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FACULTY PROFILES

A.I. Akinwande
Flat panel displays, Vacuum Microelectronics and its application to flat panel displays, RF power sources, and sensors. Wide bandgap semiconductors and applications to flat panel displays, UV emitters and RF power sources.

G. Barbastathis
Biomedical design instrumentation; precision engineering robotics; volume holographic architectures for data storage, color-selective tomographic imaging, and super-resolving confocal microscopy; interferometric surface characterization; and adaptive micro-opto-mechanics. Optical MEMS.

M. Bazant
Research focuses on transport phenomena in materials and engineering systems, especially diffusion coupled to fluid flow. My group is currently studying granular flow in pebble-bed nuclear reactors, nonlinear electrokinetic flows in microfluidic devices, ion transport in thin-film lithium batteries, and advection-diffusion-limited aggregation.

S.N. Bhatia
Research focuses on applications of micro- and nanotechnology to tissue repair and regeneration, specifically in development of microfabrication tools to improve cellular therapies for liver disease.

D.S. Boning
Semiconductor manufacturing. Modeling and control of chemical mechanical polishing. Variation modeling and reduction in fabrication processes, devices, and interconnects. Run by run and feedback control for quality and environment in semiconductor fabrication. Software systems for distributed and collaborative computer aided design and fabrication.

A.P. Chandrakasan

G. Chen
Micro- and nanoscale heat transfer and energy conversion with applications in thermoelectrics, photonics, and microelectronics; nano-mechanical devices and micro-electro-mechanical systems; radiation and electromagnetic metamaterials.

M. Culpepper
Research focuses on precision interfaces, precision manufacturing, design for manufacturing, applying precision principles as enabling technologies in multi-disciplinary product design: electronic test equipment, automotive systems, precision compliant mechanisms.

L. Daniel
Research focuses on engineering design applications to drive research in simulation and optimization algorithms and software, design of microfabricated inductors.

P. Doyle
Understanding the dynamics of single polymers and biomolecules under forces and fields, lab-on-chip separations, polymer rheology, DNA electrophoresis in microdevices. Superparamagnetic colloids. Brownian Dynamics simulations of complex molecules. Microheology of biopolymers.

A. Epstein
Smart engines, turbine heat transfer and aerodynamics, advanced diagnostic instrumentation, turbomachinery noise, environmental impact of aircraft.

D. Freeman
Biological micromechanics, MEMS, light microscopy and computer microvision.

M. Gray
Microfabricated devices for use in diagnostic medicine and biological research. Particle and fluid analysis of flowing media using absorbance and fluorescence techniques as a means for understanding cell or organism metabolism and phenotypic expression.

J. Han
BioMEMS, biomolecule analysis, micro/nanofluidics, micro-analysis systems.

J. Jacobson
Development of processes for directly and continuously printing communication, computation, and displays onto arbitrary substrates. Electronic control of biomolecules.

K.F. Jensen
S.-G. Kim  
Systems Design and Manufacturing, MEMS for optical beam steering, microphotonic packaging and active alignment, micro power generation, massive parallel positional assembly of nanostructures, and nano actuator array.

J.H. Lang  
Analysis, design and control of electromechanical systems. Application to traditional electromagnetic actuators, micron scale actuators and sensors, and flexible structures.

C. Livermore  

S. Manalis  
Application of micro- and nanofabrication technologies towards the development of novel methods for probing biological systems. Current projects focus on electrical and mechanical detection schemes for analyzing DNA, proteins, and cells.

P. Matsudaira  
The Matsudaira Lab research is focused in two areas: the mechanics of the cytoskeleton, and miniaturized ultra-fast bioanalytical devices. Cytoskeleton research involves the study of the Limulus acrosomal reaction as well as Vorticella motion. Our biological devices research is focused on bringing MEMS technology to benefit biomedical research.

D.J. Perreault  
Analysis, design, and control of cellular power converter architectures. DC/DC Converters for dual-voltage electrical systems. Electrical system transient investigation. Exploration of non-conventional electricity sources for motor vehicles.

M.A. Schmidt  

A.H. Slocum  
Precision Engineering; Machine Design; Product Design.

C.V. Thompson  
Processing, structure, properties, performance, and reliability of thin films and structures for micro- and nano-devices and systems. Reliability and Interconnect.

T. Thorsen  
Integrating microfluidic design and fabrication techniques, electronics and optics with biochemical applications. Optimizing channel dimensions, geometry, and layout to generate 3-D fluidic networks that are functional and scalable. Interface development to combine microfluidic technologies with pneumatic valves, MEMS-based detector systems, and software-based data acquisition and interpretation, creating devices for fundamental research and diagnostic applications.

H.L. Tuller  
Characterize and understand key electronic, microstructural, and optical properties of advanced ceramic materials. Fabrication and characterization of crystals, ceramics and glasses for electronic devices, lasers, electrochemical energy conversion, sensors and actuators.

J. Voldman  

B.L. Wardle  
Power MEMS microhydraulics, structural health monitoring, nanocomposites, damage resistance/tolerance of advanced composite materials, cost modeling in the structural design process, conversion of technology to value.

J. White  
Theoretical and practical aspects of numerical algorithms for problems in circuit, device, interconnect, packaging, and micromechanical system design; parallel numerical algorithms; interaction between numerical algorithms and computer architecture.
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Microfluidic Synthesis and Surface Engineering of Colloidal Nanoparticles

Microreactor Enabled Multistep Chemical Synthesis

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Crystallization in Microfluidic Systems

Microreactors for Synthesis of Quantum Dots

A Large Strain, Arrayable Piezoelectric Microcellular Actuator

MEMS Pressure-sensor Arrays for Passive Underwater Navigation

A Low Contact Resistance MEMS-Relay

Fast Three-Dimensional Electrokinetic Pumps for Microfluidics

Carbon Nanotube - CMOS Chemical Sensor Integration

An Energy Efficient Transceiver for Wireless Micro-Sensor Applications

Combinatorial Sensing Arrays of Phthalocyanine-based Field-effect Transistors

Nanoelectromechanical Switches and Memories

Integrated Carbon Nanotube Sensors

Organic Photovoltaics with External Antennas

Integrated Optical-wavelength-dependent Switching and Tuning by Use of Titanium Nitride (TiN) MEMS Technology

Four Dimensional Volume Holographic Imaging with Natural Illumination

White Light QD-LEDs

Organic Optoelectronic Devices Printed by the Molecular Jet Printer

Design and Measurement of Thermo-optics on Silicon
A MEMS Electrometer for Gas Sensing

J. Lee, L.F. Velásquez-García, A. Seisha, A.I. Akinwande
Sponsorship: DARPA

The DARPA-funded micro gas analyzer program aims to develop portable, low-power, fast, and reliable gas analyzer technology for a wide range of applications. The system architecture of the gas analyzer contemplates a MEMS electrometer at the end of the system. The electrometer characterizes the ionized species that are filtered by the quadrupole. The sensitive element of the electrometer is a MEMS structure embedded in a feedback loop of a precise oscillator circuit. The electrometer has a comb drive that sets the electrometer to oscillate. Shifts in the oscillation frequency are related to changes in the capacitance of the electrometer due to ion interception. The resolution of the device is estimated at 100 e/√Hz in vacuum [1]. Figure 1 shows a fabricated MEMS electrometer. Figure 2 shows the experimental data of one of these MEMS electrometers, in air. The experimental resonant frequency is 6.2 kHz, and the conversion gain was estimated at $2 \times 10^9$ V/C (theoretical value is $7 \times 10^9$ V/C). Current research focuses on implement lock-in detection, which will remove the noise from the drive signal because the output has twice the frequency of the input signal.

Figure 1: A micro-fabricated MEMS electrometer. The comb drive (central part) sets the electrometer in oscillation. Changes in the variable capacitors (comb structures at both sides of the central comb drive) cause shifts in the oscillation frequency that are directly related to the ion current that impact the MEMS.

Figure 2: Voltage versus charge characteristics for the MEMS quadrupole in air.

REFERENCES
A Single-Gated CNT Field-Ionizer Array with Open Architecture

L.F. Velasquez-Garcia, L.Y. Chen, A.I. Akinwande
Sponsorship: DARPA

The micro gas analyzer project aims to develop the technology for portable, real-time sensors intended for chemical warfare and civilian air purity control. The device is composed of four micro-fabricated subsystems: an ionizer, a mass filter based on a quadrupole array [1], an electrometer [2], and a positive displacement pump [3]. We are developing a single-gated fieldionizer array based on gated carbon nanotubes (CNTs). The devices achieve species ionization by tunneling of outer shell electrons due to the presence of high electric fields that the device sets. We use CNTs as field enhancers because of their small radii and high aspect ratio while the gate proximity ensures high fields at low voltage. State-of-the-art ionizers use electron-impact ionization (thermionic cathodes), incurring in excessive power consumption, low current, current density, ionization efficiency, and short lifetime. The field-ionizer arrays (Figure 1) are able to soft-ionize species, thus achieving molecule ionization. The reliability and lifespan of the field-ionizer arrays are larger than the corresponding values for electron-impact ionizer arrays because the CNTs are biased at the highest potential in the circuit, thus making it unlikely for ionized molecules to back-stream. Figure 2 shows two SEM pictures of a single-gated CNT array that implements a selective CNT-growth process. This process reduces the fabrication complexity of the device because it grows CNTs from an un-patterned catalyst (Ni). Current research efforts concentrate on improving the device and data acquisition, including benchmarking the performance of the ionizer in low-pressure oxidizing environments.

References
A MEMS Quadrupole that Uses a Meso-scaled DRIE-patterned Spring Assembly System

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

The DARPA-funded micro gas analyzer program aims to develop portable, low-power, fast, and reliable gas analyzer technology for a wide range of applications. One of the subsystems of the gas analyzer is a mass filter. An array of micro-fabricated quadrupole mass filters is being developed for this purpose. The quadrupoles will sort out the ions based on their specific charge. Both high sensitivity and high resolution are needed over a wide range of ion masses, from 20 to 200 atomic mass units. In order to achieve this performance, multiple micro-fabricated quadrupoles, each operating at a specific stability region and mass range, are operated in parallel. The proof-of-concept device is a single, linear quadrupole that has a micro-fabricated mounting head with meso-scaled DRIE-patterned springs. The mounting head allows micron-precision hand assembly of the quadrupole rods [1]—critical for good resolution and ion transmission. The micro-fabricated mounting head can implement quadrupoles with a wide range of aspect ratios for a given electrode diameter. There are currently two versions of the mounting head, able to interact with rods of diameters equal to 1588 and 559 micrometers. The choice of electrode diameter results from pondering the dimensional uncertainties and alignment capabilities with respect to the expected resolution and transmission goals. Figure 1 shows an assembled MEMS quadrupole, including some detail of the spring structure near the quadrupole transmission region. The quadrupoles that have been implemented so far span the aspect ratio range from 30 to 60. Figure 2 shows the experimental data of one of these quadrupoles on a FC-43 sample, where a mass resolution of 2 amu and a full mass range of 200 amu are demonstrated, while using a 1.2-MHz RF power supply to drive the quadrupole. Current research efforts concentrate on developing RF power supplies of higher frequency to obtain better performance from the same device.

Figure 1: A micro-fabricated quadrupole with electrode diameter equal to 559 micrometers, near a 1-cent coin for size reference. The micro-fabricated part of the device is the square base, which contains a system of meso-scaled DRIE-patterned springs. The structure of the quadrupole spring head near the transmission region is shown in a set of superimposed IR microscope pictures (lower left corner). The electrodes occupy the four cavities (A) that surround the axis of the quadrupole (B). The ion transmission occurs through the latter region.

Figure 2: Experimental characterization of a MEMS quadrupole, using the compound FC-43 to get peaks in the 1 – 200 amu mass range. The resolution is estimated at 2 amu, using an 1.2 MHz RF power supply. On the upper right corner there is a zoom of the data near 200 amu.

REFERENCES
The need for understanding the trophodynamics of the ocean has led to the development of several instruments for monitoring plankton communities, critical indicators of the ocean's health and the base of the aquatic food chain. The three competing methods for plankton observation utilize direct, acoustic, and optical sampling techniques; however, none of the current systems can provide the complete data set required for predictive modeling capabilities. The goal of this project is to develop a small, low-power, digital holographic imaging (DHI) system that allows for in situ monitoring of plankton and other aquatic communities. This system allows microbiologists to collect high-resolution, spatio-temporal data on species-specific population structures. In addition to biological studies, the DHI camera can be utilized in diverse areas such as medical analysis, quality control inspection, and MEMS device characterization.

DHI uses a digital sensor to record holograms, formed by the interference pattern between a reference wave and a field produced by scattered light from an illuminated object. The illumination source is coherent and typically provided by a laser. The recorded images are processed on a computer to reconstruct the original object field at a given axial location [1]. From the reconstructed images, information about the object such as morphology, topology, and 3D coordinates can be computed throughout a large sample volume. In addition, velocity and 3D trajectories are available under slightly modified methods.

Experiments have focused on biological applications, including marine and microbial organisms ranging from 5 to 2000 microns. In addition to the inline configuration (Figure 1), several setups have been implemented to explore smaller scales, including the use of spherical reference waves, 4f telescopes, and microscope objectives. Figure 1 shows our compact benchtop prototype DHI camera, currently being developed to be used as a sea-going instrument for deep-sea microbiology. Using a lens-free spherical configuration with a working distance of 50 mm, all lines on a 1951 USAF resolution target can be resolved, down to 2.2 microns in width. A 4f system was used to track the trajectories of 7 micron algae over several seconds. Small plankton, 50 to 500 microns long, have been imaged using all three setups with excellent clarity. Figure 2 shows a reconstruction from an inline configuration of an adult copepod. Future work includes incorporating the DHI camera into an underwater vehicle. Additional work will focus on tracking small particles under turbulent flow conditions.

