hand, consider the output signal of an oscillator as:

$$V(t) = V_0 \cos(\varphi(t)) \tag{5}$$

Where $\varphi(t)$ is the phase of the circuit and is obtained by

$$\varphi(t) = 2\pi \int_{t_0}^{t} f(t')dt' \tag{6}$$

By substituting (4) in (6), the oscillator phase can be obtained as

$$\varphi(t) = 2\pi f_0 t + (\Delta f / f_v) \sin(2\pi f_v t) \tag{7}$$

Thus, the oscillator output signal will be

$$V(t) = V_0 \cos(2\pi f_0 t + (\Delta f / f_v) \sin(2\pi f_v t))$$
(8)

Equation (8) indicates a frequency modulated signal at the oscillator output. By using the Bessel series to expand (8), The voltage can be expressed as

$$V(t) = V_0 [J_0(\beta) \cos(2\pi f_0 t) + J_1(\beta) \cos(2\pi (f_0 \pm f_v)t) + J_2(\beta) \cos(2\pi (f_0 \pm 2f_v)t)) + ...]$$
(9)

In which $\beta = \Delta f/f_v = (T.A)f_0/f_v$ is the modulation index. For $\beta < 0.1$, $J_0(\beta) = 1$, $J_1(\beta) = \beta/2$, and for n>1 J_n(β)=0. Thus, in the oscillator output spectrum, there will be two sidebands at $f_0 \pm f_v$ besides the carrier signal at f₀. The ratio of the sidebands power to the carrier power can be derived by

$$L_{v} = 20 \log(J_{1}(\beta) / J_{0}(\beta)) = 20 \log((\vec{\Gamma}.\vec{A})f_{0} / (2f_{v}))$$
(10)

By rewriting (9), the acceleration sensitivity is obtained by

$$\Gamma = (2f_{\nu} / Af_{0})10^{L_{\nu}} / 20$$
(11)

The acceleration sensitivity in oscillators that contain piezoelectric-based resonators is believed to stem from: 1) deformation of the resonator due to external forces (geometrical effects) 2) the nonlinear behavior of the material elastic properties with the latter being usually the dominant factor [7]. The external acceleration applied to the resonator acts on the resonator mass and generates a force that causes stress/strain in the device, which can either change the resonator geometry and/or the material stiffness if the elastic behavior of the material is nonlinear. As is shown by complicated theoretical expressions in [7] for bulk acoustic wave (BAW) micro-resonators the acceleration sensitivity is proportional to a complex sum of linear and nonlinear elastic stiffness coefficients. In addition, Γ is inversely proportional to the linear stiffness coefficient of the material. Hence, a material with large linear elastic stiffness and low nonlinear elastic stiffness coefficients, can exhibit low sensitivity to external vibration.

Based on this concept, since the elastic behavior of single crystal Silicon is orientation-dependent, the acceleration sensitivity of resonators oriented to different crystalline planes is expected to be orientation-dependent as well.

DEVICES CHARACTERIZAION

To explore the effect of the crystalline orientation on acceleration sensitivity, two similar devices in terms of dimensions and design have been fabricated in two different crystalline orientations: <110> and <100>. The resonators are two-port devices with an interdigitated top metal pattern that could excite both the fundamental and the third harmonic lateral-extensional modes. The resonators are fabricated on an ~8µm Phosphorusdoped (doping concertation ~ 5×10^{19} cm⁻³) silicon-on-insulator (SOI) substrate with an stack of Mo/AlN/Mo (100nm/1000nm/100nm) deposited for the piezoelectric transduction mechanism [10]. Note that the substrate in this study is a <100> SOI wafer, so the resonators aligned in parallel with the wafer flat are oriented in the <110> direction, while the ones at a 45° angle with respect to the flat are oriented to the <100> plane. The relative layout of the resonators on the wafer are shown in Figure 1. In addition, the fundamental lateral-extensional mode shape of the <100> TPOS resonator simulated by COMSOL and the direction of applied acceleration (a_z) are shown in the inset diagram.

The frequency response, measured by a Rohde & Schwarz ZNB 8 Network Analyzer, and scanning electron micrograph (SEM) of the two resonators used in this study are shown in Fig. 2. The response at fundamental resonance mode of the resonators is presented since that will be the mode utilized in the oscillator. As seen, the resonance frequency of the device in <110> direction is higher than the one in <100> direction because of the larger Silicon Young's modulus in <110> orientation. Quality factor and motional resistance of <100> and <110> resonators are 4.639 K, 322.18 Ω and 9.975 K, 236.97 Ω respectively.



Figure 1: The layout of <100> and <110> resonators relative to the wafer and the simulated mode shape of the <100> resonator.



Figure 2: Frequency response and SEM of fabricated resonators in <100> and <110> orientation.

EXPERIMENTAL RESULTS

In order to measure the acceleration sensitivity of the resonators using equation (11), they should operate in an oscillator circuit. Therefore, two identical printed circuit boards (PCB) have been assembled. The oscillator circuit is based on a CF5027 IC module commonly used in commercial quartz oscillators. The CF5027 IC is mounted on a ceramic leadless chip carrier (LCC) package and the resonator has been connected to it in a one-port configuration to keep the phase of the oscillator loop equal to 0°. Care has been taken to reduce the number of electronic components, the size of the board, and the length of the wirebonds in order to minimize the parasitic effects.

A Rohde & Schwarz FSUP Signal Source Analyzer is used to capture the oscillators phase noise plot. Both oscillators exhibit excellent phase noise performance with the <110> oscillator having an slightly better close-to-carrier performance (Fig. 3) due to the higher Q (-125 vs. -120dBc/Hz (*a*) 1kHz offset).



Figure 3. Oscillator phase noise for both resonators.

The vibration measurement setup is a closed-loop system including a magnetic motor, a pa-138 amplifier, a VR9500 controller and a DYTRAN 3055B1T accelerometer from Vibration Research Corporation. The accelerometer is mounted on the oscillator board to measure the actual acceleration that is fed back to the controller. Then the controller compares the measured acceleration of the board with the desired value of acceleration, which has been set by the operator, and provides a proper signal for the amplifier. Based on this signal the amplifier generates the required gain for the magnetic motor to reach the desired displacement and acceleration. The PCB of the oscillator and the vibration system used in this work are shown in Figures 4 and 5 respectively.



