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Printed MEMS Membrane Electrostatic Microspeakers

A. Murarka, J. Jean, A. Wang, J. Lang, V. Bulović Sponsorship: NSF Center for Energy Efficient Electronics Science

This work reports the fabrication and operation of electrostatic microspeakers formed by contact-transfer of 125-nm-thick gold membranes over cavities patterned in a micron-thick silicon dioxide (SiO₂) layer on a conducting substrate. Upon electrostatic actuation, the membranes deflect and produce sound. Additionally, membrane deflection upon pneumatic actuation can be used to monitor pressure. The microspeaker fabrication process reported enables fabrication of MEMS diaphragms without wet or deep reactive-ion etching, thus obviating the need for etch-stops and wafer-bonding. This process enables monolithic fabrication of multiple completely-enclosed drum-like structures with non-perforated membranes to displace air efficiently, in both individual-transducer and phased-array geometries.

We characterized the mechanical deflection of the gold membranes using optical interferometry. The



▲ Figure 1: Deflection profiles of gold membrane suspended over cavities, obtained via optical interferometry during 1 kHz AC actuation. Center deflections of this membrane over 83 cavities as functions of time were extracted from the profiles and averaged to yield a single deflection time function. That time function and corresponding applied voltage are plotted. (a) Applied voltage (black) and averaged peak membrane deflection (red and blue) are shown as functions of time. (b) Membrane deflections are plotted against square of applied voltage with time as a parameter. Points labeled a-b-c-d-a in (b) indicate position of corresponding points on the periodic deflection and voltage waveforms in (a).

membranes show a repeatable peak center deflection of 121±13 nm across gaps of ~25 microns at 1 kHz sinusoidal actuation with 60 V peak-to-peak amplitude and a 30 V DC bias (Figure 1). The acoustic performance of the microspeakers is characterized in the free field. Microspeaker sound pressure level increases with frequency at 40 dB/decade (Figure 2), indicating that its sound pressure output is proportional to the acceleration of its diaphragm, as expected in the spring-controlled regime for free field radiation. The microspeaker consumes 262 µW of real electric power under broadband actuation in the free field, and outputs 34 dB(SPL/Volt) of acoustic pressure at 10 kHz drive. The silicon wafer substrate (~500 μ m thick) dominates the total thickness of the microspeakers; the active device thickness is less than 2 µm. These thin microspeakers have potential applications in hearing aids, headphones, and large-area phased arrays for directional sound sources.



▲ Figure 2: Acoustic frequency response, from 2 kHz to 20 kHz, of a gold membrane microspeaker, 12.5 mm² in area. Magnitude of transfer function, $|T_{pv}|$, from driving voltage amplitude to sound output measured by the microphone is converted to decibels using 20 µPa as reference pressure, i.e., magnitude plotted is $20\log_{10}(|T_{rv}|/20\mu\text{Pa})$.

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Stretchable Pressure and Shear Sensitive Skin

M. D'Asaro, V. Bulović, J.H. Lang Sponsorship: NSF CSNE

In the fields of robotics and prosthesis design, there is need for inexpensive, wide-area pressure- and shear-sensing arrays that can be integrated into a flexible and stretchable skin analog. This project seeks to meet this need by building combined pressure and shear sensors based on the well-documented piezoresistive (strain dependent resistance) property of composites made from polydimethylsiloxane (PDMS) and carbon black (CB).

The sensor skins are fabricated of three materials which are all PDMS-based: A CB/PDMS mixture is used as the active sensing material, a CB/PDMS and ~1 μ m silver particle mixture is used to form strain-insensitive conductors into the skin, and pure PDMS is used to form the base of the skin. These materials are mixed, vacuum degassed, and then molded in custom-machined acytal and aluminum molds to fabricate the sensor arrays. Each sensor consists of a roughly hemispherical piece of CB/PDMS molded on top of a line of three conductors, thus

allowing the resistance of each half of the CB/PDMS sensor to be measured independently. A schematic representation of a single sensor is shown in Figure 1.

Our own characterization experiments performed on bulk (1 cm³) CB/PDMS samples have shown that the resistance of CB/PDMS increases under tensile, compressive, and shear strain but is much more sensitive to tensile strain then compressive strain. This symmetry allows the device to sense both pressure and shear. Under pressure, each half of the sensor has roughly equal compressive strain, and thus the resistance of each half of the device increases roughly equally. However, under shear, one half of the device is under tensile strain while the other half is under compressive strain. Due to the asymmetric response of the CB/PDMS, the resistance of the half of the sensor under tension increases much more than the half under compression, allowing a differentiation between pressure and shear; see Figure 2.



▲ Figure 1: Cut-away side-view of sensor under shear. Pure PDMS skin into which this sensor is embedded is not shown. The CB/PDMS between contacts A and B is under tension, causing large increase in resistance between them while CB/PDMS between contacts B and C is under compression, resulting in relatively little resistance chance between them.



▲ Figure 2: Plot of the resistance of one half of sensor vs. resistance of other half with time as a parametric variable. Note that when sheared, resistance in one half changes much more than the other but when put under pressure, both resistances change.

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Tunneling Nanoelectromechanical Switches Based on Molecular Layers

F. Niroui, E.M. Sletten, P.B. Deotare, A.I. Wang, T.M. Swager, J.H. Lang, V. Bulović Sponsorship: NSF Center for Energy Efficient Electronics Science

Nanoelectromechanical (NEM) switches have emerged as a promising competing technology to the conventional complementary metal-oxide semiconductor (CMOS) transistors. NEM switches can exhibit abrupt switching behavior with large on-off current ratios and near-zero off-state leakage currents. However, they typically require large operating voltages exceeding 1 V and suffer from failure due to stiction. To address these challenges, this work presents NEM switches utilizing metal-molecule-metal switching gaps. These switches operate by electromechanical modulation of the tunneling current through electrostatically-induced compression of the molecular film (Figure 1). The molecular layer helps define few-nanometer-thick switching gaps to achieve low-voltage operation. In addition, the compressed molecules prevent direct contact between the electrodes while providing a restoring force to turn off the device once the applied voltage is removed, thereby preventing permanent adhesion between the electrodes and eliminating stiction.

A prototype two-terminal tunneling NEM switch is fabricated as a laterally actuated cantilever using electron beam-lithography (Figure 2). A fluorinated decanethiol layer is deposited over the device area and into the gap between Electrodes 1 and 2 using self-assembly through thiol-chemistry. During the assembly process, Electrode 1 collapses onto the opposing electrode to form a metal-molecule-metal junction with a nanometer thickness. Experimental results based on a device with a self-assembled fluorinated decanethiol layer demonstrate repeatable switching, indicating the importance of the molecular film in alleviating stiction. Comparison of these results to the theoretically expected device behavior suggests the compression of the molecular layer during the switching process, confirming the electromechanical modulation of tunneling current as the switching mechanism. Our current research focuses on engineering the molecular layer and the device design to optimize the NEM switch performance to achieve stiction-free sub-1-V actuation with more than 6 orders of magnitude on-off current ratio.



▲ Figure 1: Schematic representation of a tunneling NEM switch using a molecular thin-film. An applied voltage compresses the nanometer-thick organic layer to modulate the tunneling current by altering the tunneling gap between the metal contacts



▲ Figure 2: Scanning electron micrograph of a tunneling NEM switch fabricated using electron-beam lithography. A fluorinated decanethiol layer is self-assembled onto the Au electrodes including the gap between Electrodes 1 and 2, forming a nanoscale molecular switching gap.