REFERENCES
Multi-Axis Electromagnetic Moving-Coil Microactuator

D.S. Golda, M.L. Culpepper

Sponsorship: NSF

Electromagnetic (EM) micro-actuators are becoming increasingly important in micro-systems requiring moderate forces operating over a large range of motion. The applications that benefit from the performance advantages of EM micro-actuators include micro-scanning systems, micro-fluidic pumps, and positioning systems. Advantages of electromagnetic actuation over other classes of micro-actuators include low-voltage operation, moderate power density, large operating distances, linear response, multi-axis capability, and high bandwidth [1]. This work leverages the advantages of EM interactions to design a moving-coil micro-actuator that enables two-axes actuation with moderate forces (10+ mN) over large operating distances (10+ micrometers) at moderate mechanical frequencies (1+ kHz) using assembled permanent magnet field sources.

The two-axes electromagnetic actuator consists of moving coils suspended on compliant silicon flexure springs above an array of 3 rectangular permanent magnets, as shown in Figure 1. The phase of the stacked coils results in Lorentz forces that are independently controllable in-the-plane and out-of-the-plane. The coil-spring fabrication scheme includes electroplating of copper coils, followed by a deep reactive-ion etch (DRIE) to pattern and release the compliant springs. Millimeter-sized permanent magnets are then aligned to the spring layer using an alignment chip. Successfully fabricated micro-coil structures have been shown to sustain current densities over 1000 Amps per square millimeter.

A quasi-analytic electromagnetic force model for the device has been developed and experimentally validated against a centimeter-size bench-level prototype actuator. Figure 2 shows the predicted lateral-actuator force per coil-footprint versus current input for a typical actuator with 900-µm² coil cross section. The actuator will be implemented in a high-speed meso-scale nano-positioner with applications in nano-fabrication and scanning-probe microscopy. When equipped with this micro-actuator, the nano-positioner is expected to be able to position millimeter-sized samples in six axes of motion (x, y, z, tip, tilt, yaw) with repeatability better than 10 nanometers at frequencies greater than 1 kHz.

**REFERENCES**

Fluid interfaces provide unique opportunities for microfluidic and nanofluidic systems. Applications range from microscale heat exchangers and miniature fuel cells to microreactors for materials synthesis. Multiphase flow in such devices can be challenging, as the interfacial forces naturally favor axisymmetric geometries that are difficult to microfabricate. The advantages of surface tension dominated microfluidics include a much richer dynamic flow behavior and enhancement of heat and mass transfer by creating secondary flows. These advantages offer many uses beyond enabling gas-liquid and fluid-solid reactions [1].

In particular, we are interested in segmented flow of gas and liquid in hydrophilic channels. Figure 1 shows several key features of this flow for reaction purposes. The presence of bubbles reduces the amount of dispersion of liquid flowing through the channels, ensuring that reactants and products spend a uniform amount of time in the system. For nanoparticle synthesis in microfluidic networks, a uniform residence time distribution translates into narrowly distributed particle sizes [2-3]. Liquid segments are efficiently mixed by circulation motion and gas bubbles are separated from microchannel walls by only a thin film (thickness < 1 µm). Thin films reduce mass transfer resistance to components immobilized on the walls, such as catalysts [4] or analytical reagents and antibodies. We are also interested in the dynamics of multiphase flow through microchannels that are populated with a forest of micropillars (diameters: 50 µm – 100 µm). The observed flow patterns (Figure 2) connect to fundamental studies of flow in porous media and to catalysis. Gas-liquid and liquid-liquid flow patterns and their dynamics are determined in pulse-laser fluorescent micrographs and with microscale particle image velocimetry (PIV) measurements. Characteristics of such three-phase systems, such as persistent static fractions, axial dispersion and mixing, are compared with multiphase flow in macroscopic unstructured beds and porous media.

![Figure 1: Measurement of axial dispersion in single-phase (green curve) and segmented flow (red curve) through a 1.5 m long microchannel (300 µm wide, 300 µm deep) that is soft-lithographically patterned. To reduce background fluorescence in the integrated spectroscopy measurement, carbon black is dispersed in the PDMS, prior to molding.](image1)

![Figure 2: Microchannel (width 1 mm, depth 300 µm) populated with 100 µm wide micropillars. The left inset shows the integrated gas-liquid feeding system. On the right side, fluorescent micrographs of instantaneous gas (dark) and liquid (bright) flow patterns between pillars are shown.](image2)

REFERENCES


Microfluidic Synthesis and Surface Engineering of Colloidal Nanoparticles

S.A. Khan, K.F. Jensen
Sponsorship: Microchemical Systems Technology Center

Metal oxide colloidal particles such as silica (SiO$_2$) and titania (TiO$_2$) have many diverse applications ranging from catalysis, pigments and photonic band-gap materials to health care products. There has also been considerable research interest over the last decade in fabricating core-shell materials with tailored optical and surface properties. Core-shell particles such as titania-coated silica often exhibit improved physical and chemical properties over their single-component counterparts and hence are potentially useful over a broader range of applications. Newer methods of engineering such materials with controlled precision are required to overcome the difficulties with conventional production techniques, which are limited to multi-step batch processes. We have developed microfluidic routes for synthesis and surface-coating of colloidal silica and titania particles.

The chief advantages of a microfluidic platform are precise control over reactant addition and mixing and continuous operation. Microfluidic chemical reactors for the synthesis and overcoating of colloidal particles are shown in Figure 1a and Figure 1b, respectively [1-2]. Figure 2a is an SEM micrograph of silica particles synthesized in a microreactor (Figure 1a) operated in segmented gas-liquid flow mode. Figure 2b shows a silica nanoparticle coated with a thick shell of titania. We have also fabricated integrated devices combining synthesis and overcoating to enable continuous multi-step synthesis of core-shell particles.

Here are the references:


As a demonstration of how microsystems can enable quantitative study and improved production of chemistries that have been too hazardous to pursue via traditional means, the kinetics of direct sodium nitrotetrazolate (NaNT) synthesis were characterized and a microsystem for its commercial production has been constructed (Figure 1). A PDMS modular microreactor system capable of both multi-step synthesis and rapid scale-out was constructed. This system minimized the necessary volume of the unstable diazonium intermediate, enabling the study of NaNT, an energetic material used in the construction of fire suppression systems that was too dangerous to test with traditional techniques. In the direct synthesis of NaNT, 5-aminotetrazole (5-AT) reacts with nitrous acid to produce the diazonium intermediate that, in a second reaction, undergoes a Sandmeyer type reaction that displaces the diazonium group by the nitrite ion. The rapid mixing and safety advantages of microsystems were incorporated into a flexible architecture, presenting an improved ability to safely probe the conditions of the reaction. The modular design of this system also enabled the same set of modules to be rearranged as parallel reactor chains for small-scale production. A second generation microsystem was constructed from silicon micromixer modules (Figure 2); this micro-system is not only more robust than the PDMS design but also capable of accommodating higher flow rates (>2 mL/min) and higher temperatures. This system allows higher throughput and longer operational lifetimes and is currently being optimized for use as a full-scale production platform.
Integrated Microreactor System

H.R. Sahoo, E.R. Murphy, A. Guenther, N. Zaborenko, K.F. Jensen
Sponsorship: Deshpande Center for Technological Innovation

The realization of integrated microchemical systems will revolutionize chemical research by providing flexible tools for rapid screening of reaction pathways, catalysts, and materials synthesis procedures, as well as faster routes to new products and optimal operating conditions. Moreover, such Microsystems for chemical production will require less space, use fewer resources, produce less waste, and offer safety advantages. The need for synthesizing sufficient quantities of chemicals for subsequent evaluation dictates that microchemical systems are operated as continuous systems. Such systems require fluid controls for adjusting reagent volumes and isolating defective units. The integration of sensors enables optimization of reaction conditions as well as the extraction of mechanistic and kinetic information.

We are developing integrated microchemical systems that have reactors, sensors, and detectors with optical fibers integrated on one platform. We are exploring new approaches for connecting modular microfluidic components into flexible fluidic networks. Real-time control of reaction parameters using online sensing of flowrate, temperature, and concentration allows for precise attainment of reaction conditions and optimization over a wide range of temperatures and flow-rates. The multiple microreactors on the system can be used together to give higher throughputs or they can be used independently to carry out different reactions at the same time. Figure 1 shows a schematic of an integrated microreactor platform along with an early stage microreactor “circuit board” [1].

![Figure 1: Schematic of an integrated microreactor platform along with an early stage microreactor “circuit board” [1].](image)

REFERENCES

Microfluidic systems offer a unique toolset for discovering new crystal polymorphs and for studying the growth kinetics of crystal systems because of well-defined laminar flow profiles and online optical access for measurements. Traditionally, crystallization has been achieved in batch processes that suffer from non-uniform process conditions across the reactors and chaotic, poorly controlled mixing of the reactants, resulting in polydisperse crystal size distributions (CSD) and impure polymorphs. This reduces reproducibility and manufactures products with inhomogeneous properties. The short length scale in microfluidic devices allows for better control over the process parameters, such as the temperature and the contact mode of the reactants, creating uniform process conditions. Thus, these devices have the potential to produce crystals with a single morphology and a more uniform size distribution. In addition, microfluidic systems decrease waste, provide safety advantages, and require only minute amounts of reactants, which is most important when dealing with expensive materials such as pharmaceutical drugs.

Figure 1 shows a microfluidic device used for crystallization and Figure 2 shows optical images of different shapes and sizes of glycine crystals produced in reactor channels. A key issue for achieving continuous crystallization in microsystems is to eliminate heterogeneous crystallization—irregular and uncontrolled formation and growth of crystals at the channel surface, which ultimately clogs the reactor channel. We have developed a sheath flow microcrystallizer using microfabrication and hot embossing of poly(dimethylsiloxane) (PDMS) and cyclic olefin copolymer (COC) to prevent heterogeneous crystallization. We are currently working on integrating an online spectroscopy tool for in situ polymorph detection. Our ultimate goal is to develop an integrated microfluidic system for continuous crystallization with the ability to control polymorphism and online detection.
Microreactors for Synthesis of Quantum Dots

B.K.H. Yen, A. Günther, M.A. Schmidt, M.G. Bawendi, K.F. Jensen
Sponsorship: Microchemical Systems Technology Center, NSF, ISN

We have fabricated a gas-liquid segmented flow reactor with multiple temperature zones for the synthesis of quantum dots (QDs). In contrast to single-phase flow reactors, the segmented flow approach enables rapid mixing and narrow residence-time distributions, factors which have a strong influence on the ultimate QD size distribution. The silicon-glass reactor accommodates a 1-m long reaction channel (hydraulic diameter ~400-µm) and two shallow side channels for collecting reaction aliquots (Figure 1). Two temperature zones are maintained, a heated reaction region (>260°C) and a cooled quenching region (<70°C). As a model system, monodisperse CdSe QDs with excellent optical properties were prepared using the reactor. Cadmium and selenium precursor solutions are delivered separately into the heated section. An inert gas stream is introduced further downstream to form a segmented gas-liquid flow, thereby rapidly mixing the precursors and initiating the reaction.

The reaction is stopped when the fluids enter the cooled outlet region of the device. Under conditions for a typical synthesis, the gas and liquid segments are very uniform (Figure 2a-b), and the QDs produced in the reactor possess narrow spectral features, indicative of monodisperse samples. The narrow particle size distributions arise directly from the enhanced mixing and narrow residence-time distribution realized by the segmented flow approach. Furthermore, the QD size can be tuned without sacrificing monodispersity by varying the Cd and Se precursor flow rates. In Figure 2d, the Se/Cd molar ratio was varied while keeping the total liquid and gas flow rates constant. Decreasing Se/Cd results in a substantial red shift of the QD effective band-gap (first absorption feature and photoluminescence peak), corresponding to larger QD diameters.

**REFERENCES**


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Figure 1: Diagram of the reactor with heated reaction and cooled outlet regions. A through-etch section ensures that the two regions are thermally isolated. Channels etched in silicon (0.65-mm thick) were first passivated with an oxide layer (0.5-µm) and then sealed with an anodically bonded Pyrex wafer (0.76-mm thick).

Figure 2: Images of the a) heated inlet and b) main channel sections of device during synthesis. Red segments: CdSe QD reaction solution. Dark segments: Ar gas. c) Time-exposure image of the cooled outlet region under UV irradiation. At reaction temperature (260°C), the QD photoluminescence (PL) is completely quenched, but once the fluid reaches the cooled region (<70°C), yellow PL is observed. d) The QD absorbance (solid) and PL (dotted) spectra obtained by varying the precursor feed ratio. The Se/Cd molar ratio is indicated.
A Large Strain, Arrayable Piezoelectric Microcellular Actuator

Z.J. Traina, S.-G. Kim
Sponsorship: Korean Institute of Metallurgy and Manufacturing (KIMM)

To provide a competitive actuating solution, micro-electromechanical-systems (MEMS)-based actuators need low operating power and form factors. Piezoelectrics provide substantially higher work-output/volume for a given voltage, when compared to other actuating solutions. A bow amplifier constructed of SU-8 beams and short length flexural pivots has been designed [1] and has demonstrated an amplification ratio of greater than 10:1. Current research focuses on increasing this amplification ratio and achieving the goal of 10% axial strain, while reducing parasitic out-of-plane bending inherent in the current fabrication process.

The overall goal of this project is to array one such actuator massively in series and in parallel in order to create a macro-scale, muscle-like actuator. Such a device would have widespread applications in mobile robotics, medicine, and aero/astronautics, where low power, high efficiency, and small form factors might be required.

\[ \text{REFERENCES} \]

MEMS Pressure-sensor Arrays for Passive Underwater Navigation
V.I. Fernandez, S.M. Hou, F.S. Hover, J.H. Lang, M.S. Triantafyllou
Sponsorship: NOAA: MIT Sea Grant College Program

MEMS pressure sensors have had broad applications in fields such as mining, medicine, automobiles, and manufacturing. Another application to be explored is in underwater vehicular navigation. Objects within a flow generate pressure variations that characterize the objects’ shape and size. Sensing these pressure variations allows the unique identification and location of obstacles for navigation (Figure 1). This concept is inspired by existing biological systems. Fish have such a sensory lateral line, which they use to monitor all aspects of their hydrodynamic environment, including obstacles [2,5].