Figure 4. The oscillator board



Figure 5. The vibration sensitivity measurement setup.

Using this setup, a 6g 1.5 kHz sine-wave vibration in zdirection, normal to the Silicon surface (see inset diagram in Fig. 1), has been applied to the resonators. The frequency spectrum of the two oscillator output signals, captured by the signal source analyzer is demonstrated in Fig. 6. As seen, the same acceleration has generated much smaller side-bands in the spectrum of the <110> device compared with the <100> resonator. Using (11), acceleration sensitivity in z-direction is calculated as $\Gamma_{Z,<100>}=3.3*10^{-8}$ and $\Gamma_{Z,<110>}=5*10^{-10}$ in <100> and <110> directions, respectively.



Figure 6. Oscillator output spectrum for <100> (top) and <110> (bottom) resonators under a 6g 1.5kHz vibration.

The acceleration magnitude and frequency is then kept constant (7g and 2 kHz) to obtain the frequency and amplitude dependency of the acceleration sensitivity (Fig. 7 and Fig. 8, respectively). As seen, for different vibration magnitudes and

frequencies, the resonator in <110> direction continues to exhibit two orders of magnitude less sensitivity to external vibration in comparison with <100> resonator.



Figure 7. Acceleration sensitivity versus vibration frequency



Figure 8. Acceleration sensitivity versus acceleration magnitude

RESULTS DISCUSSION

Based on the observations of this work, the acceleration sensitivity of a resonator made of highly-doped n-type silicon substrate is much less if it is oriented to the <110> crystalline plane compared to one oriented to the <100> plane. Considering that the acceleration sensitivity is believed to be dominated by the nonlinear elastic properties of the resonant body as described earlier in this paper, the results of this work are in agreement with the previous reports on nonlinearity of bulk-mode silicon resonators aligned to different crystalline orientations [11]. As reported in [11], the nonlinear behavior of bulk-mode resonators depends on the doping level, dopant type, mode-shape and crystalline orientation. Moreover, a length extensional mode resonator with n-type Phosphorous doped silicon substrate, devices aligned to <110> plane exhibit lower nonlinearity compared with the devices aligned to <100> plane. Hence the reported results of this work are qualitatively anticipated.

CONCLUSION

In this paper, the acceleration sensitivity of silicon-based microresonators aligned to different Silicon crystalline orientation is investigated. Two identical resonators are fabricated on a highlydoped n-type silicon wafer aligned to <100> and <110> crystalline planes. These resonators are employed in two identical oscillator circuits in order to measure their acceleration sensitivity. By applying a sine-wave vibration and measuring the oscillator output power at the resonance frequency and sidebands at the vibration frequency offset, the vibration sensitivity of the resonators were characterized. The obtained results indicate that the resonator aligned to <100> direction is more sensitive to external vibration than the one aligned to <110> direction. Since the dominant source of acceleration sensitivity in piezoelectric resonators is the nonlinear elastic properties of material, our observations confirm that nonlinear stiffness coefficients in <100> orientation are larger than that of <110> orientation in highly n-type-doped Silicon. Note that this result may be different for a different doping type/concentration since Silicon nonlinearity depends on these parameters.

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3D SELF-ASSEMBLED MICROSCALE RESONATOR AS ULTRA SENSITIVE ISOTROPIC SENSOR

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ABSTRACT

Sensors based on split-ring resonators have long been explored as viable candidates for biological applications since their frequency is a function of the permittivity of surrounding media; plagued by their detection ability being confined to the gap, they demonstrate a low sensitivity. In addition, the high directionality of their response has been a major limiting factor in the advancement of these resonant structures, which renders the spectrum incomprehensible. Thus, this study demonstrates the use of origami like self-folding to achieve a three-dimensionally coupled novel split-ring resonator which is invariant under any rotation and has ultra-high sensitivity.

INTRODUCTION

Pendry's design of a split-ring resonator array presented a technique for the realization of left-handed metamaterial [1]. The resonant frequency of these resonators is a function of their length, split size, angle of incidence of light (θ) (Fig. 1), and the permittivity of the surroundings. The resonance dependence on the permittivity of the surroundings allows their use as biological sensors which can predict changes in the cell life cycle due to changes in cell permittivity as a result of chemical reactions within the cell membrane [2]. However, the detection ability of the splitring resonator is limited only to the split in the resonant structure. which is much smaller than the total length of the resonator [3]. Furthermore, for their efficient function as a sensor it is necessary that any changes in the transmission spectrum be solely dependent on the changes in permittivity. But, the C-shape had a major disadvantage due to the high dimensionality of its response; as the resonator was rotated, the resonance shifted from magnetic to electrical (Fig. 1), creating an ambiguity in the signal transduced. If the same resonator is now forced to couple isotropically in a three-dimensional (3D) configuration, then each resonator experiences the same exact surroundings as its neighboring resonators; causing the entire structure to demonstrate a very strong resonance at a single frequency. Changes in the resonant frequency of any one of the resonators has a domino effect causing amplification in the transduced signal.

ANALYSIS AND RESULTS

The 3D folded structure with 3D star resonators (Fig. 2a, b), demonstrated a single fundamental resonant frequency. At the resonant frequency, the cubic structure did not show any changes in transmission on rotating along any/all three axis (fig 2c), as opposed to its 2D counterpart which had a highly dimensional response. The surface current of the cubic structure at the resonant frequency demonstrated a smooth and equal surface current across all the resonators, proving that it is the 3D coupling that is inducing the isotropic response in the cubic structure.

The 3D coupling of the resonator, not only increases the isotropy of the split-ring resonator, it also magnifies its sensitivity. Since each resonator induces a resonance in its neighboring resonators, a local permittivity change experienced by any one of the resonators has a ripple effect that enhances the transduced change in the transmission spectrum. The isotropic 3D star resonator thus has an ultra-high sensitivity which is 10 times higher than its two dimensional counterpart (Fig 2d). Through the

use of an origami self-folding technique [4], the resonators patterned on a 20 μ m thick substrate could be folded into a 200 μ m sized cubic structure through the use of surface tension force caused by the reflow of a polymer hinge on heating (Fig 2b).

The 3D star shaped split-ring resonator patterned on the face of 3D cubic substrate demonstrates an isotropic ultra-high sensitivity resonant structure. The isotropy of the response and the ripple effect on resonance of the structure make it an excellent candidate for use in high sensitivity chemical and biological applications.