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Electrically-Tunable Organic Microcavities

W. Chang, A. Wang, A. Murarka, J.H. Lang, V. Bulović Sponsorship: NSF Center for Energy Efficient Electronics Science

The availability of a compact, single-system tunable visible light source would benefit a wide range of fields such as remote sensing, spectroscopy, and optical switches. Organic-based materials are attractive for visible light emission over a broad tunable spectrum. However, previously demonstrated frequency-tunable lasing devices required either complex fabrication techniques, external micro-actuated mirror stages, or manual switching between gain media. Tunable airgap MEMS microcavity structures offer a scalable, integrated solution but their typical fabrication processes are incompatible with solvent- and temperature-sensitive organic gain materials.

We have demonstrated a method for fabricating integrated organic optical microcavities that can be mechanically or electrostatically actuated to dynamically tune their output emission spectra. Fabrication of the micro-opto-electro-mechanical system (MOEMS) structures (as in Figure 1) is enabled by a solvent-free additive transfer-printing method for composite membranes that we have developed. The suspended membrane incorporates an organic laser gain medium, Alq₂:DCM, into the microcavity, and the completed capacitive structure can be electrostatically actuated for dynamic tuning of the optical spectra (see Figure 2). Electrical actuation and optical characterization of a completed cavity structure show reversible resonance tuning greater than 20 nm for net membrane deflections of over 200 nm at 50 V. The device structure and transfer technique are easily scalable for large area fabrication with applications in tunable lasers as well as remote alloptical pressure sensing and low-power optical switches.



▲ Figure 1: Cross-sectional view of a MEMS tunable cavity device structure with a diagram of the optical testing setup. A microscope objective focuses laser excitation and captures cavity emission. Subsequent filter and lens focus emission into spectrograph. Inset: Fabricated device array from top view.



▲ Figure 2: Longitudinal mode emission spectra for a range of applied voltage. The 637-nm center mode shifts to 614 nm under 50 V actuation, indicating a deflection of the composite membrane of over 200 nm. The measurements were taken in order of increasing voltage. The final measurement at 0 V (indicated by black 0 V spectrum) shows that tuning is reversible.

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MEMS Tactile Displays

C. Livermore (NEU), S. Teller, L.F. Velásquez-García, X. Xie (NEU), Y. Zaitsev (NEU) Sponsorship: Andrea Bocelli Foundation

Providing information to people who are blind or have low vision is critical for enhancing their mobility and situational awareness. Although refreshable 2D graphical interfaces are preferred, it is challenging to create actuators that are compact enough to be arrayed into an unlimited number of rows and columns while still being robust, easy to sense, and rapidly switchable. Electroactive polymer actuators are small enough to be arrayed with a few- millimeter pitch and to provide quasistatic millimeter-scale actuations, but they typically have actuation times on the order of seconds. An alternative integrates piezoelectric bending beam actuators perpendicular to the tactile sensing plane, enabling large bending beam actuators to be tightly packed for fully 2D displays.

Ideally, the display's resolution should be about one tactel (i.e., tactile element) per mm², which is the density of mechanoreceptors in human finger pads. It should be refreshable in real time (hundreds of Hz, i.e., the frequency response of human touch), allowing the contents of the display to keep up with rapidly changing inputs. Since humans are much more sensitive to motions and changing stimuli than to static patterns, the display should code information not only as static patterns, but also as simulated motion against the user's finger pads. Finally, the power consumption of the display should be compatible with portable use. Although existing displays meet various subsets of these requirements, no existing display can meet all requirements simultaneously.

We are developing tactile displays based on a new type of MEMS tactile actuator created to target these requirements. This new actuator concept uses an extensional piezoelectric actuator that operates a scissor amplifier that transforms the in-plane movement of the piezo into amplified out-of-plane movement (see Figure 1). We have shown these tactile elements to be effective at the milliscale. Their measured performance agrees with the models, with maximum deflections of greater than 10 μ m and maximum forces above 45 mN (as in Figure 2) that place the devices well above the sensing

threshold. Our analytical model based on ideal pinned hinges is shown to be useful for predicting the behavior of tactels with flexural hinges, especially when coupled with FEA to predict hinge failure. The analytical model validation provides support for further downscaling of the tactile elements to achieve 100 tactels/cm². The measured performance confirms sensing thresholds of less than 4 μ m and 2 mN for the most effective tactile devices.



▲ Figure 1: Schematic diagram of piezoelectric extension actuators (red) topped by scissor amplifiers (light blue) and cap plate.



▲ Figure 2: Measured tactel force vs. peak amplitude of the applied voltage. Markers represent data; lines guide the eye.

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Real-time Manipulation with Magnetically Tunable Structures

Y. Zhu, D.S. Antao, R. Xiao, E.N. Wang Sponsorship: Air Force Office of Scientific Research

Responsive actuating surfaces have attracted significant attention as promising materials for liquid transport in microfluidics, cell manipulation in biological systems, and light tuning in optical applications via their dynamic regulation capability. Significant efforts have focused on fabricating static micro and nanostructured surfaces, even with asymmetric features to realize passive functionalities such as directional wettability and adhesion. Recent advances in utilizing materials that mechanically respond to thermal, chemical or magnetic stimuli have enabled dynamic regulation. However, the challenges with these surface designs are associated with the tuning range, accuracy, response time, and multi-functionality for advanced systems.

Here we report dynamically tunable micropillar arrays with uniform, reversible, continuous, and extreme tilt angles with precise control for realtime fluid and optical manipulation. Inspired by hair and motile cilia on animal skin and plant leaves for locomotion, liquid transportation, and thermaloptical regulation, our flexible uniform responsive microstructures (µFUR) consist of a passive thin elastic skin and active ferromagnetic microhair whose orientation is controlled by a magnetic field. We experimentally show uniform tilt angles ranging from 0° to 57° and developed a model to accurately capture the tilting behavior. Furthermore, we demonstrate that the µFUR can control and change liquid spreading direction on demand, manipulate fluid drag, and tune optical transmittance over a large range. The versatile surface developed in this work enables new opportunities for real-time fluid control, cell manipulation, drag reduction, and optical tuning in a variety of important engineering systems, including applications that require manipulation of both fluid and optical functions.



▲ Figure 1: a) Fabricated μ FUR. Dark region is micropillar array; transparent substrate is PDMS skin. Scale bar is 5 mm. b) Schematic showing concept of μ FUR; tilt angle can be controlled via external magnetic field. θ is micropillar tilt angle; α is magnetic field angle. c) Schematic of potential applications including microfluidic and optical manipulation. Side view of fabricated μ FUR with applied magnetic field strength of 0.5 T and field angles of d) α =60° and e, α = 95° respectively. Scale bars are 50 µm.



▲ Figure 2: Time-lapse images of dynamic fluid spreading. Fluid (30% IPA and 70% water) spreading direction is dynamically controlled while the fluid propagates only in the pillar tilt direction (red arrow) and is pinned in all other directions. External magnetic field direction controls pillar tilt direction. Scale bar is 0.5 mm.

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GaN MEMS Resonator Using a Folded Phononic Crystal Structure

S. Wang, L.C. Popa, D. Weinstein Sponsorship: DARPA DAHI Foundry, National Science Foundation CAREER Award, EECS

We present a gallium nitride (GaN) Lamb-wave resonator using a phononic crystal (PnC) to selectively confine elastic vibrations with wide-band spurious mode suppression. A unique feature of the design demonstrated here is a folded PnC structure to relax energy confinement in the non-resonant dimension and to enable routing access of piezoelectric transducers inside the resonant cavity. This feature provides a clean spectrum over a wide frequency range and improves series resistance relative to transmission line or tethered resonators by allowing a low-impedance path for drive and sense electrodes. We demonstrate GaN resonators with wide-band suppression of spurious modes, f.Q product up to 3.06×10^{12} , and resonator coupling coefficient k_{eff}^2 up to 0.23% (filter BW up to 0.46%). Furthermore, these PnC GaN resonators exhibit record-breaking power handling, with IIP3 of +27.2dBm demonstrated at 993MHz.