We propose to develop low-power sensors that passively measure dynamic and static pressure fields with sufficient resolution to detect objects generating the disturbance. We will also develop processing schemes that use the information from the sensors to identify objects in the flow environment. These sensors and processing software emulate the capabilities of the lateral line in fish. While active acoustic means can be used for object detection, the process is power-intensive, and depends strongly on the acoustic environment. A simpler alternative is to use a passive system that can resolve the pressure signature of obstacles. The system consists of arrays of hundreds or thousands of piezoresistive pressure sensors fabricated on etched silicon and Pyrex wafers [1,3,4,6] with diameters around 1 mm; the sensors are arranged over a flat or curved surface in various configurations, such as a single line, a patch consisting of several parallel lines (Figure 2), or specialized forms to fit the hull shape of a vehicle or its fins. The sensors will be packaged close together at distances of a few millimeters apart in order to resolve pressure and flow features near the array spacing, which in turn can be used to identify the overall features of the flow.

REFERENCES
A low contact resistance MEMS-relay featuring highly parallel and planar oblique contacts has been fabricated and is currently being tested. The contacts are etched in silicon using a potassium hydroxide (KOH) solution. An offset between the wafer-top and the wafer-bottom KOH masks produces the oblique contact geometry schematically shown in Figure 1A.

In contrast, many prior art MEMS devices [1-3] have rough, non-complementary contacts. As these surfaces touch, they do so in a small number of high points, as shown in Figure 1B, which significantly reduces the effective contact area and leads to a high contact resistance and a low current carrying capacity. Additionally, vertical contacts are prone to poor metallization, which further affects the device's contact resistance. Our MEMS-relay, shown in Figure 2, is composed of a compliant mechanism (B), a pair each of engaging (C) and disengaging (D) rolling-point “Zipper” actuators [4-5], and a pair of planar and parallel contacts (E). The relay is fabricated by a combination of deep reactive ion etching (DRIE) and KOH etching. Nested masks are used to pattern both wafer-through etches. Low stress silicon nitride (Si$_3$N$_4$), which will later be used as a KOH mask, is patterned initially on both sides of the device wafer. A silicon oxide film is deposited on the KOH mask. The compliant mechanism and actuators are then etched through DRIE and a second Si$_3$N$_4$ film is deposited. The second Si$_3$N$_4$ film is patterned using a “shadow” (through-etched) wafer as a mask. The oxide is selectively etched to reveal the buried nitride mask. The contacts are etched in KOH solution. Both Si$_3$N$_4$ and oxide films are stripped and a thermal oxide, which insulates both the electrostatic actuators and the relay contacts from the rest of the device, is grown. Gold is evaporated over both sides of the insulated contacts and the device wafer is anodically bonded to a Pyrex handle wafer. Experimental pull-in and drop-out voltages of 70 V and 40 V, respectively, agree with the model. Contact travel of 50 µm prevents arcing as the load circuit is switched on and off. A contact resistance of 50 mΩ was demonstrated by our group using an externally actuated structure as a proof of concept for the contact design [4]. Our group continues to develop these MEMS relays for power applications.

**REFERENCES**


Fast Three-Dimensional Electrokinetic Pumps for Microfluidics

J.P. Urbanski, J. Levitan, M. Bazant, T. Thorsen
Sponsorship: ISN

Electrokinetic pumps are attractive for portable and flexible microfluidic analysis systems, since they operate without moving parts using low (battery-powered) alternating potentials. Since the discovery of AC electro-osmosis (ACEO) in the late 1990s, there has been much work in designing planar, periodic pumps, which exploit broken symmetry in electrode spacing and width to produce a streaming flow over a surface. Although surface-height modulation has been suggested as another means of breaking symmetry[1], it has never been numerically or experimentally pursued. Recently, Bazant and Squires described more general flows due to induced charge electro-osmosis (ICEO) around three-dimensional metal structures[2], which has since been realized experimentally in microfluidic systems[3]. Motivated by ICEO around raised electrodes, we are developing a variety of new three-dimensional AC electrokinetic pumps capable of much faster directional flows than planar ACEO pumps (for the same applied voltage and minimum feature size) by an order of magnitude according to the usual low-voltage model. This phenomena and an example microfabricated device are illustrated in Figure 1. We test and improve our theoretical designs experimentally in a microfluidic loop[4], as shown in Figure 2. Our pumps involve interdigitated planar electrodes with raised metal structures from a simple electroplating step, which leads to greatly enhanced pumping.

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Figure 1: (A) Schematic diagram of fluid flow that may be generated by an AC field between two electrodes located on a substrate. This “fluid conveyor belt,” containing partially raised electrodes, exploits naturally occurring fluid rolls to pump fluid in microchannels with voltages $< -10$ V. (B) Electroplating is used to create the raised geometry on repeated periods of planar patterned electrodes.

Figure 2: A microfluidic device featuring a closed loop channel is used to test AC electrokinetic pump designs. The PDMS chip, which caps the electrodes, provides fluid inputs and outputs, and isolates the working pump from external pressure perturbations. This approach enables systematic characterization of pump performance as a function of input voltage and frequency. The scale bar indicates 1 mm.

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REFERENCES
In this research, we propose an energy-efficient architecture to interface carbon nanotube (CNT) chemical sensors, and the development of signal processing algorithm to reliably infer the chemical concentration in air based on the sensor read-out results. The CNT changes its conductance when exposed to certain chemicals [1] (Figure 1), and thus we can effectively utilize CNTs as resistive chemical sensors. The room-temperature operation of the chemical-sensing mechanism makes CNT an appealing candidate for low-power chemical sensor application.

However, poor control over the CNT process, the resolution requirements in conductance measurements, and the changes in conductance due to specific chemicals in air require that the front-end circuitry has a dynamic range of more than 18 bits. While such accuracy is power-consuming to attain [2], the reduction in power-supply voltage further aggravates the dynamic-range limitations in analog circuits. In order to surmount such problems, we are developing a new architecture suitable for this application.

The stochastic nature of CNT chemical sensors calls for multiple deployments of CNT sensors in one sensor node. This constraint, in turn, requires an efficient algorithm to infer the concentration of the chemical we are interested in. Thus, this research will also delve into developing an energy-efficient algorithm that can be operated in real time.

This project is currently carried out in collaboration with Kyeongjae Lee and Professor Jing Kong from the department of Electrical Engineering and Computer Science at MIT to design an integrated gas sensor.

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An Energy Efficient Transceiver for Wireless Micro-Sensor Applications

D.C. Daly, A.P. Chandrakasan
Sponsorship: DARPA Power Aware Computing/Communication Program

Large-scale wireless sensor networks require a low-power, energy-efficient transceiver that can operate for years on a single battery. To meet this demand for microwatt average power consumption, the transceiver must be scalable, support duty cycling, and be energy-efficient when “on.” A key metric for measuring the energy efficiency of the transceiver is the energy per bit ratio, which is equal to the power consumption of the transmitter or receiver divided by the instantaneous data rate. We have fabricated a custom radio for wireless micro-sensor applications that achieves energy-per-bit ratios down to 0.5 nJ/bit in receive mode and 3.8 nJ/bit in transmit mode [1]. These low ratios, combined with a fast receiver startup time of 2.5 µs, allow for energy-efficient operation.

The transceiver operates at 1 Mbps in a single channel centered at 916.5 MHz and employs on-off keying (OOK) modulation. A non-coherent, envelope-detection receiver architecture removes the need for a local oscillator and allows for a fast receiver startup time. Figure 1 shows a block diagram of the transceiver. The RF front end supports several gain settings, so that the power consumption of the receiver can be reduced in the presence of large input signals. The transmitter supports 7 digitally controlled output power levels to enable power-scaling based on node proximity. The receiver power consumption scales from 0.5 mW to 2.6 mW, with an associated sensitivity ranging from -37 dBm to -65 dBm at a bit error rate of 10^-3. The transmitter supports output power levels from -11.4 dBm to -2.2 dBm.

The chip was fabricated using 0.18 µm CMOS technology; a chip micrograph is shown in Figure 2. We acknowledge National Semiconductor for providing the IC fabrication services and NSERC for funding. Denis Daly is partially supported by an NSERC fellowship.

REFERENCES

Combinatorial Sensing Arrays of Phthalocyanine-based Field-effect Transistors

M. Bora, D. Schut (HP), M.A. Baldo

Of the millions of molecular species floating in air or dissolved in water, a substantial fraction can be smelled and uniquely discriminated [1]. Biological systems achieve this functionality with a multitude of non-specific receptors. In this project, we are developing gas sensors based on combinatorial arrays of organic transistors. The combinatorial approach reduces the need to develop specific receptors for each and every molecule of interest. Rather, our sensors are based on exploiting the wide variation in interactions between molecules and metal ions [2], an approach previously employed in colorimetric sensors [3].

We have fabricated gas-sensitive organic transistors each consisting of an approximately 10-nm-thick polycrystalline layer of a metallophthalocyanine (MPC) with gold source and drain contacts. The width and length of the channel for each transistor is 2 mm and 50 μm, respectively. The charge-carrier mobility is typically between 10⁻³ and 10⁻⁴ cm²/Vs. But the transconductances of various MPC transistors (CoPC, CuPC, ZnPC, and NiPC) are observed to vary when exposed to different gases (acetonitrile, tetrahydrofuran, and toluene); channel current in MPC transistors decreases linearly with increasing solvent concentration (Figure 1). The transient response of the current modulation (Figure 2) is chemically selective and depends on the interaction between the solvent and the central metal atom in the MPC. The linear dependence of channel current on solvent concentration, the steady state current modulation, and the transient response of the MPC transistors are all consistent with the disruption of percolation pathways leading to modulation of transistor channel currents. Since the sensors can be manufactured simply by inkjet printing on a patterned substrate, they may find application as single-use diagnostic aids.

**Figure 1:** The linearity of MPC sensors is tested by modulating the solvent concentration (V_{ds}=-20V, V_{g}=-20 V).

**Figure 2:** The transient rate of channel current recovery, k_{off}, after removal of solvent vapor in units of min⁻¹, summarized for various MPC-solvent combinations. Transistor bias conditions are V_{ds}=-20 V, V_{g}=-20 V.

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Nanoelectromechanical Switches and Memories
K.M. Milaninia, M.A. Baldo
Sponsorship: MARCO MSD, ISN

The ability to change shape is a compelling attraction of molecular semiconductors. Compared to rigid inorganic materials, molecules are soft and malleable, and their conformational changes are essential to the functionality of biological systems. Applications of nano-electro-mechanical (NEM) molecular devices include memories and transistors. Information can be stored in the conformation of molecules, potentially leading to very high density memories; molecular transistors that change shape under bias could exhibit subthreshold slopes of \(< 60 \text{ mV/decade}\)\[1\]. Indeed, as an example of the potential of NEMs, voltage gated ion channels possess subthreshold slopes of approximately 15 mV/decade \[2\].

Although many materials are available for NEM applications, carbon nanotubes exhibit low resistance and good mechanical properties. In this project, we are constructing a NEM testbed. The proposed design for our relay is shown in Figure 1. Nanotubes are directly grown at the bottom of an electron-beam defined trench etched in Si. This offers better control over nanotube growth and removes the need for additional steps that are required for the removal of surfactants and organics from the surface of the nanotubes. Because the nanotubes are vertically oriented, we are able to take advantage of the smallest size feature of the carbon nanotube - its diameter. This allows us to create dense arrays of relays for applications such as memory or logic devices. The vertical orientation allows NEM structures with very large aspect ratios. Theoretical results\[3\] have shown that increasing the aspect ratio of a carbon nanotube reduces the voltage needed to pull in the nanotube and thereby reduces the power requirement. Furthermore, because of the ability to easily functionalize the surface of nanotubes, we can functionalize the tube with charge to lower the pull-in voltage even further.

![Figure 1: Initial results were obtained by introducing a carbon nanotube mounted to a tungsten probe between two (Au/SiO2/Si) contacts. (b),(c), and (d) are schematics of the device in state 1 (its initial state), state 2, and state 3, respectively.](image1)

![Figure 2: An I-V of device being switched from state 1 \(\rightarrow\) 2 by applying a bias between Contact 1 and CNT, then switched from state 2 \(\rightarrow\) 3 by applying a bias between Contact 2 and CNT, and finally from state 3 \(\rightarrow\) 2 by applying a bias between Contact 1 and CNT.](image2)

REFERENCES
Integrated Carbon Nanotube Sensors
K.-J. Lee, T.S. Cho, A.P. Chandrakasan, J. Kong
Sponsorship: Intel, MARCO IFC

Single-walled carbon nanotubes (SWNT) exhibit unique mechanical and electrical properties that make them attractive for nanoelectronic applications. In particular, the conductance of a semiconducting SWNT is known to change significantly when exposed to certain gases [1]. The high sensitivity and fast response time make carbon nanotubes (CNT) very attractive for chemical sensing applications. However, the underlying sensing mechanism of CNT sensors is quite different from Si-based chemical transistors, and fabrication methods generally yield devices with large variations. Device variations and technology integration issues must be resolved before any CNT sensor devices can become practical [2].

In this project, we grow carbon nanotubes through chemical vapor deposition (CVD) and fabricate devices for an ultra-low-power wireless sensing system. This work is done in collaboration with T. S. Cho in Professor A. Chandrakasan’s group from the Department of Electrical Engineering and Computer Science at MIT, whose back-end circuitry will provide an interface to our CNT sensor array. The main goals of this project are to build a CNT sensor array with high yield of semiconducting SWNTs, high sensitivity and selectivity of gases, and low variability in the performance of the device. Through statistical characterization of the device, we attempt to get a better grasp of the underlying CNT sensing mechanism and find enhanced fabrication methods to reduce performance variability. In addition, chemical functionalization of CNT sensors will allow the target application to detect different types of toxic gases for environmental and industrial applications.