Figure 1: Simulated transmission characteristics of a 2D C-shaped resonator. Demonstrating the directional transmission response shifting from magnetic (1st mode) to electrical resonance (2nd mode) as a result of varying angle of incidence(θ) from 0° to 90°.



Figure 2: (a) Illustration of isotropic 3D star resonator (b) Optical images of the 3D resonator. (c)Simulated transmission response of the cube showing an isotropic behavior, (d) Resonant frequency changes of 2D and 3D resonators.

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A PRELIMINARY STUDY OF AN ELECTROSTATIC CURVED BEAM ACTUATOR FOR A BIO-MEMS FORCE SENSOR

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ABSTRACT

An electrostatic curved beam microactuator embedded in a microfluidic device meant to be a probe to measure the mechanical properties of biological cells is modeled, fabricated, and evaluated. Our study in aqueous media showed that an actuation voltage of 8 V is sufficient to generate an intended force of up to 97 μ N and achieve a static displacement of up to 9.7 μ m - these values demonstrate that we should be able to deform a cell with the appropriate force and deformation strain.

INTRODUCTION

Electrostatic curved actuators are known to achieve large displacement and large force compared to comb drives and parallel plates for a given geometric footprint and fixed actuation voltage [1], [2]. This makes them a suitable candidate for force generation where actuation voltages are constrained to be low, such as in an aqueous environment where hydrolysis limits actuation voltage to a range of ± 8 V. The actuator is designed to be used as a probe to measure the mechanical properties of cells as they transit through a microfluidic network [3], [4].

THEORY

The curved actuator comprises of three main components: beam electrode, set of curved electrodes, and an insulator film (Fig. 1). The curved electrodes are rigid while the beam electrode is a compliant structure with both ends clamped to anchors. The beam and curved electrodes are immersed in a dielectric medium. Upon the application of a voltage to the electrodes, the beam electrode zips into the curved electrodes. The insulator film on the electrodes prevents electric shorting during the zipping process.

Deflection profiles as a function of voltage are predicted using the *Rayleigh-Ritz* method [5] applied to a first-principles model of the actuator. The electrode gap (Fig. 2), out of plane deflections, and fringe field effects are neglected for simplicity. Since the actuator is symmetric about the line of symmetry, only the left-half portion of the actuator is modeled by employing a symmetry condition. In the model, a trial function for displacement w_L is assumed in terms of electrode geometry and unknown quantities. The unknown quantities are solved by minimizing the actuator's total potential energy π defined by Equation (1) with respect to the unknown quantities using MATLAB® function minimization solver *fmincon*.

$$\pi = U_s + U_{el} + U_{u} \tag{1}$$

In Equation (1), U_s is spring energy, U_{el} is electrostatic potential energy, and U_M is the moment work by the right-half portion of the actuator. Upon solving the unknown quantities, the static displacement and beam electrode shape function are determined. Critical actuator dimensions are given in Table 1.

EXPERIMENT

To validate deflection predictions, we fabricated the curved actuator from a silicon-on-glass wafer using standard lithographic micromachining methods. For our experiments, the actuator is submerged in deionized water and a square wave at 500 kHz with a pulse height of a variable \overline{V} is applied. Displacement w_L is measured using an inverted microscope equipped with a camera and image processing software.

RESULTS AND CONCLUSION

The experiment and model predictions are in good agreement despite using the simplifying assumption that the electrode gap is zero (Fig. 3). This simplifying assumption causes the model to overpredict displacement at low voltages, where the finite electrode gap required for fabrication (~2.5 μ m) causes the moving beam to be not fully zipped in and thus a lower actuation force is applied (Fig. 4). The idealized zipping motion as a function of voltage is shown in Fig. 5. Experimental results in an aqueous media demonstrate that a voltage $\overline{V} = 8$ V is sufficient to generate an intended force of 97 μ N and achieve a displacement of 9.7 μ m (Fig. 3) – these values demonstrate that we should be able to deform a cell with the appropriate force and deformation strain. Future work will study the dynamic behavior of this actuator and use the actuator to probe cell mechanical properties by pairing the actuator with a sensor unit.



Figure 1: (A) Schematic of the curved actuator in the unenergized and energized states. (B) SEM image of the actuator.

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Figure 2: Schematic of the left half of the actuator in the energized state. The beam electrode is seen partly zipped into the curved electrode with displacement w_L . Here, M designates the moment or torque by right half of the actuator on the left half.



Figure 3: Static displacement in an aqueous environment. There is a good match between experiment and model for higher voltages. However, the model is less accurate at low voltages because the simplified model omits a gap between the actuators at the base of the beam, which is required to electrically isolate the two electrodes.



Figure 4: Experiment images demonstrating the actuator zipping action for square wave at 500 kHz with different voltage amplitudes (A) $\overline{V} = 0$, (B) $\overline{V} = 6$, and (C) $\overline{V} = 8 \text{ V}$, respectively. As voltage \overline{V} is increased the zipping length increases and the initial unzipped length decreases.



Figure 5: Prediction of the actuator zipping as the actuation voltage \overline{V} is increased. The solid black line is the curved actuator profile s(x), the dashed lines are the beam electrode deflection profiles at different voltages \overline{V} .

Table 1: Actuator n	nodel para	meters
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Parameter (unit)	Numerical
	value
Beam electrode half-length (μ m)	1000
Beam electrode height (μ m)	45
Beam electrode thickness (μ m)	6
Insulator film thickness (nm)	20
Reinforcing beam stiffness (N/m)	5
Dielectric constant of insulator film	9
Beam material Young's modulus (GPa)	130
Dielectric constant of deionized water	78

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GALLIUM NITRIDE HIGH-ORDER MODE LAMB-WAVE RESONATORS AND DELAY-LINES

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ABSTRACT

This work reports on theoretical and experimental study of zero-order as well as high-order symmetric and asymmetric modes of Lamb-wave resonators and delay line structures realized in single-crystalline GaN thin films. We investigate the phase velocity, dispersion characteristics, electromechanical coupling (k_t^2) , motional impedance (R_m) and Quality factor (Q) of different Lamb-wave modes (e.g. A_0 , S_0 , A_1 , S_1) propagating in GaN thin films. The dispersion characteristics of these modes in GaN-based thin films are simulated and devices with different pitch sizes are fabricated and tested to compare their performance against simulation. High *frequency*×Q value of 3.6×10^{12} is realized for devices with a pitch size of 4.6 µm, showing a very low motional impedance of R_m = 284 Ω . Depending on the propagation and dispersion characteristics of resonance modes, certain modes are shown to be dominant experimentally (S₀ and A₁). Furthermore, we utilize the resonance modes in a one-port resonator to build two-port delay-line structures. Such delay-line topologies operate based on travelling acoustic waves, since the free boundary (trenches) do not exist in the direction of wave propagation, reflections from the free edges are minimized, thus reducing spurious modes [1]. This work marks the first steps towards building acoustic diodes in GaN based on the "Acousto-electric Effect" [2]. Upon application of DC electric field to the piezoelectric semiconductor transmission media, acoustic wave can be amplified or attenuated depending on the direction of the electric field.