This work focuses on developing MEMS resonators for channel-select filtering in RF receiver front ends. For a MEMS band pass filter, the presence of spurious modes in the constituent resonators strongly impacts filter performance. Resonators with a clean frequency spectrum help reduce ripples in the pass-band and prevent interference from unwanted signals outside the pass-band. Conventional MEMS resonator designs with free mechanical boundaries are inherently prone to spurious modes, since free boundaries act as acoustic reflectors over all frequencies. To resolve this issue, the resonator boundary needs to be frequency selective. One way is by using PnCs, which involve periodic scatters to achieve highly reflective boundary conditions only for frequencies in a specific range. This acoustic band gap can be engineered based on the unit cell size and material configuration. While the acoustic band gap of these PnCs helps reduce resonance outside the band gap, these structures provide no spurious mode suppression inside the band gap. Further, transducers must be routed through the PnC in these configurations, leading to resistive loading of *Q*. In this work, we demonstrate a new resonant structure leveraging both PnC acoustic confinement and the electromechanical benefits of GaN. The proposed GaN folded PnC structure provides several important benefits:

- wide-band spurious mode suppression, both outside and inside the PnC band gap, through relaxed confinement in the non-resonant dimension,
- · low-loss electrical routing to the resonant cavity,
- improved heat dissipation relative to other PnC or tethered resonators, and
- robust design that is immune to residual stress and handling.

The folded PnC design achieves these improvements while maintaining quality factor and transducer coupling comparable to traditional tethered resonators.



▲ Figure 1: Material stack and SEM of the fabricated resonator. IDT transducers (Ni) are routed into the resonant cavity through openings. The resonator is connected through vias to probe pads, which are made of Au/Ti.



▲ Figure 2: Measured frequency response of the resonator shown on the left with wide and narrow sweeps showing spurious-free spectrum.

GaN RF MEMS Resonators in MMIC Technology

L.C. Popa, D. Weinstein Sponsorship: Raytheon, DARPA DAHI, MIT/MTL GaN Energy Initiative

As a wide bandgap semiconductor with large breakdown fields and saturation velocities, gallium nitride (GaN) has been increasingly used in high-power, high-frequency electronics and monolithic microwave integrated circuits (MMICs). At the same time, GaN also has excellent electromechanical properties, such as high acoustic velocities and low acoustic losses. Together with a strong piezoelectric effect, these make GaN an ideal material for RF MEMS resonators. This work focuses on the optimization of L-band (1-2 GHz) GaN resonators in *standard* MMIC technology.

monolithically integrated resonators, For various constraints of the technology must be considered, such as the thickness of the GaN MMIC heterostructure, residual stresses in the GaN film, and the lack of bottom electrodes. Residual stress due to high temperature growth can affect the mechanical properties of the resonators and even lead to cracking and breaking. To achieve high performance resonators with multiple frequencies on the same chip within this technology, we designed 5th-order extensional resonators driven piezoelectrically with a top metal interdigitated transducer (IDT) as shown in Figure 1. These resonators have achieved mechanical quality factors >5500 at 1GHz, with f·Q products >5.5×10¹², the highest demonstrated in GaN to date.

Enhanced signal-to-noise ratios (SNR)at high frequencies can be obtained by using active transistor sensing. We demonstrate the first mechanicallycoupled Resonant Body Transistor, in which the drive transducer and sensing high electron mobility transistor (HEMT) are embedded in two separate cavities, as shown in Figure 1. This additional electrical isolation between drive and sense allows for an improvement in the SNR of >50× compared to previous designs. The large SNR, together with high Q (Figure 2), makes these resonators ideal for monolithically integrated low-phase noise oscillators, with applications in clocking and wireless communications.



▲ Figure 1: SEM of Mechanically Coupled Resonant Body Transistor. A top metal interdigitated transducer is used to launch acoustic waves. At resonance, strain fields modulate the HEMT's channel charge density, resulting in AC drain current.



Figure 2: Experimental measurement of electromechanical transconductance, Y_{21} - Y_{12} . Inset shows clear phase transition at the resonant frequency.

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Ion Energy Measurements of Dense Plasmas with a Microfabricated RPA

E.V. Heubel, L.F. Velásquez-García Sponsorship: NASA

The energy of ions determines the efficiency of plasma propulsion systems and governs surface chemical reactions in plasma etching chambers. In plasma diagnostics, the instrument used to measure the ion energy distribution is the Retarding Potential Analyzer (RPA). However, high-density plasmas of interest require tens- to hundreds-of-microns scale dimensions. Through MEMS processing techniques, our RPA achieves the small aperture sizes necessary to measure dense plasmas. Precise alignment between successive microfabricated grids is achieved through compliant support structures in the housing (as Figure 1 shows). The silicon spring tips mate with corresponding notches in the electrodes to provide robust alignment on the order of 1 μ m and to increase the overall sensor's ion transmission.

Our previously reported RPA, deemed "hybrid" on account of incorporating microfabricated electrodes in a conventionally machined sensor, demonstrated improved performance over conventional RPAs. By reducing the aperture size while enforcing some degree of aperture alignment, we achieved a better resolution with no loss in signal strength compared to conventional mesh RPAs. Measurements of the ion energy distribution in a helicon plasma were obtained at MIT's Plasma Science and Fusion Center using our sensors with microfabricated electrodes having 100 μ m apertures. However, as a consequence of its larger apertures, the conventional RPA design was unable to effectively trap the plasma, and therefore no ion distribution could be extracted with this traditional device.

Figure 2 shows ion energy distributions obtained with an ion source comparing the performance of a conventional RPA (with 152 μ m apertures), the hybrid RPA (with 100 μ m apertures), and MEMS RPA (with 150 μ m apertures). The MEMS RPA design utilizes a fully microfabricated housing to improve upon the inter-grid aperture alignment over the hybrid sensor. Additionally, various aperture diameters are utilized in the electrode stack to mitigate current interception within the sensor. These RPA improvements result in an order of magnitude increase in signal strength over the conventional device and a threefold increase in energy distribution resolution.



▲ Figure 1: MEMS RPA housing exploded view (left) of actual wafer layers. Concept and cross-section view of sensor with assembled electrodes (right).



▲ Figure 2: Ion energy distribution measurements of a mass spectrometry ion source. The MEMS RPA shows an order-of-magnitude increase in signal strength compared to a conventional RPA with similar aperture sizes, accompanied with a threefold reduction in peak width associated with increased sensor resolution.

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High Throughput Electrospinning of Nanofibers from Batch-microfabricated, Externally-fed Emitter Arrays

P.J. Ponce de Leon, F.A. Hill, L.F. Velásquez-García

Nanofibers promise to be a key engineering material in the near future due to their unique, nanoscale morphological properties. In particular, the large specific surface area of the porous webs they form make them highly desirable as scaffolds for tissue engineering; layers in multifunctional filters/membranes; and components in devices such as fuel cells, solar cells, and ultra-capacitors. However, their integration into almost all of these technologies is unfeasible as a result of the low throughput, high cost, and poor control of current production methods. The most common process for producing nanofibers involves applying strong electric fields to polar, high-molecular-weight polymeric liquids pumped through a syringe in what is known as electrospinning. Electrospinning is the only known technique that can generate nanofibers of arbitrary length; it has tremendous versatility as it can create non-woven or aligned mats of polymer, ceramic, semiconducting, and/or metallic fibers.