REFERENCES
Organic Photovoltaics with External Antennas

J.K. Mapel, T.D. Heidel, M.A. Baldo
Sponsorship: DARPA, NIRT

The structures and processes of photosynthesis are evolved, highly efficient, robust, and possess high power density. We attempt to leverage these characteristics by incorporating photosynthetic architectural motifs into organic semiconductor solar cells. We adapt the organization of processes in photosynthesis and introduce a synthetic light harvesting structure into an organic photovoltaic so that it couples light energy to the active device area by near field energy transfer. Light energy absorbed in an artificial antenna layer is transferred to an artificial reaction center in the interior of the solar cell. The energy transfer is of the Förster type, mediated by surface plasmons polaritons. While the introduction of the antenna necessarily adds a step to the energy transduction process, decoupling photon absorption and exciton dissociation can be exploited to increase each separately.

We have experimentally examined the efficiency of energy transfer for this process. We utilize a film of photoluminescent chromophores placed immediately adjacent to an organic solar cell with dual silver electrodes as an antenna layer. We predict and verify that energy transfer can occur in technically relevant device structures with energy transfer efficiencies of approximately 50% and demonstrate this transfer result in increased quantum efficiency.

Figure 1: The device structure utilized in these studies is composed of aluminum tris(8-hydroxyquinoline), bathocuproine, copper phthalocyanine, 3,4,9,10-perylenetetracarboxylicbis-benzimidazole, 4-dicyanomethylene-2-methyl-6-(p-dimethylaminostyryl)-4H-pyran, and silver. To tune the emission of the Alq3 antenna it was doped with either CuPc or DCM at 1% weight ratio.

Figure 2: The comparison of devices with functional (dotted line) and nonfunctional (solid line) antennas demonstrate external energy transfer. Devices with functional external Alq3 antenna layers (dotted line) exhibit an increase in external quantum efficiency over the wavelength range where Alq3 absorption occurs (dashed line). The photocurrent spectra are identical outside the spectral range where Alq3 absorbs. Functional antennas employ the laser dye, X = DCM, whereas nonfunctional antennas employ the quencher X = CuPc).
Integrated Optical-wavelength-dependent Switching and Tuning by Use of Titanium Nitride (TiN) MEMS Technology

S. Takahashi, G.N. Nielson, G. Barbastathis

Sponsorship: DARPA

Ring resonators are integrated optical components that have the capabilities to filter specific bands of wavelength from a broad-band input signal. Although these devices themselves are passive components, by use of evanescent field coupling, the ring resonator can be switched or tuned by electrostatic actuation of a MEMS bridge-type structure above the ring waveguide (Figure 1). This procedure of switching and tuning will allow for a faster speed than thermal tuning and will not induce severe loss, as does carrier injection.

One major issue that must be faced when implementing this concept is the residual stress that the MEMS bridge structure exhibits. Precise vertical positioning of the bridge is crucial for this type of device, and hence the bridge structure must not deflect due to residual stress within the structure. Use of TiN was investigated as the structural material due to its appealing mechanical properties, high electrical conductivity as well as its ability to relax its residual stress by annealing.

The TiN MEMS bridges were fabricated and tested. The MEMS structures were successfully annealed to a flat state, and the actuation voltage of the switch was 90V (Figure 2).

Recently, we have been investigating an architecture which allows for a more flexible design of the device, as well as the capabilities for implementing ultra fast-switching and feedback control of the position of the wafer via capacitance measurement [1]. In this architecture, the ring resonator is fabricated on one wafer and the MEMS structure on another, and the wafers are flip-chip bonded by thermocompression waferbonding with gold.

Wavelength tuning of the ring resonator filter has been numerically simulated by a combination of FEM structural analysis and mode-solver electromagnetic analysis. This analysis showed that tuning of a range of 30 nm with an actuation voltage of 10V was feasible with a silicon oxynitride membrane as the dielectric material.

**Figure 1:** Concept of ring resonator switching via evanescent field coupling (top) and the implemented MEMS-based switching device (bottom). The same concept can be used to tune the resonating wavelength of the ring resonator by using a dielectric material instead of a lossy material, in which case the dropping wavelength on the top left figure will be altered to another wavelength when the membrane is pulled down close to the ring resonator.

**Figure 2:** The profile of the TiN MEMS bridge before and after annealing at 500°C (top), and the voltage vs. deflection curve of the actuated MEMS bridge (bottom).

**REFERENCES**

Volume holographic imaging is a 3D imaging technique that uses volume holographic lenses. We devised a rainbow volume holographic imaging (RVHI) system, shown in Figure 1. The system projects a rainbow illumination on objects by means of a diffraction grating. Collimated white light is decomposed by a diffraction grating. After passing through a cylindrical lens, the rainbow is focused to the focal plane of a volume holographic lens. All the in-focus points along the $x$ dimension are Bragg-matched; in the $y$ dimension they are also Bragg-matched because of degeneracy. Therefore, the entire rainbow plane is Bragg-matched, resulting in a broad field of view (FOV). When shifted out of focus, each point source in the rainbow becomes Bragg-mismatched due to defocus, much as a narrow-band source at the same wavelength would be [1].

A four-dimensional (3D + spectral) imaging system was devised recently. One unique advantage of this system is that it can be used under natural broadband light illumination on this principle [2]. The imaging process of the 4D imaging system can be understood as a combination of non-invasive optical slicing and spectral analysis. A narrow slit portion (along the $y$ axis) of the object on the focal plane is sampled and its spectrum is laterally imaged on the CCD camera. To cover the entire four-dimensional object space (three spatial dimensions plus one spectral dimension), auxiliary mechanical scanning (rotational mirror as shown in Figure 2) or multiplexing holograms corresponding to different slits are necessary. Also both the 1st order and 0th order diffraction from the volume hologram are measured simultaneously to normalize the brightness of objects.

**REFERENCES**


White Light QD-LEDs
P.O. Anikeeva, J.E. Halpert, M.G. Bawendi, V. Bulović
Sponsorship: ISN, NSF Materials Research Science and Engineering Center at MIT, PECASE

We are developing white-light emitting quantum dot LEDs (QD-LEDs) for use as planar white-light sources in the full-color active-matrix displays with color filters and in future solid-state lighting. Our white QD-LEDs consist of organic charge transport layers with a QD monolayer sandwiched between them. This device architecture enables independent processing of the charge transport layers and the emissive layer. The independent processing of QDs is a result of the recent development of the QD printing technique, which allows for the solvent-free deposition of QD monolayers onto various organic materials [1]. By mixing different amounts of high quantum-yield colloidal core/shell QDs (red CdSe/ZnS, green ZnCdSe/ZnS, blue CdS/ZnS), we demonstrate different QD-LED colors. Figure 1a shows electroluminescence of the white QD-LED with the CIE (Commission International de l’Eclairage) coordinates of (0.42, 0.41). Figure 1b and inset of Figure 2 also show that CIE coordinates vary only slightly under the different applied bias and different operation time [2].

![Figure 1: a) Photograph of a white QD-LED operated at 10 V of applied bias. b) CIE coordinates of QD-LEDs: red (red triangle), green (green triangle), blue (blue triangle), and white (purple diamond at 12 V). Change of color with operation lifetime is shown with progression from red circle to purple circle for a QD-LED operated at 9 V.]

![Figure 2: Electroluminescence of the blue (blue line), green (green line), red (red line), and white (black line) QD-LEDs shown not to scale to demonstrate the presence of the spectral signatures of all three QD colors in the white device spectrum. Inset: We observe slight change of the spectral shape of the white QD-LED with operation time.]

REFERENCES
Organic Optoelectronic Devices Printed by the Molecular Jet Printer

J. Chen, V. Leblanc, M.A. Baldo, M.A. Schmidt, V. Bulović
Sponsorship: Hewlett-Packard

Using a micro-fabricated silicon printhead, we developed a novel printing technique, molecular jet printing (MoJet) [1-2] that allows direct patterning of small molecular weight organics and metals by additive deposition at high resolution. Today’s dominant method for patterning vacuum-deposited semiconducting molecular organics uses a thin metal stencil as a shadow mask through which material evaporates. Once the metal stencil is fabricated, it cannot be reconfigured to define arbitrary patterns or be scaled up with substrate size. In contrast, the MoJet printing technique utilizes a silicon printhead that integrates a moving micro-shutter with a micro-aperture. The shutter can be opened or closed with a DC control signal. Evaporated organic molecules can either pass through the aperture to reach the substrate when no bias is applied or be obstructed by the shutter when the control signal is above 30 V DC. This reconfigurable printhead together with a moving stage allows arbitrary patterning capability and scalability of the MoJet printer to larger substrate sizes.

We demonstrate that active organic devices such as organic LEDs and organic FETs (see graphics below) can be fabricated directly using the MoJet printer. The MoJet printing is a solvent-free process (in contrast to ink-jet printing) that combines the high quality of thermally evaporated thin films with the high precision and scalability enabled by MEMS technology. The MoJet printed organic electronic devices have the same performance characteristics as those defined by the shadow-mask patterning method, but the size of the substrate plate can now be expanded beyond GEN 2/3. As such, the MoJet printer surpasses the capability of the metal-stencil shadow mask and has the potential to become the next generation patterning tool for making organic optoelectronic devices.

Figure 1: (a) Layer structure of printed OLED array. The green electroluminescent layer (EL) is directly printed with the MoJet printer. (b) EL micrograph of active OLED array at 7.5V applied voltage. The image is taken through a 470-nm low-pass filter to reveal that green pixel patterned by the MoJet. The pixel size is 30 µm, equivalent to 800 dpi resolution.

Figure 2: (a) Structure of the printed Pentacene transistor. Pentacene layer is printed down first and silver contacts are printed sequentially. (b) Micrograph of a single-printed OFET on silicon oxide. (c) The I-V characteristics of one of the transistors. The gate voltage Vg is varied from 0 to –60V in 10V steps.

REFERENCES
Design and Measurement of Thermo-optics on Silicon

R. Amatya, R.J. Ram
Sponsorship: DARPA

The vision of optoelectronics started with the integration of optics and electronics on the same substrate. Various optical and electrical components on the same chip would have better performance and functionalities than the components taken alone. Electrical integrated circuits (IC) have been very successful on silicon substrate. Thus, silicon is one of the more desirable materials for optoelectronic devices. Silicon (Si) as well as silicon nitride (SiN) optical waveguides are becoming integral components for optical systems because of their advantages of high index contrast and compatibility with IC. Thermally tunable optical ring resonators made of SiN on silicon can be used as switches and filters. A small ring diameter (≈20 µm) allows a large free-spectral range. The temperature dependence of the refractive index (i.e., the thermo-optic effect of the core and cladding) is utilized to tune these ring resonators. Resistive heaters are designed to sit on top of the cladding for thermal tuning. One of the key aspects while designing the heaters is minimum power dissipation per GHz in terms of tuning flexibility. Figure 1 shows a thermal simulation for a heater over a two-ring filter.

Waveguides formed on silicon-on-insulator substrates can have sub-microsecond switching capabilities. These waveguides are heated by passing current through them; it is important to be able to study and measure the thermal characteristics of the device. Thermoreflectance spectroscopy is one of the many ways of measuring the temperature of the device. A temperature profile for a silicon-melt waveguide of 0.5 µm thickness is shown by measuring the reflectance changes due to modulating the current. Figure 2 shows the image of the melt waveguide along with temperature profile and the thermoreflectance image. The figure on the left inset shows the non-uniform temperature distribution within the waveguide. For the silicon melt waveguide, the measured thermo-optic coefficient ($\kappa$) was 1.1E-4/K, which is similar to the reported values for silicon for the specific wavelength (510 nm). Similar measurements can be done on SiN ring resonators to obtain the thermo-optic coefficient.
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A Microfabricated Platform for Investigating Multicellular Organization in 3-D Microenvironments

D.R. Albrecht, R.L. Sah, S.N. Bhatia
Sponsorship: Whitaker Foundation, NSF, NIH

Understanding how complex intrinsic and external cues are integrated to regulate cell behavior is crucial to the success of cell-based therapies in the treatment of human disease. Systematic and quantitative investigation of these microenvironment signals was first enabled by precise cell positioning using 2-D micropatterning tools [1]. However, cellular signaling is often altered in adherent tissue culture where structural cues are lacking (including tumor, stem, and differentiated cells), in contrast to 3-D culture systems that more closely resemble in vivo cell behavior [2]. Our goal was to develop new micropatterning tools capable of micron-scale cell patterning and organization within a 3-D hydrogel with tissue-like properties. We developed a technique for the rapid formation of reproducible, high-resolution, 3-D cellular structures within a photo-crosslinkable hydrogel using dielectrophoretic forces (Figure 1) [3]. We demonstrate parallel formation of ~20,000 cell clusters of precise size and shape within a 1 x 2 cm² slab of tissue (Figure 2a), with high cell viability and differentiated cell function maintained over 2 weeks in culture. By modulating cell-cell interactions in clusters of various size (independent of hydrogel geometry, chemistry, or volumetric seeding density; Figure 2b), we present the first evidence that 3-D microscale tissue organization regulates chondrocyte behavior (Figure 2c) [3]. This dielectrophoretic cell patterning (DCP) technology enables further investigation of the role of tissue architecture in many other multicellular processes from embryogenesis to regeneration to tumorigenesis.

Figure 1: Rapid, parallel dielectrophoretic cell patterning method. (a) Assembled DCP chamber viewed in inverted microscopy demonstrates the hexagonal electrode array. (b) Swiss 3T3 fibroblasts before and after electropatterning within a 15 wt% PEG-diacylate prepolymer (viscosity: 3.3 cP) by +DEP toward high electric field at the electrodes, s = 100 µm apart, after 60 s exposure to 3.0 Vrms at 3.0 MHz.

Figure 2: Microscale organization alters cell function. (a) Thousands of micropatterned cell clusters are embedded within a thin hydrogel. (b) Cluster size (and cell-cell interaction) can be varied from single cells (U) to large clusters (III) within a single hydrogel slab. Cells have high viability (green), indicated by fluorescent staining. Scale bars, 100µm. (c) In articular chondrocytes, biosynthesis of a matrix molecule, GAG, is downregulated over 14 days in clusters of increasing size, independent of volumetric seeding density. “Sham” DCP hydrogels, in which cells were initially electropatterned but then randomized prior to hydrogel crosslinking, were indistinguishable from unpatterned controls. Data are mean ± s.e.m., n = 5 - 6. *, p<0.001 vs. “U”; †, p<0.001 vs. “Sham.”