INTRODUCTION

Lamb-wave resonators, mostly realized in AlN and ZnO platforms, have been extensively used for timing and sensing applications over the past decade [3]. However, there have only been few reports on GaN Lamb-wave resonators [4,5], mainly focused on the zero-order resonance modes (S_0 or A_0). Lamb waves propagating in thin piezoelectric films have drawn significant attention since they combine the advantages of bulk acoustic waves (BAW) with surface acoustic waves (SAW); high phase velocities, compact designs and low motional impedances along with multiple frequency excitation defined by inter-digitated transducers (IDTs). In this work, we investigate acoustic properties of different Lamb-wave modes (*e.g.* A_0 , S_0 , A_1 , S_1) propagating in GaN thin films and demonstrate high-performance Lamb-wave GaN resonators with exceptionally low motional impedances.

EXPERIMENTAL RESULTS

Figure 1(a) shows the schematic of a one-port Lamb-wave GaN resonator with 31 IDT fingers and four narrow tethers placed at nodal points. The top AlGaN layer is etched and the contours of the resonators are fully etched with a Cl₂-based plasma recipe. Fig. 1(b) shows the schematic of a two-port delay line topology. The pitch size, finger width, spacing and aperture are 4.6 μ m, 2.3 μ m, 2.3 μ m and 80 μ m in both cases. The fabrication process of such devices is compatible with standard GaN HEMT foundry, with an addition of a final release step to form suspended membranes. Our devices consist of 1.8 um-thick MOCVD-grown GaN on Si (111). More details about the fabrication process are reported in [6].



Figure 1: Schematic of (a) one-port Lamb-wave GaN resonator with Ni IDT fingers, (b) two-port delay-line configuration with two IDT sets. No trench is etched along the wave propagation direction to minimize free-boundary acoustic wave reflections.

Figure 2 shows the dispersion characteristics of A_0 , S_0 , A_1 , and S_1 modes. One-port resonators with pitch sizes of 4, 4.6, 5, and 6 μ m are fabricated and their phase velocity is measured showing a good match with the simulated results.



Figure 2: Dispersion characteristic of A_0 , S_0 , A_1 and S_1 Lambwaves simulated for various GaN thickness to lambda ratios. The phase velocity of four Lamb-wave resonators with various pitch sizes are measured and compared against simulation results. The stack composition is shown in the inset, consisting of AlN, $Al_xGa_{(1-x)}N$ and GaN layers. Since the acoustic properties of the AlGaN transitional layer is unknown, this layer is simulated as a stack of several layers with acoustic properties approximated by a linear combination of that of AlN and GaN.

Figure 3(a) demonstrates the wide-band admittance plot of the one-port Lamb-wave resonator (f < 2.5 GHz), highlighting A_0 , S_0 , A_1 and S_1 modes along with their mode shapes. The simulated frequency response is in very good agreement with the measured result. It is observed that S_0 and A_1 modes are the two dominant modes in our devices showing *frequency* ×*Q* values as high as 3.6×10^{12} . The SEM image of the one-port resonator is shown in the inset with the device dimensions. The effect of capacitive feed-through is de-embedded for S_0 and A_1 modes and shown in Fig. 2(b) and (c) respectively.

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Figure 3: Measured and simulated wide-band admittance response of one-port lamb-wave resonator along with the mode shapes at each frequency of resonance (mode shape shown for one wavelength). An intrinsic quality factor of 1000 is assumed for the COMSOL simulation. Inset shows the SEM image of the fabricated one port resonator. (b) De-embedded admittance response of S_0 mode at 0.905 GHz and (c) A_1 at 1.61 GHz. The frequency $\times Q$ value is 3.6×10^{12} A very low motional impedance of only 284 Ω is extracted for the A_1 mode response.

The dominant resonance modes in the one-port Lamb-wave resonators are used to build two-port Lamb-wave delay line structures. The geometry of the delay line is identical to conventional acoustic delay lines with two pairs of transmit and receive IDT sets and a transmission media (delay line). Figure 4(a) shows the SEM image the two-port delay-line configuration. The input/output impedance of this device is matched to 50 Ω termination using an LC matching network. The frequency response of this device for S₀ and A₁ modes are shown in Fig. 4(b) and (c), respectively.

CONCLUSION & FUTURE WORK

This work reports on the dispersion characteristics of A_0 , S_0 , A_1 and S_1 Lamb-wave resonators. A high Q of 2210 is achieved at 1.6 GHz (for A_0 mode) with a very low motional impedance of 248 Ω . Furthermore, we demonstrated a two-port travelling Lamb-wave delay-line topology with the goal of building acoustic diodes.

As the Lamb-wave propagates through the 2DEG, it induces a travelling sinusoidal potential that causes bunching of carriers and hence a periodic variation in 2DEG electrical conductivity. Upon application of an applied DC field, this periodic conductivity leads to an additional travelling periodic potential that induces lattice distortion, thus amplifying or attenuating the travelling acoustic wave, under certain conditions ($V_{electron} > V_{Lamb-wave}$) [2] (Fig. 5). Such devices mark critical steps towards enabling non-reciprocal acoustic communication.



Figure 4: (a) SEM image of the two-port delay-line structure showing the device dimensions. Frequency response is measured using matching network to match the device impedance to 50 Ω terminations. The delay-line transmission is shown for (b) S₀ mode with a center frequency of 0.93 GHz, and (c) A₁ mode with a center frequency of 1.62 GHz.