We implement high throughput arrays of externally-fed, batch-microfabricated electrospinning emitters that are precise, simple, and scalable. We fabricate monolithic, linear emitter arrays that consist of pointed structures etched out of silicon using DRIE and assemble these into a slotted base to form a two-dimensional array. By altering the surface chemistry and roughness of the emitters, we can modify their wetting properties to enable wicking of fluid through the micro-texture (as in Figure 1). The interplay between electric, viscoelastic, and surface tension forces governs the fluid transport and fiber formation. We achieve over 30 seconds of stable electrospinning of polyethylene oxide (2-4% w/v in 60/40 water/ethanol solution) from 9 emitters in a two-dimensional array with a density of 11 emitters/cm² using bias voltages around 10kV (see Figure 2). This density is 7 times greater than the emitter density achieved in similar array-based approaches. Current work focuses on characterization of larger, denser arrays to demonstrate uniform emission.



▲ Figure 1: Capillary rise against gravity of a 2% w/v polyethylene oxide (PEO) solution through microtrenches. Sufficient liquid supply is necessary for continuous electrospinning.



▲ Figure 2: Stable production of PEO nanofibers from a 3×3 array of 6-mm tall micropillar-patterned emitters.

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Near-Monochromatic X-ray Sources Using a Nanostructured Field Emission Cathode and a Transmission Anode for Markerless Soft Tissue Imaging

S. Cheng, F.A. Hill, E.V. Heubel, R. Gupta (MGH), L.F. Velásquez-García Sponsorship: DARPA

A conventional X-ray generator consists of a thermionic cathode and a reflection anode inside of a vacuum chamber that has an X-ray transmission window. The cathode generates a beam of electrons that is accelerated towards the anode, which is biased at tens of kilovolts above the cathode voltage. Some of the electrons collide with the anode and convert their kinetic energy into radiation, a fraction of which escapes the vacuum chamber through a transmission window made of a suitable material, such as beryllium. The X-ray emission is a mix of bremsstrahlung radiation (broad, continuous spectrum) and fluorescence (emission at specific peaks corresponding to atomic shell transitions). Conventional X-ray technology requires high vacuum to operate, does not efficiently produce X-rays, and has overall low power efficiency. Conventional X-ray generators cannot image well soft tissue unless contrast media, i.e., markers, are employed.

We are developing efficient X-ray generators capable of soft tissue imaging using batch-microfabricated field emission cathodes composed of arrays of selfaligned, gated, and nanometer-sharp n-silicon tips, and a microstructured transmission anode (Figure 1). The nanostructured silicon cathode operates at low voltage and reliably achieves high-current emission with high transmission. The transmission anode efficiently generates X-rays while reducing the background radiation, resulting in emission of X-rays with narrow spectral linewidth for sharp imaging of biological tissue.

Using our first-generation X-ray source (a tabletop apparatus), we have obtained absorption images of ex-vivo samples that clearly show soft tissue and fine bone structures (Figure 2). Current work focuses in miniaturizing the X-ray source into a portable system, and in improving the cathode and anode components to achieve generation of coherent X-rays to make possible phase contrast imaging at a low cost.



▲ Figure 1: Schematic of an X-ray generator with a field emission cathode and a transmission anode.



▲ Figure 2: Absorption X-ray image of an ex-vivo human wrist showing soft tissue structures and fine details of the bone structure.

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Multiplexed MEMS Electrospray Emitter Arrays with Integrated Extractor Grid and CNT Flow Control Structures for High-Throughput Generation of Ions

F.A. Hill, E.V. Heubel, P. Ponce de Leon, L.F. Velásquez-García Sponsorship: DARPA

Electrospray is a process to ionize electrically conductive liquids that relies on strong electric fields. Charged particles are emitted from sharp tips that serve as field enhancers to increase the electrostatic pressure on the surface of the liquid, overcome the effects of surface tension, and facilitate the localization of emission sites. Ions can be emitted from the liquid surface if the liquid is highly conductive and the emitter flowrate is low. Previous research has demonstrated successful operation of massive arrays of monolithic batch-microfabricated planar electrospray arrays with an integrated extractor electrode using ionic liquids EMI-BF, and EMI-Im—liquids of great importance for efficient nanosatellite propulsion and nanomanufacturing. The current design builds upon a previous electrospray array designs from our group by increasing the area density of the emitter tips and increasing the output current by custom-engineering nanofluidic structures for flow control.

Our MEMS multiplexed electrospray source consists of an emitter die and an extractor grid die (Figure 1), both made of silicon and fabricated using deep reactive ion etching. The two dies are held together using a MEMS high-voltage packaging technology based on microfabricated springs that allows precision packaging of the two components with low beam interception. The emitter die contains dense arrays of sharp emitter tips with over 1,900 emitters in 1 cm². A voltage applied between the emitter die and the extractor grid die creates the electric field necessary to ionize the ionic liquid. A carbon nanotube forest grown on the surface of the emitters transports the liquid from the base of the emitters to the emitter tips. Our electrospray arrays operate uniformly (Figure 2), and mass spectrometry of the emission demonstrates that our devices only produce ions.



▲ Figure 1: (in counter-clockwise starting on the upper left corner) array of 2,000 electrospray emitters in 1 cm² with integrated extractor grid; close-up of electrospray emitter forest; close-up of a single electrospray emitter covered by a layer of CNTs; detail of an emitter tip.



▲ Figure 2: Collector current per emitter as a function of emitter-to-extractor bias voltage for (A) the electrospray sources with 4, 9, 25, 49 and 81 emitters in 1 cm² evidencing great array uniformity.

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Exploration of the Packing Limits of Ultrafast, Optically-triggered Silicon Field-emitter Arrays Using the Finite Element Method

C. Dong, M.E. Swanwick, P.D. Keathley, F.X. Kärtner, L.F. Velásquez-García Sponsorship: DARPA

Ultrafast optically-triggered field emission cathodes bypass several disadvantages demonstrated by current state-of-the-art ultrafast cathodes, such as requiring ultra-high vacuum to operate and short lifetime, and are a promising technology for implementing spatially-structured electron sources for applications such as free-electron lasers, compact coherent X-ray sources, and attosecond imaging. Ultrafast optically-triggered cathodes composed of massive arrays of high aspect-ratio silicon pillars capped by nano sharp tips and 5 μ m pitch were fabricated at MIT MTL. The effect of the geometry and the morphology of the Si pillar arrays on the ultra-fast emission characteristics of such cathodes is now explored using the finite element modeling in 2D and 3D.

Since the field-emitted current depends exponentially on the surface electric field, we are interested in studying how the electric field is enhanced by the geometry and the morphology of the Si pillar arrays. We selected COMSOL Multiphysics to simulate the electric field of the devices. The 3D model

(see Figure 1) consists of a single tapered pillar 2.0 µm tall and 0.7 µm wide at the base with a 6-nm radius hemispherical cap. Perfectly matched layers (PMLs) are added on the top and bottom to absorb the excited and higher order modes. Floquet periodicity is applied on the four sides of the unit cell to simulate the infinite 2D array. The port boundary condition is applied on the interior boundary of the PML as the excitation port to simulate the 800-nm incident wave at a glancing angle of 84° from normal (the same experimental setup described in the third reading below). This model is validated by verifying the Fresnel equations between Si and vacuum before inserting the Si pillar. The 2D slice contour plot (see Figure 2) shows the simulated electric field from a 1 GV/m incident field on an emitter with 1-µm pitch using frequency domain analysis. The maximum electric field at the tip is about 4.2 GV/m, i.e., the emitter tip has an field enhancement factor of ~4.2. Both 2D and 3D models are utilized to explore the effect of the geometry and the morphology of the Si pillar arrays on the field enhancement.