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This project utilizes microfluidic systems to study how groups of liver cells acquire emergent tissue properties. Hepatocytes (the parenchymal cells of the liver) respond to many cues in their microenvironment: neighboring cells, growth factors, extracellular matrix, dissolved oxygen, and their interactions. One tissue property of interest is the compartmentalization of gene expression in multicellular domains along the liver sinusoid. This process, often described as “zonation,” underlies much of liver physiology and regional susceptibility to toxins. We have previously shown oxygen gradients can be used to compartmentalize mixed populations of hepatocytes in a large-scale reactor [1]. Here, we present a microdevice that enables one to explore the crosstalk between two inputs (oxygen gradients and soluble growth factors) in a systematic fashion. The device consists of a two-layer PDMS microfluidic network with an on-chip dilution tree bound to a glass slide with an array of microreactors. Hepatocyte zonation is induced in each microreactor through local oxygen concentration, which is modulated through gas channels separated from the bioreactor by a 100-µm PDMS layer as shown in Figure 1. The local oxygen concentration in the microchannels is quantified in Figure 2. Primary rat hepatocytes are seeded into microreactors together with 3T3 fibroblasts, which act to stabilize the hepatocyte phenotype as described previously. This device will be useful to further explore liver tissue biology in vitro including the dynamics of zonation, mechanisms of oxygen sensing, and the role of growth factors in zonal response.

**REFERENCES**

Micromechanical Control of Cell-Cell Interaction

E.E. Hui, S.N. Bhatia
Sponsorship: NIH NIDDK

Cellular behavior within tissues is driven by environmental cues that vary temporally and spatially with granularity on the order of individual cells. Local cell-cell interactions via secreted and contact-mediated signals play a critical role in these pathways. In order to study these dynamic small-scale processes, we have developed a micromechanical platform to control microscale cell organization so that cell patterns can be reconfigured dynamically. This tool has been employed to deconstruct the mechanisms by which liver-specific function is maintained in hepatocytes upon co-cultivation with stromal support cells. Specifically, we examine the relative roles of cell contact and short-range soluble signals, duration of contact, and the possibility of bi-directional signaling.

The device consists of two silicon parts that can be locked together either to allow cell-cell contact across the two parts or to separate the cells by a uniform gap of approximately 80 µm (Figs. 1 and 2). Switching between these two states is actuated simply by pushing the parts manually using tweezers; no micromanipulation machinery is necessary. Micron-scale precision is possible due to a 10:1 mechanical transmission ratio and microfabricated snap locks, both of which are monolithically incorporated into the silicon structure. The entire device is fabricated in a simple single-mask process using through-wafer deep reactive ion etching. To provide a surface compatible with cell culture, the surface is coated with a layer of polystyrene and plasma-treated, providing a standard tissue-culture surface.
A MEMS Drug Delivery Device for the Prevention of Hemorrhagic Shock

H.L. Ho Duc, M.J. Cima
Sponsorship: ARO (Institute for Soldier Nanotechnology)

Hemorrhagic shock is the number one cause of preventable death on today’s battlefield [1]. It is a hypotensive state of deficient organ perfusion caused by blood loss from wounds of the extremities or internal injuries. Hemorrhagic shock is normally treated by hemorrhage control, fluid replacement, and the injection of vasoconstrictors. Battlefield conditions, however, can prevent the timely administration of these measures. Hemostatic dressings developed for battlefield application are useful in controlling open wound hemorrhage but cannot stop internal bleeding or avert shock if too much blood has been lost [1]. Arginine vasopressin is a vasoconstrictor that causes peripheral and abdominal arteries to constrict, shunting blood to the vital organs in case of hemorrhage [2]. It improved survival by restoring blood pressure in pre-clinical experiments and clinical case studies of hemorrhagic shock when treatment was not immediately available [3-7]. This property makes it a perfect candidate for battlefield injection to keep wounded soldiers alive until they can be properly treated. Self-injection may not always be possible, however, due to the nature of these traumas.

We are currently developing an implantable drug delivery microelectromechanical system (MEMS) to deliver vasopressin to wounded soldiers on the battlefield. This device consists of a silicon substrate in which pyramidal wells are etched using common MEMS processing techniques. The wells are capped by metallic membranes and the chip is hermetically bonded to a Pyrex macroreservoir (Figure 1). The macroreservoir can be injected with 25 µL of a vasopressin solution to be released on demand. Applying an electric pulse through a metallic membrane melts it by resistive heating, exposing the macroreservoir to the environment. We also observed the formation of multiple thermal bubbles inside the macroreservoir, which enabled rapid delivery of the solution. We are redesigning the device to better control this mechanism. Future challenges include insuring long-term hermeticity and wireless activation of the device.

Figure 1: Picture of the MEMS device showing the Pyrex macroreservoir (left) bonded to the silicon chip (right). The metallic layer on the silicon chip controls the opening of micro-wells allowing vasopressin out of the macroreservoir.

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Recent reports indicate that it takes nearly $800 million dollars and 10-15 years of development to bring a drug to market. Nearly 90% of the lead candidates identified by current in vitro screens fail to become marketable drugs. One of the reasons for the high failure rate of drug candidates is the lack of adequate models. To address the problem, we have developed a new cell culture analog amenable to routine use in drug development. It is based on the standard multiwell cell culture plate format but it provides perfused three-dimensional cell culture capability.

The multiwell plate microbioreactor array [1, 2] consists of a fluidic and a pneumatic manifold with a diaphragm sandwiched in between them. The fluidic manifold contains an array of microbioreactor and reservoir pairs (Figure 1). Each microbioreactor/reservoir pair is fluidically isolated from all other microbioreactors on the plate. A key component of a microbioreactor is a scaffold for tissue morphogenesis (Figure 2). The scaffold is a thin wafer containing an array of channels in which cells self-assemble into 3D pieces of tissue. It is backed by a filter and a support scaffold. Tissue in the scaffold is perfused by cell culture medium. The medium is re-circulated between the reactor and reservoir by a diaphragm pump. The diaphragms of all pumps and rectifying valves are actuated in parallel via three pneumatic lines distributed by the pneumatic manifold. Fluidic capacitors control flow pulsatility.

The system provides a means to conduct high throughput assays for target validation and predictive toxicology in the drug discovery and development process. It can be also used for evaluation of long-term exposure to drugs or environmental agents and as a model to study viral hepatitis, cancer metastasis, and other diseases and pathological conditions.

**REFERENCES**


Characterization of Nanofilter Arrays for Biomolecule Separation

H. Bow, J. Fu, J. Han
Sponsorship: DuPont-MIT Alliance, NIH, Singapore-MIT Alliance

In the past decade, microfabricated devices have been developed that can separate, detect, and analyze various biomolecules [1]. In contrast to the sieving gels that are historically used in these studies, microfabricated devices are precisely designed and constructed. The deterministic structure of these devices facilitates experiment design and testing of theory. Periodic nanofilter arrays have been shown to separate DNA from 100 bp to 10 kbp [2]. These nanofilters consist of a regular sequence of free and constricted regions, with 50-100 nm being the characteristic dimension of the constricted region. In this context, the DNA is smaller than the constriction size, suggesting applicability of the Ogston sieving mechanism. Movement is characterized by the partitioning between the free and constricted regions due to steric constraints [3-4]. DNA has a persistence length of 50 nm (150 bp) and can be approximated as semi-rigid rods in this size range, facilitating theoretical analysis.

We investigated the effects on separation efficiency and resolution of changing various device and experiment parameters. These parameters include the strength of the electric field; depth of the deep region; depth of the thin and deep regions, while maintaining their ratio; silicon substrate bias; buffer strength; and period of the nanofilter array.

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We are currently developing an integrated nano- and microfluidic system for the parallel detection of multiple cancer biomarkers. The system will be based on the nanofluidic concentrator developed by the Han lab and is expected to achieve a detection limit for biomarker concentration that is significantly lower than what is possible with existing immunoassays such as ELISA and radioimmunoassays. A key attribute of the nanofluidic concentrator design is that the region where biomolecules are concentrated must not be larger than about 10-100 picoliters. In order to measure the concentration of specific biomarkers within this region, we propose to integrate the concentrator with a detector of similar volume in order to avoid dilution. The detection method is conceptually similar to ELISA; however, the readout of the biomarker/antibody binding is based on the direct detection of mass that absorbs to the surface of a sensor known as the suspended microchannel resonator (SMR). The SMR detector has a volume of 10 picoliters and a mass resolution of near 100 femtograms. Thus, we anticipate that the combined concentrator and SMR detection system will allow a specific biomarker to be detected at a resolution near 1 pg/mL. Since the SMR provides a real-time readout and the concentrator is easily controlled by an applied voltage, the integrated system can operate in a closed-loop mode in which the sample is amplified until the biomarker concentration reaches the vicinity of the dissociation constant of the biomarker/antibody complex. Thus, the linear range of the detector can be dynamically tuned over a wide range of biomarker concentrations.
Patterned Periodic Potential-energy Landscape for Fast Continuous-flow Biomolecule Separation

J. Fu, J. Han

Sponsorship: NSF, Singapore-MIT Alliance, NIH NIBIB

Manipulation of charged biomolecules through confining environments has broad applications in life science. Recent progress in fabricating well-defined spatial constraints allows direct observation of novel molecular dynamic behavior in molecular-sized confining structures. Further, it shows exceptional promise for providing regular sieving media with superior separation performance. Here we demonstrate a continuous-flow, biomolecule-separation device that makes use of a patterned anisotropic sieving matrix consisting of a two-dimensional periodic array of nanofilters. The electrophoretic drift of biomolecules in the sieving medium involves a differential bidirectional motion through two-dimensional, periodically modulated, free-energy landscapes that results in a vectorial apparent electrophoretic mobility that directs molecules of different sizes to follow radically different paths. This method provides a novel basis for dispersing small fluid-borne biomolecules into distinct fractions. A fluorescently labeled dsDNA mixture (50-766 bp) used to characterize the device was separated in 1 minute with a resolution of about 10%. The patterned anisotropic sieve was also used for size-fractionation of SDS-protein complexes of size ranging from 11 to 200 kDa in 1 minute. By virtue of its gel-free and continuous-flow operation, this device suggests itself as a key component of an integrated microsystem that prepares and analyzes biomolecule samples.

**Figure 1:** (a) Partitioning of rigid, rod-like molecules in a slit-like nanofilter. (b) Free energy landscape of a nanofilter. (c) An SEM images of a periodic array of nanofilters with alternating thin and thick regions. (d-f) Separation of SDS-protein complexes (d & e) and dsDNA molecules (f) in a one-dimensional nanofilter array chip ($d_1$: 55 nm, $d_2$: 300 nm, L: 1 µm). Band assignment for SDS-protein complexes: (1) cholera toxin subunit B (MW: 11.4 kDa); (2) lectin phytohemaglutinin-L (MW: 120 kDa); (3) low-density human lipoprotein (MW: 179 kDa), for DNA: (1) 50 bp; (2) 150 bp; (3) 300 bp; (4) 500 bp; (5) 766 bp.

**Figure 2:** (a) Bidirectional transport of biomolecules in the 2-D nanofilter arrays. (b) Fluorescence images of pulsefield separation of SDS complexes inside the 2-D nanofilter arrays. Different values of vertical and horizontal fields can be applied with different durations. Band assignments for SDS-protein are the same as in Figure 1.

**REFERENCES**


In this work, we have developed a simple microfluidic chip that can sort biomolecules based on their isoelectric point (pI) values in a simple buffer system. The new method differs from previous approaches such as transverse isoelectric focusing [1] or free-flow electrophoresis [2] in that this process involves no external power supply and no special ampholyte. Instead, we utilize the diffusion potential generated by the diffusion of different buffer ionic species in situ at the laminar flow junction. The use of diffusion potential in microfluidics was previously demonstrated with the mass transport of dye molecules between the two streams in [3]. However, they did not explicitly demonstrate a separation of two species. In our device, we establish a laminar flow junction between two buffers with different pH and concentrations. A potential gradient is developed across the liquid junction, generating a high-enough electric field to mobilize and to collect biomolecules at the boundary when their pI values fall between the two buffer pH values. The computational modeling shows a decreasing potential gradient from 17.1 V/cm to 6.9 V/cm along the 2-mm-long microchannel (20 μm deep, 100 μm wide), as the concentration gradient becomes shallower toward the end of the channel due to mixing (Figure 1). In our initial experiment, two pI-markers (Figure 2) as well as two proteins were successfully sorted in this device, with a flow rate of 5~10 µL/min. To characterize the accuracy of this pI-based sorting process, we tested sorting behavior of the device by changing the pH value of the sample buffer in 0.1 pH step. It was shown that a peptide can be sorted into a different output stream with a ~0.1pH unit resolution. We are currently working on the development of new buffer systems as well as on the hybrid approach with a superimposed external electric field to increase the sorting efficiency and resolution. Once fully developed, it can potentially be a pI-based sample fractionation tool for proteomic analysis of complex biomolecule samples.

REFERENCES
Cell Stimulation, Lysis, and Separation in Microdevices

J. Albrecht, J. El-Ali, S. Gaudet, K.F. Jensen

Sponsorship: NIH

Quantitative data on the dynamics of cell signaling induced by different stimuli require large sets of self-consistent and dynamic measures of protein activities, concentrations, and states of modification. A typical process flow in these experiments starts with the addition of stimuli (cytokines or growth factors) to cells under controlled conditions of concentration, time, and temperature, followed at various intervals by cell lysis and the preparation of extracts. Microfluidic systems offer the potential to do this in a reproducible and automated fashion. Figure 1 shows quantification of the stimulation of a T-cell line with antibodies performed in a microfluidic device with integrated cell lysis. The device is capable of resolving the very fast kinetics of the cell pathways, with protein activation levels changing 4-fold in less than 15 seconds. The quantification of the lysate is currently performed off-chip using electrophoretic separation. To extract meaningful data from cellular preparations, many current biological assays require similar labor-intensive sample purification steps to be effective. Micro-electrophoretic separators have several important advantages over their conventional counterparts including shorter separation times, enhanced heat transfer, and the potential to be integrated into other devices on-chip. However, the high voltages required for these separations prohibit metal electrodes inside the microfluidic channel. A PDMS isoelectric focusing device with polyacrylamide gel walls has been developed to perform rapid separations by using electric fields orthogonal to fluid flow (Figure 2). This device has been shown to focus low molecular weight dyes, proteins, and organelles in seconds.