Figure 5: Schematic of an acoustic diode realized on AlGaN/GaN material systems, consisting of two sets of transmit (TX) and receive (RX) IDT sets and a delay line or transmission media. The external DC electric field determines the direction of propagation of acoustic wave.

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SHOCK MITIGATED MICRO-ELECTROMECHANICAL SYSTEMS STRUCTURE

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ABSTRACT

We report a simple, yet effective, MEMS structure to mitigate the external mechanical perturbation, such as shock. A conventional single membrane can travel beyond allowed distance to be damaged upon an external shock. The excessive travel can be reduced by having an additional electrode to attract the membrane electrostatically. The added elements, in conjunction with fine control algorithm, mitigate the impact of shock. The dual membrane structure effectively reduces the travel distance by 37 %, upon deploying merely 0.5 V. The dynamic implementation of shock mitigation method, using an on-board accelerometer together, delivered successful *in-situ* mitigation of shock on a dual-membrane MEMS microphone.

INTRODUCTION

Despite many successful MEMS sensors/actuators penetrating in the market, several challenges still exist, including protecting the MEMS devices from the external perturbation. Many theoretical and experimental studies on the response to shock impacts have been explored, yet a small number of prior studies demonstrated the methods of mitigating the external shock impact. One group reported hard shock stops [1], which, tends to generate secondary impacts and causes undesirable device oscillations. Nonlinear springs and soft coatings were suggested by a group in order to improve shock mitigation skills [2]. Another group presented adaptive control to reduce vibration-induced bias errors in inertial sensors [3]. Those were either ineffective or required sophisticated structures and algorithms. In this work, we attempt to mitigate the impact of external shock by adding an electrode to control the excessive movement of a membrane via electrostatic stimuli and restoration force in a dynamic behavior upon the shock.

RESULTS

In a conventional structure, the movable membrane travels beyond allowed distance to be damaged (Figure 1(b&d)) upon an external shock (Figure 1(a)). By adding an additional electrode on top of the membrane to attract the movable membrane, the excessive movement can be effectively reduced, as shown in Figure 1(c&e).



Figure 1: Schematics; (a) Shock load profile; (b) Single movable membrane device; (c) Dual membranes (a movable membrane and an electrode) device; (d) Single membrane device upon shock; (e) Dual membranes device in presence of shock.

We fabricated a fully CMOS-compatible device, using n-doped polysilicon as structural layers and SiO₂ as sacrificial layers, as illustrated in Figure 2.



Figure 2: Process flow of shock-mitigated dual-membrane structure. (a) Deposit Si_3N_4 ; (b) Define substrate contact; (c) Deposit and pattern the 1st sacrificial layer; (d) Deposit Si₃N₄/Polvsilicon/Si₃N₄; Etch 1st (e) membrane(Si₃N₄/Polysilicon/Si₃N₄); (f) Deposit and pattern 2^{nd} 2nd membrane sacrificial layer; Deposit (g) 2nd *membrane(Si₃N₄/Polysilicon);* (h) Etch membrane(Polysilicon/Si₃ N_4); (i) Define and deposit metal pad; (j) Remove sacrificial layers.

Unlike prior studies that demand incorporating sophisticated structures, we added one additional layer to mitigate the shock effect. The top view of the device is shown in Figure 3.



Figure 3: Top view of fabricated dual membranes device.

First we validated our method by theoretical modeling. The electrostatic attraction between the additional electrode and the movable membrane subdues the excessive travel distance of the movable membrane by 37%, using merely 0.5 V. We performed the drop tests from 15 cm high, corresponding to ~40g calibrated by a commercial accelerometer, as shown in Figure 4.



Figure 4: Experiment Setup: MEMS devices along with readout interface sit on a drop test platform. The platform folds to let the board drop vertically. The outputs of DUT were recorded through DAQ.

The drop tests show that the shock-mitigated scheme reduces the excessive movement of the membrane by different voltages between the electrode and membrane. The travel distance of the conventional structure without shock-mitigated electrostatic force was compared as a control to demonstrate the effectiveness of the shock-mitigation. The dual membrane shock-mitigated structure dramatically reduced the membrane travel distance (Figure 5).



Figure 5: Experimental results: response upon a shock of \sim 40g. (a) Shock response of single membrane device. (b) Shock response of dual membrane device reduced 37% by using merely 0.5 V between electrode and membrane.

We also imposed acoustic excitation of 96 dB SPL using a speaker along with the shock, 22 cm height, corresponding to \sim 100 g, to emulate realistic setting of MEMS microphones in operation. The experimental results have good correspondence with the theoretical calculations, having a discrepancy of less than 25%, in Figure 6.



Figure 6: Trend line of shock response with acoustic excitation under different potential differences (0 V, 1V, 1.5 V). g_m is the travel distance of membrane, g_0 is the nominal gap distance. V_A is the voltage between electrode and membrane. V_B is the voltage between membrane and substrate.

Finally, the shock mitigation method was demonstrated as dynamic *in-situ* implementation using a commercial accelerometer, MMA1200KEG from Freescale Inc., to trigger the on-board microcontroller, Arduino Zero from Arduino Inc., to control the electrostatic force between electrode and membrane. Upon the external shock, the accelerometer triggers the micro-controller to control the electrostatic force within 200 µs before the membrane reaches its maximum displacement (Figure 7(a)).



Figure 7: Dynamic in-situ control of shock-mitigation: (a) The accelerometer triggers the micro-controller, at threshold voltage of > 1.16 V, to control the electrostatic force between electrode and membrane within 200 µs. (b) Shock response without any mitigation. (c) Shock response with mitigation of the dynamic control reduced the membrane travel distance by 50% using 1.56 V.

CONCLUSIONS

A dual-membrane MEMS structure was presented to effectively mitigate the external perturbation, including shock. We elaborately demonstrated the validity of the approaches based on theoretical and experimental results. A smart shock mitigation system consists of an accelerometer and controller; the dualmembrane structure showed that it automatically senses the shock, controls the electrostatic force to mitigate the shock response, and make compensation to restoration force in order to reduce the excessive movement of membrane.

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REDUCED RADIUS OF CURVATURE AND OPTICAL ACTUATION OF NITINOL SHAPE MEMORY ALLOY MEMS ACTUATORS

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NOVELTY / PROGRESS CLAIMS

Through the use of a thinner platinum (Pt) and NiTi, we have achieved a 5.45 X reduction in radius of curvature (R_C) for thin film NiTi on Pt actuators that fold flat when heated beyond 60 °C. The radius of curvature reduced from 1.2 mm [1] to 220 μ m, enabling devices with an overall smaller size. We have also optically actuated devices at 1.44-24.0 W/cm², demonstrating actuation times from 4 ms to 240 ms.