▲ Figure 1: The 3D COMSOL model consists of a single pillar, PMLs on the top and bottom, Floquet periodicity on four sides, and a source port with an incident 800-nm wave at a glancing angle of 84°.



▲ Figure 2: 2D slice contour plot from the 3D model with 1-µm pitch shows the maximum electric field at the tip, with a field enhancement factor of ~4.2.

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High-Current Field Emission Cold Cathodes with Temporal and Spatial Emission Uniformity

M.E. Swanwick, F.A. Hill, L.F. Velásquez-García Sponsorship: DARPA

Field emission arrays (FEAs) are an attractive alternative to mainstream thermionic cathodes, which require high vacuum and high temperature to operate. Field emission of electrons consists of the following two processes: first, the transmission of electrons (tunneling) through the potential barrier that holds electrons within the material (workfunction ϕ) when the barrier is deformed by a high electrostatic field and second, the supply of electrons from the bulk of the material to the emitting surface. Either the transmission process or the supply process could be the limiting step that determines the emission current of the field emitter. Due to the exponential dependence on the field factor, the emission current from the tips is extremely sensitive to tip radii variation. We have a process to achieve uniform emission from nanosharp FEAs by both fabricating highly uniform tip arrays and controlling the supply of electrons to the emitting surface (see Figure 1).

We have designed and fabricated FEAs in which each field emitter is individually ballasted using a vertical ungated field effect transistor (FET) made from a high aspect ratio (40:1) n-type silicon pillar. Each emitter has a proximal extractor gate that is self-aligned for maximum electron transmission to the anode (collector). Our modeling suggests that these cathodes can emit as much as 30 A.cm⁻² uniformly with no degradation of the emitters due to Joule heating; also, these cathodes can be switched at microsecond-level speeds. The design process flow, mask set, and pillar arrays have been completed (as Figure 2 shows) with the self-aligned extractor gate. An ultra-high vacuum chamber has been built to test the devices. The chamber can test full 150mm wafers with six high voltage feed through and a step-down anode at 2x10⁻¹⁰ torr pressure while also imaging the electron emission on a phosphorus screen.



▲ Figure 1: IV curves for high aspect ratio Si (1-2 Ω .cm) pillars showing current saturation for 1, 4, 16, 64, and 121 pillars. Each pillar is 25 µm tall, 500 nm in diameter, and capped with Al for ohmic contact.



▲ Figure 2: SEM image of a large array of high aspect ratio pillars with 10-nm radius tips with 5-µm hexagonal packing for individually ballast-ed field emission with self-aligned gates.

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Photoactuated Ultrafast Silicon Nanostructured Electron Sources for Coherent X-ray Generation

M.E. Swanwick, P.D. Keathley, C.D. Dong, F.X. Kärtner, L.F. Velásquez-García Sponsorship: DARPA

Nanostructured cathodes that can be switched at an ultrafast time scale (<50 ps) have applications in free-electron lasers and coherent X-ray sources. This project is creating the theory, modeling, and experimental results for a compact coherent xray source for phase contrast medical imaging based on inverse Compton scattering of relativistic electron bunches. The X-ray system requires a low-emittance electron source that can be switched at timescales in the low femtosecond range.

The focus of our work has been the design, fabrication, and characterization of massive arrays of a nanostructured high aspect-ratio silicon structures to implement low-emittance and high-brightness cathodes that are triggered using ultrafast laser pulses to produce spatially uniform electron bunches. Laser pulses at 35 fs, 800 nm and a 3 kHz repetition rate from a titanium sapphire laser at an 84° glancing angle, inside a vacuum chamber at ~10⁻⁸ torr bathe a highly uniform array of ~2200 silicon pillars with a 5-µm pitch. The cathode chip is connected to ground through a picoammeter while the anode, a 0.25-inch plate 3mm above the cathode, connects to a voltage supply (see Figure 1). The cathodes show stable emission and emit over 1.2 pC average charge for over 8-million pulses when excited with 9.5-µJ laser energy with no degradation of the emission characteristic of the cathode. This result shows that silicon-based photontriggered cathodes processed with standard CMOS processes and operated at high vacuum can function for extended periods without performance degradation.

The cathodes are fabricated from single-crystal <100> n-Si 1-10 Ω -cm wafers. The result is massive arrays of pillars (over half a million elements with 5-µm hexagonal packing) capped by tips with under-5-nm average tip radius and less than 1-nm standard deviation (see Figure 2). Through simulation and experiment we have demonstrated that the emitters operate in two distinctive regimens, i.e., the low-electric field multiphoton regime (similar to a typical photocathode), and the high-field quantum tunneling regime (similar to a field emission cathode). Actuation of the devices with laser pulses of 10 μ J or lower results in electron emission with no device degradation.



▲ Figure 1: Experimental set-up with an 800-nm 35-fs laser beam with 80-µm spot size hitting the cathode at 84°.



Figure 2: (a) SEM of high aspect-ratio single-crystal Si pillars with uniform nanosharp tips. (b) Damage to Si tips after exposed to $20.5-\mu J$ laser pulses.

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Field Emission Neutralizers for Electric Propulsion of Small Spacecraft in Low Earth Orbit

A.A. Fomani, A.I. Akinwande, L.F. Velásquez-García Sponsorship: DARPA

Electric propulsion (EP) systems are excellent candidates for small spacecraft since EP systems consume less propellant than chemical rockets. In EP systems such as field emission electric propulsion thrusters (FEEPs), ion engines, and hall thrusters, a beam of positive ions is ejected at high speed to produce thrust. If the ejecting charge is not compensated, the operation of the EP system will negatively charge the spacecraft, reducing the propulsion efficiency and eventually stopping the thruster. Hence, development of robust, low-power, and high-current neutralizers that do not consume propellant is necessary to advance the state of the art of EP systems for small spacecraft. Field emission neutralizers (FENs) are promising candidates because of their low power consumption, high specific current, small size, and lack of propellant consumption. For operation in LEO, neutralizers must withstand long-term operation in environments with oxygen partial pressures of ~5×10⁻⁷ Torr. Carbon nanotube-based FENs could satisfy

these requirements; however, they require biases higher than 600 V for 1 mA emission current.

This work develops arrays of Pt-coated, self-aligned, gated tips as low-voltage FENs for electric propulsion of small spacecraft in low Earth orbit. The neutralizers consist of 320,000 tips with 10 µm pitch and 5-10 nm tip radii; they have an integrated self-aligned gate electrode with 3 µm apertures. The devices emit currents higher than 1 mA at bias voltages as low as 120 V, i.e., similar currents at five-fold less bias voltage and emission area than state-of-the-art CNT neutralizers. The devices have a 2.5-µm-thick gate dielectric to prevent device failure due to dielectric breakdown; the tips are coated with a 10-nm-thick Pt film to improve the tip resistance against ion bombardment and reactive gasses. Continuous emission for 3 hours at pressures of 5×10⁻⁶ Torr in air was demonstrated. Less than 60 V increase in the gateemitter voltage was sufficient to maintain the emission current at 1 mA.



▲ Figure 1: SEM image of Pt-coated self-aligned gated Si tips. The inset shows optical image of a field emitter neutralizer with 0.32 cm² active area consisting of 320,000 gated nanometer-scale tips.



▲ Figure 2: . Continuous field emission at 5×10⁻⁶ and 5×10⁻⁷ Torr in air. Less than 60 V increase in the gate-emitter voltage was sufficient to maintain the current at 1 mA.