Figure 1: ERK and JNK signaling in Jurkat E6-1 cells stimulated with α-CD3 for different times. Stimulation and cell lysis were performed with the microfluidic device (Chip stimulation) and with conventional methods (POS control). The error bars denote one standard deviation.

Figure 2: Layout of transverse IEF device. Top view (a) shows the PDMS device with the sample channel bordered by left and right gel regions (cross-hatched areas) and anode and cathode, respectively. It is separated from the gel sections by 40 µm × 40 µm, 50 µm tall posts (b). The device presented has a single inlet and single outlet. Side view (c) shows the device in cross-section (not to scale).
This project aims at developing high-throughput platforms for bioprocess developments. Based on the membrane-aerated microbioreactor [1], we have realized a microliter-volume, actively-mixed, and polymer-based microbioreactor by microfabrication and precision machining of PDMS and PMMA for batch [2] and continuous cultures [3] of microbial cells. Biological applications of microbioreactors, such as global gene expression of yeast cells [4], were demonstrated, and the parallel operation of multiple batch fermentations was realized by a multiplexed system [5].

As a very important operation for bioprocess developments, fed-batch process allows extensive control over environmental conditions in fermentations. Fed-batch fermentations in the microbioreactor were made possible by applying water evaporation through the PDMS membrane as a fluidic exit, and by combining passive feeding of water and active feeding of base, acid, and glucose solutions. Commercial microvalves were used to control pressure-driven liquid feeds to realize closed-loop pH control in the microbioreactor. For Escherichia coli fermentations, the pH value was successfully maintained within a certain range (Figure 1). Cells were physiologically healthier and remained active for longer periods of time (as shown by the dissolved oxygen curve), which in return yielded significantly higher biomass concentration at the end of experiments.

The microbioreactor was also integrated with the plug-n-pump microfluidic connectors [6], as well as incorporation of fabricated polymer micro-optical lenses and connectors for biological measurements to realize “cassettes” of microbioreactors (Figure 2). The fabrication process included precision machining and thermal bonding of PMMA devices. These integrations greatly simplified the setup and operation procedure and increased the signal-to-noise ratio for optical measurements for the cassettes, thus made the microbioreactors more compatible with high-throughput bioprocessing in multiplexed systems.

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Micro-fluidic Bioreactors for Studying Cell-Matrix Interactions

S. Chung, P. Mack, V. Vickerman, J. Hsu, R. Kamm
Sponsorship: NIBIB, Draper Laboratories

Mechanical forces are important regulators of cell biology in health and disease. Cells in the vascular system are subjected to fluid shear stress, cyclic stretch, and differential pressure [1]. Numerous investigations have revealed the vast pathological and physical responses of endothelial cells to fluid shear stress by culturing the cells on the rigid surfaces of a flow chamber. This approach, however, fails to mimic the true environment of cells in vivo that grow on flexible, porous basement membranes with a defined microstructure [1-4]. In order to create an improved model for this in vivo condition, we developed a new microfluidic bioreactor system that enables us to study cell-matrix interactions on soft substrates made of gel under conditions of controlled shear stress and pressure difference. A gel cage consisting of three thin layers (Figure 1) is constructed from PDMS using a silicon master made by the deep RIE process. Flow chambers, also made of PDMS, are cured on an SU-8 patterned master. Separate channels are included that allow for filling this central chamber with a gel that mimics the extracellular matrix and also allows for independent control over the flows in the upper and lower channels. The assembled bioreactor is shown in Figure 2. To conduct experiments, we introduce a peptide solution into the gel cage, allow it to gel, and then seed cells on the gel surface exposed through the holes of gel cage. After cell adhesion, the flow chambers are sealed by the application of a vacuum to the top and bottom sides of the gel cage. Flows are then applied to each chamber with controlled pressures and flow rates. With this system, we will apply controlled shear stress and pressure on the cell layers. We plan to study the process of angiogenesis that entails the growth of vascular sprouts emanating from one endothelial surface and connecting with the other.

REFERENCES
A Nanoscanning Platform for Biological Assays

S. Kim, S. Gouda, S.-G. Kim (P. So group)
Sponsorship: Intelligent Microsystems Center

An in-plane nanoscanning platform with switchable stiffness being developed at the Micro & Nano Systems Laboratory (MNSL) [1] can be an alternative to the existing atomic force microscope (AFM) system. The nanoscanning platform has a carbon nanotube (CNT) tip, which is known as one of the ideal candidates for AFM tips because of their superior mechanical and chemical properties. Raman Spectroscopy has gained a lot of interest as a tool for single molecule detection since it has easy and fast sample preparation and measurement compared to the existing technologies, such as X-ray crystallography and nuclear magnetic resonance. Among the several approaches attempted in order to enhance the weak Raman signals is tip enhanced raman spectroscopy (TERS). The enhancement of the electric field due to the plasmon resonance on the coated metal surface was predicted qualitatively [2]. The metal-coated CNT or CNT filled with Ag, Au, or Cu with a small diameter tip and high aspect ratio is ideal for TERS. The switchable stiffness AFM can work as a tool for imaging and placing the tip at the sub-nanometer proximity to a soft, molecular-scale biological sample, which would enhance the Raman signals.

\[ \varepsilon_r(\omega) \]

![Figure 1: Dependency of frequency on the permittivity of silver. The negative sign of the real part of permittivity contributes to the enhancement of the electric fields near the metal surface.](image)

\[ \varepsilon_r(\omega) \]

![Figure 2: Schematic of TERS showing the enhancement of electric fields near the metal tip or metal-coated CNT tip.](image)

**REFERENCES**


Label-free Microelectronic PCR Quantification
Sponsorship: AFOSR, Hewlett Packard, MIT Sea Grant Program

The introduction of real-time monitoring of the polymerase chain reaction (PCR) represents a major breakthrough in specific nucleic acid quantification. This technique employs fluorescent intercalating agents or sequence-specific reporter probes to measure the concentration of amplified products after each PCR cycle. However, the need for optical components can limit the scalability and robustness of the measurement for miniaturization and field-uses. Moreover, the addition of external fluorescent reagents can induce inhibitory effects [1] and require extensive optimization [2].

We have developed a robust and simple method for direct label-free PCR product quantification using an integrated microelectronic sensor (Figure 1) [3]. The field-effect sensor can sequentially detect the intrinsic charge of multiple unprocessed PCR products and does not require sample processing or additional reagents in the PCR mixture. The sensor measures nucleic acid concentration in the PCR relevant range and specifically detects the PCR products over reagents such as Taq polymerase and nucleotide monomers. The sensor can monitor the product concentration at various stages of PCR and can generate a readout that resembles that of a real-time fluorescent measurement using an intercalating dye but without its potential inhibition artifacts (Figure 2). The device is mass-produced using standard semiconductor processes, can be reused for months, and integrates all sensing components directly on-chip. As such, our approach establishes a foundation for the direct integration of PCR-based in vitro biotechnologies with microelectronics.

Figure 1: Cross-sectional drawing demonstrating the basis of the device measurement. Binding of charged molecules such as DNA on the sensor’s surface alters the distribution of positive mobile charge carrier in silicon, results in a modulation of the depletion depth (red arrow), hence changing the capacitance. This change in capacitance is monitored by measuring the AC current between the sensor and the gold electrode.

Figure 2: Comparison between steady state response of electronic measurements (black), real-time monitoring of PCR using Sybr Green I intercalating dye (red), and concentration analysis of the products using DNA Labchip kits (blue). No fluorescent labels were used for electronic detection and concentration measurements. However the discrepancy with the Sybr Green I measurement is likely due to partial inhibition of the PCR reaction by the fluorescent reagent.

REFERENCES
Vacuum-Packaged Suspended Microchannel Resonant Mass Sensor for Biomolecular Detection


Sponsorship: AFOSR, NIH

Microfabricated transducers enable the detection of biomolecules in microfluidic systems with nanoliter size sample volumes. Their integration with microfluidic sample preparation into lab-on-a-chip devices can greatly leverage experimental efforts in systems biology and pharmaceutical research by increasing analysis throughput while dramatically reducing reagent cost. Microdevices can also lead to robust and miniaturized detection systems with real-time monitoring capabilities for point-of-use applications.

We have recently fabricated, packaged, and tested a resonant mass sensor for the detection of biomolecules in a microfluidic format [1]. The transducer employs a suspended microchannel as the resonating element, thereby avoiding the problems of damping and viscous drag that normally degrade the sensitivity of resonant sensors in liquid (Figure 1). Our device differs from a vibrating tube densitometer in that the channel is very thin, which enables the detection of molecules that bind to the channel walls; this provides a path to specificity via molecular recognition by immobilized receptors. The fabrication is based on a sacrificial polysilicon process with low-stress LPCVD silicon nitride as the structural material, and the resonator is vacuum packaged on the wafer scale using glass frit bonding (Figure 2). Packaged resonators exhibit a sensitivity of 0.8 ppm/(ng•cm²) and a mechanical quality factor of up to 700. To the best of our knowledge, this quality factor is among the highest so far reported for resonant sensors with comparable surface mass sensitivity in liquid.

REFERENCES

Microbial Growth in Parallel Integrated Bioreactor Arrays
H. Lee, R.J. Ram, P. Boccazzi, A. Sinskey
Sponsorship: DuPont-MIT Alliance

Bioprocesses with microbial cells play an important role in producing biopharmaceuticals such as human insulin and human growth hormone and other products such as amino acids and biopolymers. Because bioprocesses involve the complicated interaction between the genetics of the microorganisms and their chemical and environmental conditions, hundreds or thousands of microbial growth experiments are necessary to develop and optimize them. In addition, efforts to develop models for bioprocesses require numerous growth experiments to study phenotypes of microorganism.

We have designed and developed integrated arrays of microbioreactors that can provide the oxygen transfer and control capabilities of a stirred tank bioreactor in a high-throughput format. The devices comprise a novel peristaltic oxygenating mixer and microfluidic injectors (Figure 1), which are fabricated using a process that allows the combination of multiple scale (100 µm-1 cm) and multiple depth (100 µm-2 mm) structures in a single mold. The microbioreactors have a 100 µL working volume, a high oxygen-transfer rate \( k_L a \approx 0.1 \text{s}^{-1} \), and closed loop control over dissolved oxygen and pH (±0.1). Overall, the system supports eight simultaneous batch cultures in two parallel arrays with two dissolved oxygen thresholds, individual pH set points, and automated near real-time monitoring of optical density, dissolved oxygen concentration, and pH.

These capabilities allowed the demonstration of multiple *Escherichia coli* aerobic fermentations with growth to high cell densities (>12g-dcw/L, Figure 2), and individual bioreactor performance on par with bench scale stirred tank bioreactors. The successful integration of diverse microfluidic devices and optical sensors in a scalable architecture opens a new pathway for continued development of parallel bioreactor systems.

![Figure 1: Photograph and schematic of parallel integrated bioreactor array device. a) Photograph of four reactors integrated into a single module. b) Cross-section showing peristaltic oxygenating mixer tubes and fluid reservoir with pressure chamber. c) Top view of schematic showing optical sensors and layout of peristaltic oxygenating mixer and fluid injectors. d) Cross-section showing the fluid-injector membrane pinch valves.](image)

![Figure 2: Four *E. coli* fermentations on a defined medium performed in a single micro-bioreactor array module. The heavy black line indicates the mean of the cell density and pH replicates and the minimum of the dissolved oxygen replicates. The pH was controlled at 6.9 until the base reservoirs were depleted. Due to differences in the \( k_L a \), the oxygen concentration in three of the reactors did not remain near the set point. The oxygen concentration of the mixer actuation gas (bold magenta dashes) is shown by the orange dashed line and approximately follows the exponential growth of the cells.](image)
The stem-cell microenvironment is influenced by several factors including cell-media, cell-cell, and cell-matrix interactions. Although conventional cell-culture techniques have been successful, they offer poor control of the cellular microenvironment. To enhance traditional techniques, we have designed a microscale system to perform massively parallel cell culture on a chip.

To control cell-matrix and cell-cell interactions, we use dielectrophoresis (DEP), which uses non-uniform AC electric fields to position cells on or between electrodes [1]. We present a novel microfabricated DEP trap designed to pattern large arrays of single cells (Figure 1, left). We have experimentally validated the trap using polystyrene beads and cells, showing excellent agreement with our model predictions [2]. In addition, by placing interdigitated electrodes between the traps, we can prevent cells from sticking to the substrate outside the traps (Figure 1, right).

To control cell-media interactions, we have developed a microfluidic device for culturing adherent cells over a logarithmic range of flow rates (Figure 2, left) [3]. The device controls flow rates via a network of geometrically-set fluidic resistances connected to a syringe-pump drive. We use microfluidic perfusion to explore the effects of continuous flow on the soluble microenvironment. We have demonstrated logarithmically-scaled perfusion culture of mouse embryonic stem cells over 4 days, with flow rates varying > 300x across the array. Cells cultured at the slowest flow rate did not proliferate while colonies at higher flow rates demonstrated healthy round morphology (Figure 2, upper and lower right) and expressed the stem-cell marker Oct-4. These microfabricated platforms will enable precise and unique control over the cellular microenvironment, allowing novel cell biology experiments at the microscale.