BACKGROUND / STATE OF THE ART

Sputter-deposited NiTi shape memory alloys are well characterized on a material basis, and several demonstrations of NiTi MEMS cantilevers or actuators have been reported [2]. These devices are variations of sputtered NiTi films on released silicon or other micro machined substrates, where both layers are typically several micrometers or more in thickness [3] ultimately putting a limit on bending radius and range of motion. Cantilever tip deflections in the range of 100's of micrometers for cantilevers several millimeters in length are typical [4].

One reason for the limited selection of substrate materials for MEMS applications is the need to carry out a crystallization step at temperatures of 450°C or higher. This anneal step is critical to realizing any shape memory effect, but the fact that significant interdiffusion occurs between NiTi and any substrate material can limit how thin the initial NiTi film may be. Consequently, thicker films are typically used to insure that any interdiffusion layer is an insignificant part of the overall device.

DESCRIPTION OF NEW METHOD / SYSTEM

Here, we report on the characterization of NiTi films on Si and Pt layers, with NiTi deposited at 600 °C at an in-situ crystallization process. We focused on equiatomic NiTi, which thermally transforms between martensite and austenite phases near room temperature, corresponding to significant changes in residual stress, Young's modulus, and other properties that translate into large force and deflection.

Figure 1 shows the process flow used to fabricate the devices which was accomplished via a blanket Pt evaporation and NiTi co-sputter deposition (Figure 1a) and subsequent lithographic device patterning with a dilute HF wet etch and ion mill for NiTi and Pt films, respectively (Figure 1b). An optional Au electroplate step is possible to help stabilize wider cantilevers and restrict bending to 1 dimension (Figure 1c). We have shown that this step is not necessary when the cantilever width is ~20 μ m or less. A

xenon difluoride (XeF₂) dry release was performed to release devices from the Si substrate (Figure 1d) where a 5214 mask was used on both the wafer front and backside and a post-release 100 °C thermal activation was required to set the devices into an upward curl.

RESULTS

XRD data for NiTi (270-1600 nm) on Si in Figure 2 shows that the NiTi has martensite phase present at RT and confirms that the phase change is beyond ambient, which we have confirmed additionally with wafer bow vs T and DSC measurements (not reported here).

Figure 3 shows the trend in measured and predicted radius of curvature for different NiTi/Pt film stacks based on Klein's bimorph model [5]. The model is in excellent agreement for 1.4 μ m NiTi on 200 nm Pt, but 20 nm Pt devices deviate significantly from the model prediction. Despite this disagreement, we have obtained devices with very tight ~200 μ m R_c using 600 nm NiTi on 20 nm Pt.

Figure 4 shows a SEM image of our tightest curled device with $\sim 200 \ \mu m$ radius of curvature at room temperature, which is nearly flat above 70°C as shown in Figure 5. Figure 6 shows the optical actuation of the 600 nm NiTi on 20 nm Pt devices with 200 $\mu m R_c$ in ~ 20 ms.

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Figure 1: Process flow for NiTi on Pt MEMS SMA actuators with dry release



Figure 3: Predicted (solid and dashed lines) and measured (symbols) radius of curvature R_C vs. NiTi thickness for 20 nm and 200 nm Pt bimorphs



Figure 5: Thermal activation of 600 nm NiTi on 20 nm Pt devices between tightly curled at RT and flat at 100 °C



Figure 2: XRD spectra for various NiTi thickness on Si confirming martensitic phase at RT



Figure 4: SEM image of released and thermally activated 600 nm NiTi on 20 nm Pt devices with thinnest reported radius of curvature of ~200 µm



Figure 6: Video frames showing optical actuation of a cantilever array of 600 nm NiTi on 20 nm Pt using a 532 nm, 440 mW laser with intensity of 6 W/cm².

TEMPERATURE SENSITIVITY OF SOLID-WAVE GYROSCOPES

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ABSTRACT

We analyze the change of angular gain and vibration frequency of solid-wave gyroscopes as a result of geometry perturbations due to thermal expansion. We formulate a temperature sensitivity analysis by assuming a linear dependence of material properties to temperature, and quantify it for common device geometries.

INTRODUCTION

The lumped model of a solid-wave gyroscope involves two degenerate, coupled, damped and driven harmonic oscillators:

$$\ddot{\mathbf{q}} + 2(\gamma \mathbf{I} + A_g \Omega \mathbf{J})\dot{\mathbf{q}} + \omega^2 \mathbf{q} = \mathbf{f}, \ \mathbf{I} \equiv \begin{bmatrix} 1 & 0 \\ 0 & 1 \end{bmatrix}, \ \mathbf{J} \equiv \begin{bmatrix} 0 & -1 \\ 1 & 0 \end{bmatrix}$$

where the vectors $\mathbf{q}(t)$ and $\mathbf{f}(t)$ correspond to generalized coordinates and drive forces respectively. ω is the undamped angular vibration frequency and Ω is the rotation rate of the gyro platform. $\gamma = \omega/2Q$ is the damping coefficient and A_g is known as angular gain [1].

As the device temperature T changes, the coefficients of the lumped model drift. Computing $\gamma(T)$, $A_g(T)$ and $\omega(T)$ requires modeling geometric changes of the resonator. Since the damping coefficient $\gamma(T)$ depends on the relevant damping mechanisms, we only compute sensitivity of $A_g(T)$ and $\omega(T)$.

METHOD

Typical solid-wave gyroscope resonators are axisymmetric shells clamped at one of two boundaries and free on the other (Fig. 1). The cross section of the shell's midsurface forms a plane curve on (r, z)-plane. We parametrize this curve by its arclength s and use h, L and d to represent the shell thickness, the length of the midline curve, and the distance from the clamped boundary to the symmetry axis, respectively.

Temperature variations change both material properties and geometry of resonators. The elastic coefficients of conventional materials used in microfabrication change almost linearly within the standard range of operational temperatures: $E(T) = E_0(1 + \alpha_E(T - T_0)), \nu(T) = \nu_0(1 + \alpha_\nu(T - T_0))$. Also, due to thermal expansion, the device geometry and material density change slightly: $\rho(T) = \rho_0(1 - 3\alpha_L(T - T_0))$, where α_L is the thermal coefficient of expansion of the resonator material. We assume the post, attached at r = d, expands in radial direction with thermal coefficient α_S corresponding to the substrate material.