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Field Emission Arrays with Integrated Vertical Current Limiters and Self-aligned Gate Apertures

S.A. Guerrera, A.I. Akinwande Sponsorship: DARPA

Field emission cold cathodes are some of the brightest electron sources ever reported, making them an ideal source in a variety of applications, including microscopy, lithography, imaging, and the generation of terahertz and X-ray radiation. Field emission arrays (FEAs) suffer from emitter tip radius variation across the array and sensitivity to the state of the emitting surface, resulting in spatial and temporal variations of emission current. To address these issues, we previously demonstrated that a high-aspect-ratio silicon vertical current limiter (VCL) that is connected in series with each field emitter in a field emission array could regulate the supply of electrons to each emitter and result in uniform emission; however, due to the lack of an integrated extractor gate, these devices operate at high extraction voltages and 99% of the total emitted current is intercepted by the extraction gate. Large extraction gate voltages are required due to the low field factor, β (cm⁻¹), and result in a high Fowler-Nordheim

(FN) slope b_{FN} (V), arising from the large extractor gatetip distance.

To reduce the extractor voltage and enable lowvoltage operation, we report Si FEAs with 1 million individual field emitters that have a 1-micron pitch with integrated VCLs poly-silicon extractor gates. These VCLs are Si pillars that have diameter less than 100 nm and are 10 microns tall, with tip radius under 20 nm. A schematic diagram and circuit diagram are shown in Figure 1 (a),(b). To fill in the gaps between the pillars and to support the self-aligned gate, a novel gap-filling process consisting of silicon dioxide and silicon-rich nitride deposition and chemical-mechanical planarization was employed, resulting in the structure shown in Figure 1 (c). The diameter of the extractor gate aperture is under 200 nm. As shown in Figure 1(d), these devices exhibit turnon voltages less than 20 V and saturation currents of approximately 1 pA / emitter.



▲ Figure 1: (a) Schematic diagram of a Si field emitter with Si VCL. (b) Circuit diagram of the structure. The VCL behaves as an ungated FET. (c) SEM image of a tilted (top) and cross-section view of the completed structure. (d) I-V characteristics (top) and Fowler-Nordheim characteristics of the completed device. The FEA turns on at <20 V and demonstrates current saturation at <30 V.

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Low-Voltage High-Pressure Gas Field Ionizers

A.A. Fomani, L.F. Velásquez-García, A.I. Akinwande Sponsorship: DARPA

Low power consumption, soft-ionization capability, and the potential for operation at high pressures are characteristics desired in gas ionizers for application to portable analytical instruments. Unlike impact ionization techniques, field ionization provides an efficient method for producing stable molecular ions-even from complex organic compounds. Consequently, field ion sources can generate nonfragmented ions for exact measurement of the mass-to-charge ratio of an analyte. These devices are used in various analytical instruments such as field ion mass spectrometers (FIMS) and atom beam microscopes. Other applications include gas chromatography FIMS for analysis of petroleum products and neutron generators for detection of shielded nuclear material and oil-well logging. Despite the attractive features offered by field ion sources, long-term, reliable, and high pressure operation has not been reported due to high voltages (> 500 V) needed for field ionization using the current state-of-the-art devices.

We have developed low-voltage Torr-level gas field ionizers with operating voltages as low as 150 V even for He. which has the highest ionization potential among molecules. The ionizer consists of a large array of Pt-coated self-aligned gated Si tips with radii <10 nm and gate apertures of 3 µm. The tips were designed to generate fields above 20 V/nm at gate-to-tip voltages lower than 200 V while the field at the edge of the gate remains below 0.2 V/nm. A 2.5-µm-thick stack of silicon oxide/silicon nitride was employed as the gate dielectric to limit the field intensity inside the gate dielectric to less than 100 V/ μ m, allowing prolonged operation of the device. Continuous field ionization of He and N₂ for 10⁴ s was achieved at pressures as high as 10 Torr. A slow decay in ion current was observed over time, which can be explained by adsorption of particles at the tip surface. Nevertheless, the original device characteristics can be recovered by operating the device as field emitter in a high vacuum (<10⁻⁷ Torr).



▲ Figure 1: SEM images of the fabricated gated tip arrays employed as gas field ionizers.



▲ Figure 2: Field ionization of helium—ion current vs. tip-to-gate voltage at different pressures.

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Large-Area Field Emission Arrays for High-Current Applications

A.A. Fomani, L.F. Velásquez-García, A.I. Akinwande Sponsorship: DARPA

Gyrotrons, free electron lasers (FELs), and THz vacuum electronic devices require intense high-current electron beams. High-current, high-current-density electron beams are also needed for X-ray generation, pumping of gaseous lasers, and surface treatment of materials. Field emission sources show great promise for these applications as they can produce current densities higher than 10 A/cm² at voltages below 100 V. Despite these promising attributes, the state-of-the art devices have produced currents less than 300 mA due to limitated array size (1–10 mm²) because of fabrication issues that result in failure or severe sub-utilization of the array. The major challenges include low yield of fabrication, large variation in gate and tip dimensions across the array, and point defects in the gate dielectric.

We have developed a high-yield process for fabrication of large-area, self-aligned, gated tip arrays with low sensitivity to processing conditions. The fabricated field emission arrays (FEAs) demonstrate average field factor >10⁶ cm⁻¹ using nanometer-scale tips (radii < 10 nm) surrounded by individual gates with 1.5 µm radius of aperture. This ensures low-voltage operation of the device and a turn-on voltage below 50 V. For reliability a thin Pt layer was deposited over the FEA and a SiO_v/SiN_v dielectric stack thicker than 2.5 μ m was used as the gate insulator. The Pt coating ensures chemical resistivity of the tips against corrosive gasses/ ions, and the thick insulator stack limits the field inside the gate dielectric to < 150 V/ μ m at Gate-Emitter voltages of < 300 V. Our FEAs consisting of 320,000 tips in 0.32 cm² are capable of emitting currents as high as 350 mA at densities of ~1.1 A/cm². The device operation at higher emission currents was prevented due to plasma ignition because of the excessive outgassing of the anode. At low pressures, long-term (~3 hrs) operation not only was possible but also lowered emission voltage and gate current.



▲ Figure 1: SEM images of a fabricated array showing angle-view, cross-section and close-up images of a gated tip, confirming the self-aligned structure of the device and tip radius of less than 10 nm.



▲ Figure 2: Field emission characteristics of an FEA consists of 320,000 Pt-coated and gated tips with radii below 10 nm. Currents as high as 350 mA were emitted at gate-emitter voltage of 300 V.

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Evaporation through Nanoporous Membranes for High Heat Flux

D. Hanks, J. Sircar, Z. Lu, S. Narayanan, D. Antao, B. Barabadi, R. Xiao, R. Raj, R. Enright, E.N. Wang Sponsorship: Avram Bar-Cohen, DARPA MTO

The development of ever more compact electronic circuits has brought the demands for thermal management to unprecedented levels. Although there has been extensive research on single phase and multi-phase cooling in microchannels, evaporative cooling in the thin film regime has the potential to reach an even higher heat flux. We report the design and fabrication of a novel silicon-based evaporation device for direct integration into high power density electronics.

We designed a micro-scale device, relying on the evaporation of a very thin liquid film to dissipate over 1000 W/cm² with an overall temperature difference of less than 30 K. Evaporation occurs in a 200 nm thick silicon membrane patterned with 100 nm pores using interference lithography. The nanopores create a large thin-film evaporation area and generate a large capillary

pumping pressure to supply fluid to the membrane. The membrane is thermally bonded to an arrayed supply network of 4 μ m x 4 μ m microchannels whose walls provide mechanical support and a thermal conduction pathway from the substrate. The substrate is resistively heated, and the temperature is measured with RTDs fabricated with a lift-off pattern. A finite element model is developed to optimize the microchannel and membrane geometry. The convective heat transfer coefficient is modeled by numerically solving governing equations of heat, mass, and momentum conservation at the pore level. Evaporation through nanoporous membranes has the potential for achieving ultra-high heat flux dissipation (5 kW/cm²) for high-performance electronic devices.