REFERENCES
Microfluidic/Dielectrophoretic Approaches to Selective Microorganism Concentration

H.-Y. Lee, K.A. Puchala, J. Voldman
Sponsorship: Draper Laboratories, NASA, MIT

This project focuses on the development of microfabricated microfluidic/dielectrophoretic devices capable of concentrating micron-size particles from complex liquids, for example water containing contaminants such as dust, sand, protein or soot. The concentrated particles of interest, such as pathogenic bacteria and spores, can then be delivered in small aliquots to the appropriate sensor for identification.

The micro-concentrator exploits the phenomenon of dielectrophoresis—the force on polarizable particles in spatially non-uniform electric field [1]—to trap the particles from the flow stream in order to subsequently concentrate them by release into a smaller volume of liquid. Dielectrophoresis does not negatively affect the liquid or the particles on which it operates. In our device the non-uniform electric field is created by interdigitated electrodes (IDE) at the bottom of the channel through which the contaminated solution is passed (Figure 1).

To maximize the exposure of particles to the DEP field, we mix the liquid using passive micro-fluidic mixers (Figure 1). Preliminary results with different fabricated micro-fluidic mixers exhibit up to 70% improvement in trapping efficiency as compared to devices without mixers (Figure 2). Although both the herringbone mixer (HM) and slanted groove mixer (SGM) show notable improvements over smooth channel configurations, the staggered herringbone mixer (SHM) provides the greatest enhancement in trapping efficiency. We believe that the chaotic mixing associated solely with the SHM exposes more particles to the concentrator’s bank of IDEs, thus resulting in higher trapping efficiency when compared to other mixer types.

The magnitude and direction of the dielectrophoretic (DEP) force depends on the particle’s dielectric properties (i.e., conductivity and permittivity); therefore, when the operating frequency of the field and the conductivity of the medium are chosen, the DEP force can be selectively applied to trap and concentrate some particles (bacterial spores of interest) and not others (dust, soot, sand or protein). In our device, initial banks of interdigitated electrodes are driven to maximize interferent trapping, while final stages capture spores from a purified solution. Using this mode of operation, we demonstrated selective trapping of B. subtilis spores while rejecting interferents such as pollen, chitin, sand and depleting interferents such as soot and dust. Future work will focus on improving purity and efficiency of trapping.

REFERENCES

Microfabricated Approaches for Sorting Cells Using Complex Phenotypes

B. Taff, S. Desai, J. Kovac, N. Mittal, J. Voldman
Sponsorship: NSF Graduate Research Fellowship, NIH NCRR, Singapore-MIT Alliance

We are developing microfabricated approaches to create sorting cytometers for genetic screening of complex phenotypes in biological cells. Our goal is to create technologies that combine the ability to observe with the ability to isolate individual mutant cells from a population under study. Such cytometry merges benefits of microscopy and flow-assisted cell sorting (FACS) to offer unique capabilities on a single platform. Biologists will be able to use these technologies to isolate cells based upon dynamic and/or intracellular responses, permitting creation of new types of genetic screens.

We currently are developing optical and electrical approaches to enable image-based sorting. One of our current approaches uses an array of switchable traps (Figure 1) that rely upon the phenomena known as dielectrophoresis (DEP) [1]. The DEP-enabled traps allow for capturing and holding cells in defined spatial locations and then subsequently releasing a desired subpopulation for further study. The traps in our device are controlled using a series of row and column electrical connections. This setup avoids any need for separate connections to each of the traps in our arrays. Our chip-to-world interconnect needs thus scale only as $2\sqrt{n}$ for any $n \times n$ trap footprint. This condition enables site-specific addressing within arrays sized appropriately for bio-relevant assays (10,000 sites) using a minimal number of electrical ties (200 wires). To date, we have captured, held, and sorted small populations of individual HL60 human leukemia cells using a demonstrative $4 \times 4$ trap array [2]. Figure 2 shows a proof-of-concept assay where orange- and green-stained HL60 cells are first held in the 16-site array and then we sorted each of the green cells from the grid.

Developing and scaling such a platform for screening applications requires performance characteristics that are easily met only by using quantitative modeling [3]. Using such an approach, we have developed updated trap geometries and system configurations for use in larger $20 \times 20$ array structures. Currently we are fabricating these enhanced devices, their affiliated control and automation systems, and specific RFP-tagged cell lines for planned complex phenotype-based sorting assays. In tandem with this design cycle, we are investigating the effects of DEP trapping on cell health and the impact that it may have on our ability to assess specific phenotypic behaviors. Complementary and alternative approaches for implementing these sorting functionalities are similarly under study in an attempt to lower the threshold for acceptance and use in biological laboratories.

REFERENCES:
A Continuous, Conductivity-Specific Micro-organism Separator

M.D. Vahey, J. Voldman
Sponsorship: NIH NIBIB, MIT Buschbaum Fund, CSBi/Merck Graduate Fellowship

Increased throughput in the techniques used to engineer new metabolic pathways in unicellular organisms demands similarly high throughput tools for measuring the effects of these pathways on phenotype. For example, the metabolic engineer is often faced with the challenge of selecting the one genomic perturbation that produces a desired result out of tens of thousands of possibilities [1]. We propose a separation method—iso-dielectric separation, or IDS—which separates microorganisms continuously based on their dielectric properties. This technology would enable high-throughput screening of cells based upon electrically distinguishable phenotypes.

Iso-dielectric separation uses dielectrophoresis (DEP) and media with spatially-varying conductivity to separate cells by their effective conductivity. It is similar to iso-electric focusing, except that it uses DEP instead of electrophoresis, and is thus applicable to uncharged particles, such as cells [2]. We apply this method to the separation of polystyrene beads (based on surface conductance), vesicles (based on the conductivity of the internal fluid), and cells (based on viability). Current efforts are focused on the separation of *Escherichia coli* based upon the amount of the intracellular polymer poly(hydroxybutyrate) that each cell contains.

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**Figure 1:** (A) Trial separation of polystyrene beads using nDEP. The smaller, more conductive beads separate out into higher conductivity, as would be expected. The 1.9 µm particles reach their IDP in-frame, where they are seen passing over the electrodes. (B) Using pDEP, the smaller beads still separate into higher conductivity, corresponding to a further displacement (from left to right) to reach the IDP than that for the larger beads.

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**REFERENCES**


Polymer Waveguides for Integrated Biosensors
K. Lee, R.J. Ram
Sponsorship: DuPont-MIT Alliance, NSF Center for Material Science and Engineering

To overcome the challenge of fabricating large-dimension waveguides with optical-quality surfaces, a conventional fabrication approach using plastics is utilized over a micro-fabrication approach generally used for micro-fluidics. While conventional milling can easily create structures with dimensions of 1 mm, roughness from the fabrication process leads to diffuse surfaces and variations greater than 1 µm. To overcome the roughness due to milling and ultimately create large features with optical quality roughness, a solvent vapor polishing process is used in which a solvent vapor such as methylene chloride vapor is used to liquify the plastic surface [1]. Varying the solvent pressure and the exposure time can control the degree of polishing. Under optimal polishing conditions, the average roughness from sanded polycarbonate samples can be reduced from 1000 nm to 70 nm as shown in Figure 1. When combined with vapor polishing, CNC milling becomes a viable process for both optical and micro-fluidic fabrication of master molds.

After mold fabrication, standard soft lithography processes are used to create negative PDMS replicas of the polycarbonate molds. In addition, PDMS molds are subject to e-beam deposition of evaporated silver to provide reflective surfaces where necessary. The same fabrication process is used for both waveguides and microfluidic components of the chip and the final oxygen sensing device is shown in Figure 2. The oxygen sensors used for this experiment are created by a mixture of Platinum(II) octaethylporphine ketone (PtOEPK) [2] and polystyrene deposited on glass disks. This fluorescent dye has a maximum absorption at 592 nm and emits at 759 nm.

Fabricated waveguides exhibit a propagation loss of 0.136 dB/cm at 626 nm and a measured 50% intensity numerical aperture of 0.5. E-beam deposited silver on PDMS measured a reflectivity of 72% with a HeNe laser at 632 nm. Oxygen measurements were performed by fiber coupling amplitude modulated LEDs into the input guides, collecting the output through a colored glass filter and photodetectors, and measuring the phase shift between the input and output signals. The sensor system achieves an SNR greater than 40 and provides a phase shift of 30 degrees with 1 degree variance from nitrogen to air.

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A Double-gated CNF Tip Array for Electron-impact Ionization and Field Ionization

Sponsorship: DARPA

Carbon nanofibers have been investigated for a wide range of applications today. In particular, due to their remarkable conductivity, carbon nanofibers have generated a lot of interest for applications in vacuum microelectronic devices [1-2]. For example, the ionization sensor for gases is one of the most important applications since the conventional ionization sensors are bulky, require high-voltages, and consume high power. The purpose of this project is to fabricate carbon nanofiber field emission and field ionization arrays, which can be utilized in a micro-gas sensor. This device can help reduce the size of the sensor and operating voltages required for gas analysis.

In this project, the PECVD method is used to grow vertically oriented carbon nanofibers. The number of carbon nanofibers per site is controlled by the Ni catalyst dot size. It has been demonstrated that the diameter of the Ni catalysts disk must be 300 nm or less to ensure the growth of only a single carbon nanofiber [3]. The 250-nm Ni dots used in this work were defined by ebeam lithography. Figure 1 shows a close-up SEM picture of vertically aligned single carbon nanofiber array grown by PECVD. Later, these vertically-aligned single carbon nanofibers will be integrated into a double-gated field emission/ionization structure developed by L. Dvorson [4]. Figure 2 shows the schematic drawing of the final device.

Using the device shown in Figure 2, two approaches for ionizing gas molecules will be investigated for micro-gas sensors. One approach is electron impact ionization, which uses strong electric fields to emit electrons followed by collision between the energetic electrons and neutral gas molecules resulting in ionization. The second approach is field ionization, which is a gentler process in comparison to electron impact ionization. It results in molecular ionization and a simpler mass spectrum due to lower fragmentation of molecules.

REFERENCES

A Double-gated Silicon Tip, Electron-Impact Ionization Array

L.-Y. Chen, A.I. Akinwande
Sponsorship: CreatV Micro Tech, NIH, DARPA

A device with the ability to ionize gases is needed for a variety of applications, of which the mass spectrometer (MS) [1-2] is one of the most important. The ionization method in the majority of gas analyzers in MS is electron-impact ionization, which uses a beam of electrons that collides with gas molecules. Through this collision process, energy is transferred from the electrons to the gas molecules, which causes electrons on the gas molecules to be stripped off (i.e., ionization of the gas molecules).

Traditionally, thermionic emission, which consists of a filament that produces electrons when heated, is the most common way of generating electrons for MS using electron impact ionization. However, thermionic emission has several disadvantages: slow switch-on time, large power consumption, and lack of robustness. These disadvantages, however, are eliminated when field emission is used instead.

In this project, a double-gated silicon field emission device is used to generate the electron source for electron impact ionization. Figure 1 shows a SEM picture of a double-gated silicon field emission device used here. Using this device, we have demonstrated the linear relationship between the emission current (IE) and the ion current (II) at a fixed pressure (10⁻⁴ torr) as shown in Figure 2.

![Figure 1: The SEM picture of the cross section of the silicon field emission and field ionization array.](image1)

![Figure 2: Plot of emission current (Iₑ) and ion current (Iᵢ) versus gate voltage (VG).](image2)

REFERENCES


A Single-Gated CNT Field-Ionizer Array with Open Architecture
L.F. Velasquez-Garcia, L.Y. Chen, A.I. Akinwande
Sponsorship: DARPA

The micro gas analyzer project aims to develop the technology for portable, real-time sensors intended for chemical warfare and civilian air purity control. The device is composed of four micro-fabricated subsystems: an ionizer, a mass filter based on a quadrupole array [1], an electrometer [2], and a positive displacement pump [3]. We are developing a single-gated fieldionizer array based on gated carbon nanotubes (CNTs). The devices achieve species ionization by tunneling of outer shell electrons due to the presence of high electric fields that the device sets. We use CNTs as field enhancers because of their small radii and high aspect ratio while the gate proximity ensures high fields at low voltage. State-of-the-art ionizers use electron-impact ionization (thermionic cathodes), incurring in excessive power consumption, low current, current density, ionization efficiency, and short lifetime. The field-ionizer arrays (Figure 1) are able to soft-ionize species, thus achieving molecule ionization. The reliability and lifespan of the field-ionizer arrays are larger than the corresponding values for electron-impact ionizer arrays because the CNTs are biased at the highest potential in the circuit, thus making it unlikely for ionized molecules to back-stream. Figure 2 shows two SEM pictures of a single-gated CNT array that implements a selective CNT-growth process. This process reduces the fabrication complexity of the device because it grows CNTs from an un-patterned catalyst (Ni). Current research efforts concentrate on improving the device and data acquisition, including benchmarking the performance of the ionizer in low-pressure oxidizing environments.

Figure 1: Schematic of a field-ionizer array. The gas inlet provides neutral species to the field enhancers. If the molecules of the gas come close enough to the CNT tips, an electron from the outer shell of the molecule will tunnel to the CNT, thus ionizing the molecule.

Figure 2: A single-gated CNT field-ionizer array grown at MIT. (A) Field view of an array section; (B) cross section of a single ionizer well; (C) detail of the well foot intended to grow CNTs; (D) detail of the well foot when CNTs are grown. The ionizer well has a film of silicon dioxide 5µm-thick below the gate that acts as electrical insulator between the gate and the CNTs. The gate can be made of either Ti, Au, or W. The CNT catalyst was 4-nm-thick Ni.