First, we discretize the axisymmetric thermal expansion problem: $\mathbf{K}_T \mathbf{u}_T = \mathbf{f}_T$, which is inherently nonlinear; but we only need $\frac{d\mathbf{u}_T}{dT} = \mathbf{K}_T^{-1} \frac{d\mathbf{f}_T}{dT}$ in our analysis. We use this solution to differentiate the resonator shape with respect to



Fig. 1: Left: Axisymmetric shell. Clamped at the bottom boundary, free at the top boundary. Center: Cross section in (r, z)-plane. Right: Computational mapped domain.

temperature and solve for the sensitivities of the lumped model coefficients [2]. We non-dimensionalize the equations using units of length $l^* = L$, time $\tau^* = L/\sqrt{E_0/\rho_0}$, mass $m^* = \rho_0 L^3$ and temperature $T^* = \alpha_L^{-1}$.

RESULTS

We investigated temperature sensitivities for three common geometries (Fig. 2). They all have the same midline length and shell thickness. We selected d so that radial extents are the same. Table 1 shows the numerical values used in simulations. In Table 2, we tabulated sensitivities for m = 2 and m = 3 modes for each resonator.

Since all thermal coefficients are small, we can write temperature sensitivity of angular gain or frequency as a sum of all effects: $y = C_L \alpha_L + C_S \alpha_S + C_E \alpha_E + C_\nu \alpha_\nu$, where y is either $(1/A_g) dA_g/dT$ or $(1/\omega) d\omega/dT$, and C's are constants for a particular resonator. By setting all α 's to zero except one, we can compute the contribution of each thermal effect (Table 3, 4).

Design C is particularly sensitive to Poisson's ratio, and so is more sensitive to temperature variations than \mathcal{H} and \mathcal{T} . The effects of substrate thermal expansion matter just as much to thermal sensitivity in the other designs, highlighting the importance of selection of the post material and radius. For all devices and modes, angular gain is independent of Young's modulus. Frequency sensitivity depends strongly on thermal variations of Young's modulus, much more than on any geometric effects; hence, new geometries are unlikely to improve frequency sensitivity. In contrast, by proper choice of the post radius, we can achieve temperature insensitive angular gain (Fig. 3). We can also formulate basic shape optimization problems as in Fig. 4, where the cost function is defined proportional to $(\frac{1}{A_a} \frac{dA_a}{dT})^2$.

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Fig. 2: Left: Cylindrical shell C. Center: Truncated hemispherical shell \mathcal{H} . Right: Half-toroidal shell \mathcal{T} .

Table 1: Material properties and geometry of resonators

Parameter	Symbol	Value
Young's modulus	E_0	80 GPa
Poisson's ratio	$ u_0$	0.26
Material density	ϕ_0	$2200 kg/m^{3}$
Thermal expansion coefficient of resonator	α_L	$1.4 \times 10^{-6} K^{-1}$
Thermal expansion coefficient of substrate	α_S	$2.6 \times 10^{-6} K^{-1}$
Thermal coefficient of E	α_E	$2.5 \times 10^{-4} K^{-1}$
Thermal coefficient of ν	$\alpha_{ u}$	$3.0 \times 10^{-4} K^{-1}$
Thickness to length ratio	h/L	0.01
Post radius to length ratio (cylindrical)	d_1/L	$12/5\pi$
Post radius to length ratio (spherical)	d_2/L	$12\sin(\pi/12)/5\pi$
Post radius to length ratio (toroidal)	d_3/L	$2/5\pi$
Radius of hemisphere to length ratio	R_2/L	$12/5\pi$
Minor radius of torus to length ratio	R_3/L	$1/\pi$



Fig. 3: Temperature sensitivities of angular gain of truncated hemispherical shell as a function of post radius to shell radius ratio. Smaller posts are desirable for m = 2, 3 modes. It seems possible to design m = 4 with A_g insensitive to temperature.



Fig. 4: The simplest shape optimization problem. Bottom left: Three control points representing truncated hemispherical shell. The middle point is free to move along the dashed line while the other two are fixed. Bottom middle and right: Optimal shape and new position of the control point which minimizes temperature sensitivity of angular gain for modes m = 2 and m = 5 respectively. Top: Local minimum of the cost function for m = 2 and m = 5 by sweeping the position of control point on the dashed line.

Table 2: Temperature sensitvities

\mathcal{D}	m	A_g	$\frac{1}{A_g} \frac{dA_g}{dT} \left(\frac{ppb}{K}\right)$	$\omega \left(\frac{1}{L}\sqrt{\frac{E_0}{\rho_0}}\right)$	$\frac{1}{\omega}\frac{d\omega}{dT} \left(\frac{ppm}{K}\right)$
C	2	0.744	-613	0.345	102.2
	3	0.577	-1694	0.214	108.5
11	2	0.554	-305	0.028	108.2
n n	3	0.487	-126	0.061	101.5
τ	2	0.304	-576	0.040	121.6
'	3	0.357	-220	0.091	110.5

Table 3: Thermal components of angular gain sensitivity

\mathcal{D}	m	$\frac{1}{A_g}\frac{dA_g}{dT} \left(\frac{ppb}{K}\right)$	$C_L \alpha_L$	$C_S \alpha_S$	$C_E \alpha_E$	$C_{\nu}\alpha_{\nu}$
C	2	-613	56	-313	0	-356
	3	-1694	251	-199	0	-1747
21	2	-305	-105	-269	0	70
Π	3	-126	0	-145	0	18
τ	2	-576	-4	-441	0	-131
'	3	-220	0	-231	0	11

Table 4: Thermal components of frequency sensitivity

\mathcal{D}	m	$\frac{1}{\omega}\frac{d\omega}{dT}\left(\frac{ppm}{K}\right)$	$C_L \alpha_L$	$C_S \alpha_S$	$C_E \alpha_E$	$C_{\nu}\alpha_{\nu}$
C	2	102.16	-4.90	0.67	125	-18.6
C	3	108.48	-7.96	1.05	125	-9.5
11	2	108.25	-6.73	0.37	125	-10.3
п	3	101.55	0.64	-1.06	125	-23.0
τ	2	121.63	0.64	-0.28	125	-3.7
	3	110.52	0.70	-0.60	125	-14.6