▲ Figure 1: Schematic of supported membrane evaporation device. Liquid from manifold is drawn through ridge channels to surface of membrane pores, where evaporation occurs (not to scale). Membrane and manifold are fabricated using SOI wafer, which is then bonded to a wafer with etched ridge structures.







 Figure 2: (a) SEM image of pores etched with interference lithography pattern into silicon.
 (b) SEM image of etched ridge structures. Dark material on top is photoresist used to define etch.
 (c) SEM image of liquid manifold channels etched through membrane and BOX into handle.

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Experimental Investigation of Thin-film Evaporation from Microstructured Surfaces for Thermal Management

S. Adera, D.S. Antao, R. Raj, E.N. Wang Sponsorship: Office of Naval Research with Mark Spector as program manager, National Science Foundation GRFP

Thermal management is a primary design concern for numerous high power density devices such as integrated circuits, electric vehicles, military avionics, photonic devices, and solar energy convertors. This is especially true in the microelectronics industry where the increase in the number of integrated circuits and operating speed has increased the waste heat that is generated at the device footprint from 30 W/cm2 in the 1970's to 100 W/cm2. Moreover, this heat flux is projected to reach 300 W/cm2 in the next few years introducing new challenges in thermal management that has forced the industry to seek advanced cooling solutions. Unfortunately, the widely used conventional single-phase cooling systems are inferior in performance and cannot be used for applications that require removal of high heat fluxes in excess of 100 W/cm2. As a result, state-of-the-art single-phase cooling systems are limited to low heat flux devices and the proposed solution is to use liquid-vapor phase change systems such as thin-film evaporation [1, 2] to make use of the high latent heat of vaporization that can be harnessed during the phase change process.

In this experimental study, we investigated the complex fluidic and thermal transport processes when a thin-liquid film is evaporating from a microstructured surface. We fabricated well-defined microstructured surfaces using contact photolithography and deep reactive ion etching. In addition to offering rich opportunities to manipulate the fluid dynamics, microstructured surfaces in combination with chemical functionalization have long been recognized for enhancing thermal performance in phase-change process. The induced roughness generates capillary pressure for passive liquid transport [3]. The liquid transport was further assisted by incorporating microchannels which reduce the overall flow resistance of the porous media. For integrated heating and temperature measurement, we used electron-beam evaporation and acetone lift-off to create a thin-film heater and sensors. This work elucidates new and innovative techniques to utilize microstructured surfaces for thermal management.



A Figure 1: Test sample. (a) Front, (b) back, and (c) cross sectional view of the schematic of a typical test sample. The overall dimension of the test sample was 3x3 cm² while the microstructured area was only 1x1 cm². The diameter, height, and spacing of the micropillars were 4-12, 10-100, and 10-20 µm, respectively. Liquid was supplied to the microstructured area from the surrounding water reservoir via capillary-wicking. Microchannels were also used to assist the liquid transport by reducing the overall flow resistance of the porous media. The microstructured area was heated using a thin-film heater and the backside temperature was measured using sensors.

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Scalable and Direct Water Purification Technology by Ion Concentration Polarization

B. Kim, H.J. Kwon, V.S. Pham, J. Han Sponsorship: ARPA-E

We demonstrate a scalable and direct water purification technology using ion concentration polarization (ICP). Although nonlinear ICP was shown to generate a strong depletion zone near the ion exchange membrane (IEM), several challenges (power consumption, expandability, etc.) must be overcome for ICP to be a competitive technology in desalination. To resolve and improve them, we propose a modified ICP platform for water desalination by involving two identical cation exchange membranes (CEMs); it demonstrates better salt removal and energy efficiency than conventional electrodialysis (ED), as Figure 1 shows. Between two parallel CEMs, ion depletion/enrichment zones are generated near the CEMs under an electric field. As cations selectively transfer through the CEMs, anions relocate to achieve electro-neutrality, resulting in a decreased/increased concentration in the ion depletion/enrichment zone. Given that the desalted and brine flow streams form on the cathodic and anodic CEM in the main channel,

(a) 2CEM $_{V+}$ с desalted brine flow flow CEM brine desalted α GND (b) ED V+ С desalted AFN flow desalted CEM GND

▲ Figure 1: Desalination/purification strategy. (a) 2CEM and (b) ED. Blue and red arrows indicate movement of cations α + and anions β -. Ion depletion zone d_{de} with low ion concentration occurs at anodic side of CEMs (green lines). Ion enrichment zone d_{en} with high ion concentration occur at other side of CEMs (green dotted lines).

respectively, we can separate and collect each desalted and brine flow by bifurcating the channel at the end. Our technique offers a significant advantage for reducing the number of water purification stages over other conventional technologies since we can obtain desalted flow delivering any charged particles (contaminants) to the brine channel simultaneously.

To visualize the electrokinetic phenomena between the membranes, we fabricated a PDMS-based microfluidic chip with thin channel depth (~0.2mm) and injected a sodium chloride solution mixed with fluorescent dye, as in Figure 2(a). Additionally, to increase system throughput, we built a plastic-based desalination prototype (~1ml/min) by expanding the channel depth and successfully operated it over ten hours, as shown in Figures 2(b) and 2(c). Therefore, we expect our ICP desalination to be a practical technology for water purification, providing both lower energy cost and high throughput.



▲ Figure 2: (a) Fluorescent image of typical desalination process between two cation exchange membranes (~1.2mm). Photograph of (b) high-throughput desalination prototype (~1ml/min) and (c) its long-time operation.

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Electrostatic Precursor Films

S.R. Mahmoudi, K. Adamiak (University of Western Ontario), G.S.P. Castle (University of Western Ontario), K.K. Varanasi

When a liquid spontaneously spreads over a solid surface, a progressive microscopic structure—conventionally known as van der Waals driven precursor film—develops ahead of the moving contact line. Here, we report a new class of electrostatically assisted precursors containing microscopic charged particles. This precursor manifests itself as the late stage of forced-spreading of a macroscopic dielectric film subjected to a unipolar ionic discharge in a gas containing particulates. We put a model forward to predict dynamic behavior of this electrostatic precursor dynamics. The spreading of the precursor film is predicted to be proportional to the square root of exposure time, which is consistent with the ellipsometric measurements.



✓ Figure 1: (a) Figurative description of corona discharge assisted spreading of dielectric films. Thin film interferometry of oil/air interface around apparent contact line of oil film exposed to 2400 s corona discharge exposure (b) in the presence of dust particle (Arizona Road Dust[®]) with a mean size of 5µm. Silicone oil viscosity, surface tension and electrical conductivity were measured to be 50 cst, 21 mN/m, 920 pS/m, respectively at 25°C±2°C.

Continuous-flow Microcalorimetry Using Silicon Microreactors and Off-the-shelf Components

E.W. Lam, M.A. Schmidt Sponsorship: MTL

Calorimetry is an important method for studying the kinetics and energy requirements of chemical and/ or biological reactions. In particular, calorimetry can characterize the heats of reaction (Δ H) to determine the necessary heat transfer requirements when scaling up production, for example whether the system has the appropriate amount of heating/cooling elements to sustain its optimal reaction conditions. There are many products and devices capable of characterizing Δ H, such as differential scanning calorimeters, thermal activity monitors, and isothermal nanocalorimeters; however, these systems utilize fixed volumes of reactants and are inherently incapable of being run in-line with continuous flow without complex modifications.