A MEMS BASED METHOD TO FABRICATE A MICRO PRESSURE SWIRL ATOMIZER AND ITS PERFORMANCE

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INTRODUCTION

Miniaturization of spray nozzle geometry [1] is likely to create the next generation innovations in the development of gas turbine injectors [2]. Several techniques have been explored to fabricate the atomizers in the microscale [3]. Here we use siliconglass wafer bonding and micromachining techniques to realize micro-atomizer arrays [4]. We have used a SetScan[®] optical patternator, consisting of six laser sheets and six array camera, to non-invasively characterize the uniformity of the micro-atomizer spray [5] along with high-speed shadow graphic imaging technique using a Photron Fastcam[®] to measure the spray angle. This study reports a parametric variation of the micro-atomizers to explore the limits of miniaturization while not compromising spray quality. The flow numbers [1] that we have been able to achieve in this study are well below those commercially available at acceptable spray quality. This technology holds promise to reliably manufacture microspray injectors, which could lead a paradigm shift in atomizer technology.

MICRO ATOMIZERS FABRICATION PROCESS

Figure 1 illustrates top and cross-sectional views of the microspray atomizer chip and the fabrication process used. A 450 μ m thick 4 inch, (100) double-side polished silicon wafer was photolithographically patterned with the swirlers pattern. The wafer was then etched using a deep reactive ion etcher (DRIE), creating 100 µm deep channels (see Figure. 1(A)). The etched silicon is anodically bonded to a 150 µm thick borofloat glass wafer. Thereafter, silicon surface of the bonded wafer was patterned to define the outlet for the spray (see Figure 1(B)) and etched through the remaining thickness of silicon in the patterned region. The bonded stack was next placed into 49% HF to reduce the thickness of the glass to \sim 50 μ m. Finally, four supply (inlet) holes were patterned on the glass side. Nickel electrodeposition was performed to create a mask for RIE glass etching. The exposed glass was etched down through the glass thickness to form the inlet orifices (see Figure 1(C)). Figure 1 shows three atomizers, multiple spray configurations. A set of 25 variations of the base design atomizers have been fabricated by varying one parameter at a time.

RESULTS AND DISCUSSION

All the micro-atomizer spray experiments have been performed at 8 bar supply pressure and the optical patternation measurement plane B'B" is located 5 mm below from the exit orifice. Device number (D_N) 2 has the highest spray angle and also exhibited the most uniform spray among all the design variations. Figure 2 shows the high speed shadow graph image of the spray from D_N -2. The time averaged spray pattern of D_N -2 is shown in Figure 3. Figure 4 shows the line-of-sight averaged surface area per unit length of the spray contour in Figure 3, which indicates the degree of hollowness of the spray. The flow rate (Q) and the spray angle (2 Θ) are well correlated as seen in Figure 5. Increase in the flow rate increases the spray angle. The flow rate is increased by increasing the number of inlet slots (S_n). The spray angle is increased by decreasing the swirl chamber diameter (D_s) to exit orifice diameter (d_o) ratio as well as slot length (S_i) to slot width (S_w) ratio. These dependencies are shown in Figure 6. The detailed parametric variations of the atomizers along with the experimentally measured flow and spray angle data are listed in Table 1. Increasing the number of inlet ports increases the swirl velocity inside the swirl chamber, which results in large spray angle. The time averaged spray pattern contour for the various parametric atomizer designs are shown in Table 2. This table qualitatively describes the effect of the various flow parameters on spray uniformity.

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Figure 1: Cross-sectional views of the devices



Figure 2: High speed shadow graph image captured at 50 kHz on Device No.2



Figure 3: Time averaged spray pattern on D_N *-2 in* B' - B'' *Plane*

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Figure 4: *Line-of-sight averaged surface area per unit length showing the hollowness of the spray in* D_{N-2} *in* B' - B'' *Plane*



Figure 5: Spray angle (2Θ) and flow rate (Q) measurements.



Figure 6: Spray angle (2 Θ) measurements with respect to geometric variation

Table	1: Devic	e specifica	itions and	experimentall	v measured	data
					/	

D_N	S_n	S_l/S_w	D_{s}/d_{o}	Q (lpm)	20 (degrees)
1	4	8.069	0.951	0.0242	36.44
2	4	8.069	0.792	0.0287	67.18
3	4	7.15	1.188	0.0231	46.22
4	4	7.15	0.951	0.0279	55.38
5	4	7.15	0.792	0.0315	63.84
6	3	12.75	1.188	0.0168	31.83
7	3	10.72	1.188	0.0173	33.69
8	3	10.72	0.951	0.0208	45.12
9	3	10.72	0.792	0.0235	55.61
10	3	9.22	1.188	0.0193	32.21
11	3	9.22	0.951	0.0221	45.44
12	3	9.22	0.792	0.0246	53.67
13	3	8.069	1.188	0.0203	39.65
14	3	7.15	1.188	0.0196	36.26
15	3	7.15	0.951	0.0244	50.04
16	2	12.75	1.188	0.0109	0.00
17	2	12.75	0.951	0.0157	22.72
18	2	12.75	0.792	0.0161	19.29

19	2	10.72	1.188	0.0153	17.20
20	2	9.22	0.951	0.0202	34.03
21	2	9.22	0.792	0.0200	43.54
22	2	8.069	1.188	0.0170	28.13
23	2	8.069	0.951	0.0189	39.51
24	2	8.069	0.792	0.0214	43.72
25	2	7.15	1.188	0.0156	19.51

Table 2: Devices and their time averaged spray pattern contours. The following key observations can be made: (i) S_n -4 sprays are generally more uniform than S_n -3 & S_n -2 sprays. (ii) In S_n -2 devices, the spray is not fully open as it remains in the onion formation stage. (iii) The hot spots in the D_N -2 spray shows some memory of the inlet slots but is the best atomizer design of the lot tested in terms of pattern uniformity (iv) Hot spots in D_N -7 is not a result of inlet slot memory; it is a result of the reunion of the dispersed liquid sheet due to the atomizer's inability to form a fully developed hollow cone.(v) Some contour plots have more number of hot spots than inlets (for example, D_N -14 and D_N -8). This is due to the high temporal spray angle fluctuation.



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