Unlike traditional calorimeters, this microcalorimeter is designed for continuous flow and to run in-line with an automated microfluidic reaction optimization system with little-to-no modifications. Previously, a similar microcalorimeter was proposed; however, the design had a high heat flux threshold (>50mW), limiting its usefulness to high-energy and/or high-concentration reactions (>1M for reactions where $\Delta H \ge 50 \text{kJ/mol}$). This previous design had several other drawbacks including long thermal time constants due to its large thermal mass and requiring a control (baseline) reaction to be run sequentially with the sample reaction. Our design utilizes a parallel-reactor setup, enabling the baseline and sample reactions to run concurrently and allows for direct measurement between the parallel reactors. This parallel setup reduces the thermal mass and experimental time and results in a predicted 5x increase in thermal sensitivity. As such, the microcalorimeter is capable of characterizing ΔH 's faster than the previously mentioned design while at lower (<1M) concentrations.

Currently, the continuous-flow microcalorimeter consists of two parallel silicon microreactors, one running the chemical reactions and the other running a baseline reaction. The microreactors are sandwiched in between a series of thermoelectric modules and a machined aluminum jig, and the Δ H is measured by heat flux between the microreactors. The microcalorimeter was used to characterize a Paal-Knorr reaction, resulting in the thermoelectric modules measuring a voltage of 3.70±0.27mV, corresponding to a heat flux of 170.7±12.5mW. When running the baseline reaction in both reactors, the system had a noise floor of 0.19mV. Extrapolating the signal to the noise floor, we predict that the microcalorimeter will be capable of measuring heat fluxes as low as 8.6mW.

Our next step for the microcalorimeter is to continue refining the thermal control mechanisms to further improve the heat flux sensitivity. Additionally, we will designed and fabricate a specialized silicon microreactor for the Δ H characterization of solar thermal fuels, molecules designed by our collaborators that are capable of storing solar energy and subsequently releasing the solar energy as heat at a later date. Finally, the system will be inserted into an automated cycling setup to monitor to analyze the stability and cycling longevity of the solar thermal fuels.



▲ Figure 1: Photograph of assembled microcalorimeter. The chiller is powered by pumping 0°C water through the aluminum block. Cartridge heaters connected to a PID controller are used to heat the reactors. Sample and reference inlet/ outlet ports attach to top and bottom of the chiller blocks, respectively.



▲ Figure 2: Voltage of TE module 2 as a function of time when running sample and baseline reactions concurrently. As can be seen, the system takes 5 minutes to reach steady-state and results in average response of 3.70mV, which corresponds to an average heat flux of 170.7mW.

MEMS Two-stage Diaphragm Vacuum Pump

E. Newton, H. Li, M. Schmidt Sponsorship: DARPA

Portable sensing devices such as microscale mass spectrometers need vacuum pumping to lower samples at atmospheric pressure to the desired measurement pressure range. Further improvements for MEMS accelerometers, gyros, and other resonant sensors require internal pressures as low as a few microtorr, which is possible only with active vacuum pumping. While these pressures are easily achieved using macroscale vacuum pumps, the larger pumps are not portable, negating the benefits gained from making small, low-power sensors in the first place. To realize the full potential of portable sensors, a chip-scale vacuum pump needs to be developed.

We have developed what is to our knowledge the first two-stage MEMS displacement pump with integrated electrostatic actuation. Two pump stages, along with an efficient layout that minimizes dead volume and a new actuation scheme should enable it to reach pressures below 30 Torr. Actuation is achieved by electrostatically zipping a thin flexible membrane down onto a stiff curved electrode. This actuator topology allows for large displacements and large forces at relatively low voltages (< 100 V). An image of a fabricated two stage micropump is shown in Figure 1 below.

We have developed two methods for producing curved electrodes in MEMS devices: 1) hot air trapped during wafer bonding expands with enough pressure to plastically deform thin silicon membrane and 2) strain induced when epoxy cures can pull a membrane into a curved shape. Using the plastically deformed electrodes, we have demonstrated that we can reliably and repeatably zip a thin membrane at low voltages and we have mapped out how the critical voltage depends on the deformation magnitude and the oxide thickness. This is shown in Figure 2 below. These accomplishments have helped us understand the fabrication process and physics of device operation. We plan in the next year to further examine the reliability of plastic deformation in our process, testing the actuators at low pressures, and we hope to fabricate a working micropump that is capable of achieving pressures as low as 30 Torr.



▲ Figure 1: Microfabricated two-stage vacuum pump. Top and bottom views of the pumps are shown (top) as are internal fluidic connections (bottom).



▲ Figure 2: Pull-in voltages for 6 mm diameter curved electrodes with a 25 µm thick zipping membrane.

FURTHER READING

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A Tabletop Fabrication System for MEMS Development and Production

P.A. Gould, M.D. Hsing, K.K. Gleason, M.A. Schmidt Sponsorship: MTL

A general rule of thumb for new semiconductor fabrication facilities (fabs) is that revenues from the first year of production must match the capital cost of building the fab itself. With modern fabs routinely exceeding \$1 billion to build, this rule serves as a significant barrier to entry for small entities seeking to develop or commercialize new semiconductor devices. The barrier is especially formidable for those groups whose devices target smaller market segments or those which require exotic materials or nontraditional process sequences. The foundry fab model has arisen partially to overcome this inefficiency, but to remain profitable, these foundries typically offer only a few standardized processes that limit customer customization. The limited diversity afforded by these foundries can make some devices with smaller market sizes economically viable, but many devices (particularly in the MEMS sector) require process customization beyond the level currently offered by commercial foundries.

To address these problems, we are working to create a suite of tools that processes 1-2" substrates. This suite of tools (known colloquially as the 1" Fab) takes advantage of modern processing techniques, but at a fraction of the normal cost. We anticipate a full set of tools for product development and small-scale production to cost ~\$1 million and require <50 ft² of space (roughly a large conference table, see Figure 1 for a rendering), compared to >\$1 billion and >50,000

ft² for a typical 8" fab. In addition to the reductions in equipment cost and required space, a 1" Fab also uses significantly less total materials and reagents, requires far less energy to operate, and lessens the environmental impact of fabrication. The total throughput possible in a 1" Fab certainly cannot match a typical 8" fab, but the vast majority of devices that are unsuitable for traditional foundries simply do not require this advantage in production rate. For these devices, the cost savings of the 1" Fab platform and its ability to quickly prototype designs far outweigh any expansion in production schedules.

We are currently developing a deep reactive ion etcher (DRIE) tool for the 1" Fab. DRIE tools are used to create highly anisotropic, high aspect-ratio trenches in silicon—a crucial element in many MEMS processes. The modularized design of our DRIE system can be easily adapted to produce other plasma-based etching and deposition tools (like PECVD and RIE). Our DRIE, shown in Figure 2, is the about the size of a large microwave oven and costs just a small fraction of a commercial system. We have demonstrated etch rates of >6 microns per minute and anticipate achieving etch rates of 10 μ m/ min with further process tuning. In the coming year we will continue to optimize our DRIE design and begin developing PECVD and high-temperature process (e.g. oxidation and LPCVD) tools for the 1" fab.



▲ Figure 1: Computer rendering of envisioned 1" Fab toolset. A complete set of tools as shown would allow for standard silicon processing: maskless lithography system, wafer bonder, tabletop SEM, LPCVD / oxidation / annealing furnace, DRIE, PECVD, and a sputtering / evaporation tool.



▲ Figure 2: 1" Fab Tabletop DRIE. The associated labeled parts are as follows: A) process gas inlet, B) viewport, C) RF coil, D) alumina chamber, E) liquid cooled and RF powered wafer chuck, F) vacuum pump inlet.

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