REFERENCES
Aligning and Latching Nano-structured Membranes in 3D Micro-Structures

N.S. Shaar, G. Barbastathis, C. Livermore
Sponsorship: ISN

The 3D micro-electro-mechanical systems (MEMS) manufacturing is an emerging technology that promises to solve many of the problems in the microfabrication industry. In microelectronics, as the feature sizes of the components approach their physical limits, packing more transistors on a microprocessor or on a memory chip requires expanding the circuitry into the third dimension. In optical switches, the traditional 2D MEMS-based switches do not scale easily beyond 32 ports; to increase the number of ports, companies have been developing 3D micro-mirror arrays that can reflect light in multiple directions. The Nanostructured Origami™ 3D fabrication process is a two-step method for fabricating 3D MEMS; it involves patterning films on a surface and then folding the patterned films to create three-dimensional structures [1]. This method is advantageous because it uses state-of-the-art 2D patterning methods and it involves patterning all the parts of the structure in one step, eliminating problems of feature misalignment. However, in creating the 3D structures, two major challenges arise; the first is to accurately place the folded membranes in their desired positions and the second is to fix the membranes in those positions to maintain the final 3D configuration. Current positioning solutions involve the use of mechanical motion-limiters that prevent folded membranes from moving beyond a certain point [2]. We propose two methods for aligning and latching folded nano-patterned membranes in 3D microstructures. The first method uses photoresist pads to glue together two mating surfaces of the structure (Figure 1). What distinguishes this method from previous polymer gluing attempts is that we use dense gold patterns as a local heater to melt the photoresist pads. This allows us to control the membranes we latch and the time when we latch them. We use patterned gold wires to form the hinges that hold the membranes together. Thin dense gold patterns also serve as local heaters to melt the photoresist gluing pads. The surface tension in the molten pads aligns the surfaces and solidification of the photoresist latches them together. The second method uses mechanical alignment and latching features that allow edges-to-surface latching (Figure 2). One major advantage of this method is that the structural components and the alignment features are patterned in the same lithographic step, which lowers costs and minimizes misalignment errors. Another interesting aspect is the cascaded alignment; the alignment features are designed so that they function sequentially, starting from the features closest to the hinge. With proper design of those features, the alignment system can achieve accurate positioning using the features away from the hinge while tolerating a large initial positioning error range by virtue of the short radius sustaining the features closest to the hinge.

REFERENCES
Characterization and Modeling of Non-uniformities in DRIE

H.K. Taylor, H. Sun, A. Farahanchi, D.S. Boning
Sponsorship: Singapore-MIT Alliance

Our previous work on spatial non-uniformities in deep-reactive ion etch (DRIE) has provided a method by which an etching tool and associated "recipes" of operating parameters may be pre-characterized [1]. That work allowed the wafer-average pattern opening density (or "loading") to be related to wafer-scale etch rate variations. Such variations have been attributed to loading-dependent interactions of the flux densities of $S_F$ ions and F neutrals and to shifts in the gross flows of fluorine across the wafer [2]. Unlike some other approaches [3–5], our method captures asymmetries in the fluxes within the chamber. Our approach is now supplemented by an understanding of how uniformity depends on the localization of etched patterns within the wafer (Figure 1). A semi-physical model represents the diffusion of monatomic fluorine etchant parallel to the wafer’s surface, giving a two-dimensional filter which translates a discretized map of pattern density into a prediction of how etch rate will vary within and between dies [6]. This die-level model is readily combined with the existing wafer-level model. To tune this combined model for a new recipe, a set of about five test wafers is etched, and fitting algorithms are run with etched-depth data. Collaborative experiments with Surface Technology Systems Ltd have demonstrated our approach in use with a prototype etch tool. Further experiments have compared the characteristics of different manufacturers’ tools. We have also quantified a memory effect whereby the average pattern density of one etched wafer can affect the average rate and non-uniformity with which a subsequent wafer etches (Figure 2). In the future we aim to incorporate well-known feature size or aspect ratio effects into our model [7]. We envisage our approach being integrated into computer-aided design systems for MEMS and believe that it will be of particular use when one is keen to preserve a fast-average etch rate and is thus loath to win uniformity by reducing the chamber pressure.

Figure 1: Diametrical variation of etch rate for 11 wafers with differing average pattern density. Heavy bars indicate the portion of the diameter into which etched silicon was concentrated.

Figure 2: The influence of the average pattern density ($\rho$) of one etched wafer upon the rate and uniformity with which the subsequent wafer etches. It is observed that a wafer’s etch rate is slightly accelerated when the preceding wafer has a very high etched density; moreover, the shape of the nonuniformity is also echoed.

REFERENCES

The hot embossing of thermoplastic materials, such as polymethylmethacrylate (PMMA) or cyclo-olefin copolymer (COC), is a promising way to manufacture microfluidic channels and networks [1]. Hot embossing potentially offers lower per-area cost than the micromachining of quartz or silicon and easier scaling-up of production than soft lithography using polydimethylsiloxane [2]. In hot embossing, a microfabricated mold (typically of silicon or nickel) is pressed into a flat sample of polymeric material that has been softened by heating it above its glass-transition temperature. We are particularly interested in how the spatial distribution of mold features—their diameters, shapes, and areal densities—may influence the quality of embossed patterns. We are developing a simulation approach whose building-block is a simple model in which, for given embossing conditions, a feature-sized disk of viscous polymer is compressed at a rate inversely proportional to the square of the radius of the disk [3] (Figure 1). Such a model implies that the mold will sink into the substrate at a spatially uniform rate when the product of the areal density of mold features and the square of their average radius remains constant across the mold. We aim to construct a reliable model that is computationally efficient and that can predict the combination of embossing pressure and duration required by any mold design. We are investigating the measurement of birefringence of embossed samples [4] as a way of monitoring the embossing process (Figure 2). We are also pursuing a technique for the bonding of polymer surfaces that promises minimal deformation of pre-embossed features: the polymer surfaces are exposed to an oxygen plasma for ~1 minute and then pressed together [5].

\[ \text{Figure 1: Proposed model for the spatially non-uniform filling of embossing mold features with heated thermoplastic material (blue). Arrows indicate the displacement of material. Regions of the mold with higher areal densities of protruding features, e.g., on the left in this figure, are expected to be filled more quickly (a), and to “coagulate” into effectively larger features (b). Eventually all features would be filled and the polymeric substrate may continue to be compressed as one large disk (c).} \]

\[ \text{Figure 2: Light transmitted by each of two embossed PMMA samples sandwiched between perpendicular polarizers. The two samples were embossed under } \sim 1 \text{ MPa for equal lengths of time. At } 110^\circ \text{C (a), material within about 1 mm of the corners of embossed, 30 } \mu \text{m-deep rectangular channels exhibits substantially higher birefringence than the rest of the sample, implying more concentrations of residual stress there. At } 150^\circ \text{C (b), feature-scale birefringence becomes less important than sample-scale birefringence. Samples fabricated by Wang Qi.} \]

**REFERENCES**


Atomic Force Microscopy with Inherent Disturbance Suppression for Nanostructure Imaging
A. Sparks, S.R. Manalis
Sponsorship: AFOSR

Scanning probe imaging is often limited by disturbances, or mechanical noise, from the environment that couple into the microscope. We demonstrate on a modified commercial atomic force microscope that adding an interferometer as a secondary sensor to measure the separation between the base of the cantilever and the sample during conventional feedback scanning can result in real-time images with inherently suppressed out-of-plane disturbances (Figure 1) [1]. The modified microscope has the ability to resolve nanometer-scale features in situations where out-of-plane disturbances are comparable to or even several orders of magnitude greater than the scale of the topography. We present images of DNA in air from this microscope in tapping mode without vibration isolation, and show improved clarity using the interferometer as the imaging signal (Figure 2). The inherent disturbance suppression approach is applicable to all scanning probe imaging techniques.

We do not claim that image improvement will be comparable to these results on all SPMs and in all imaging environments. At present, this technique will be most effective in very noisy environments, such as a microfabrication facility, where Z disturbances overwhelm sample topography. However, there are two significant implications of this work: 1) vibration isolation, which is costly and consumes space, can be rendered unnecessary for noisy environments; and, 2) this technique can potentially outperform vibration isolation in any environment with further reduction of the interferometer noise floor.

REFERENCES
Vacuum-Sealing Technologies for Micro-chemical Reactors

K. Cheung, K.F. Jensen, M.A. Schmidt
Sponsorship: ARO MURI

Current portable power sources may soon fail to meet the demand for increasingly larger power densities. To address this concern, our group has been developing MEMS power generation schemes that are focused around fuel cells and thermophotovoltaics. At the core of these systems is a suspended tube micro-reactor that has been designed to process chemical fuels [1]. Proper thermal management is critical for high reactor efficiency, but substantial heat loss is attributed to conduction through air. A straightforward solution is to eliminate the heat-loss pathways associated with air by means of a vacuum package. This work explores a glass-frit bonding method for vacuum sealing.

Optimization of pre-sintering and bonding parameters of the glass frit produced a repeatable and robust hermetic seal. Encounters with outgassing issues prompted an alternate two-step packaging process illustrated in Figure 1. New capping dies were fabricated, test devices were packaged, and the final seal-off was attempted with various materials [2]. Several experimental results appear in Figure 2. The glass frits are undesirable since they produce holes from material breakdown when heated in a vacuum. The gold-indium solder appears promising but holes formed due to internal outgassing. Extended heating to assist outgassing resulted in the delamination of the solder from the wetting metal. Recent work has been conducted to evaluate oxidized caps and lead-tin solder as solutions to these problems. Enhancements through the incorporation of non-evaporable getters will be assessed once a vacuum package is achieved.

![Figure 1: Basic concept of the two-step approach.](image)

(a) Initial bond in box furnace (blue = frit, black = silicon, yellow = metallization); (b) place solder/frit (orange) into pump-out hole; and (c) final seal-off.

![Figure 2: Experimental results of final seal-off attempts with various materials and conditions](image)

(1) Diemat DM2700PF (2) Semcom B-10105 (3) Semcom B-10127 (4) 82Au/18In (5) 80Au/20In (6) 80Au/20In extended outgassing.

REFERENCES


Direct Patterning of Organic Materials and Metals Using Micromachined Printheads

V. Leblanc, J. Chen, D.M. Schut, P. Mardilovich, V. Bulovic, M.A. Schmidt

Sponsorship: Hewlett-Packard

Organic optoelectronic devices are promising for many commercial applications if methods for fabricating them on large-area, low-cost substrates become available. Our project investigates the use of MEMS in the direct patterning of materials needed for such devices. By depositing the materials directly from the gas phase, without liquid phase coming in contact with the substrate, we aim to avoid the limitations of inkjet printing such materials.

In our first demonstration, we used an electrostatically actuated micromachined shutter integrated with an x-y-z manipulator to modulate the flux of evaporated organic semiconductors and metals and to generate patterns of the deposited materials. We printed arbitrary patterns of organic semiconductor Alq3 (tris(8-hydroxyquinolinate) aluminum) and metal silver on glass substrates. We also printed pentacene/silver organic field effect transistor (OFET) and arrays of organic light emitting devices (OLED), as shown in Figure 1. This printing technique can pattern small-molecule organic light-emitting devices at high resolution (800 dpi).

The next stage of this project investigates the use of a microporous layer with integrated heaters for local evaporation of the materials. The microfabricated device is shown in Figure 2. The material to be printed is delivered to the porous region in liquid or gas phase and deposits inside the pores. An integrated heater then heats up the porous area and the material is re-evaporated from the pores onto the substrate. Compared to the first generation of printheads, the problems of crashing and stiction are avoided, since there is no moving part. Clogging is also limited since most of the material is removed during each printing cycle. Other advantages include the smaller quantity of organic material used, and the reduced substrate heating. Such a printhead would ultimately be integrated with an ink-jet printer for the delivery of liquid phase material into the porous region.

REFERENCES


MEMS Vacuum Pump

V. Sharma, M.A. Schmidt
Sponsorship: DARPA

There are many advantages to miniaturizing systems for chemical and biological analysis. Recent interest in this area has led to the creation of several research programs, including a micro gas analyzer (MGA) project at MIT. The goal of this project is to develop an inexpensive, portable, real-time, and low-power approach for detecting chemical and biological agents. Elements entering the MGA are first ionized, then filtered by a quadrupole array, and sensed using an electrometer. A key component enabling the entire process is a MEMS vacuum pump, responsible for routing the gas through the MGA and increasing the mean free path of the ionized particles so that they can be accurately detected.

There has been a great deal of research done over the past 30 years in the area of micro pumping devices [1, 2]. We are currently developing a displacement micro-vacuum pump that uses a piezoelectrically driven pumping chamber and a pair of piezoelectrically driven active-valves; the design is conceptually similar to the MEMS pump reported by Li et al. [3]. We constructed accurate computer models for all aspects of the pump’s operation: a compressible mass flow model of the flow rates, the pressure, the density, and the Mach number in the different parts of the pump in both the sonic and subsonic regimes [4], and a nonlinear plate deformation model of the stresses experienced by the pistons, tethers, and walls of the pump during operation [5], for any chosen dimensions and material properties.

Using these models we have defined a process flow for our first-generation MEMS vacuum pump designed to meet our first-term goals. A schematic of this pump that we started fabricating is shown in Figure 1 below. For ease in testing we have decided to fabricate only Layers 1-3 and constructed a testing platform that will drive the pistons pneumatically. This will allow for rapid characterization of pumping performance as well as chamber and valve designs for several dies at once without having to incorporate piezos in each case. The final device will be driven using low-voltage, low-loss, piezoelectric-stacks incorporated into Layer 4 and will include Layer 5 for structural support.

![Figure 1: Schematic of the MEMS vacuum pump. Layers 1 and 4 are glass, Layer 2 forms the chambers and channels using double-side polished silicon, Layer 3 forms the pistons and tethers being silicon-on-insulator, and Layer 5 is single-polished silicon. For testing and characterization, only Layers 1-3 are being fabricated.](image)

REFERENCES

We present approaches for growth of aligned carbon nanotube (CNT) structures on silicon substrates, based on atmospheric pressure chemical vapor deposition (CVD) using a Fe/Al$_2$O$_3$ catalyst film in C$_2$H$_4$/H$_2$. First, vertically-aligned films of small-diameter (5-10 nm) multi-walled CNTs (MWNTs) are grown to 0.9 mm thickness in 15 minutes and 1.8 mm in 60 minutes, using a conventional 1-inch-diameter tube furnace [1]. The catalyst is patterned by photolithography, and the growth rate of CNT microstructures depends on the local areal density of catalyst, which is analogous to loading effects in plasma etching process. Further, using a novel apparatus where the silicon substrate is resistively heated, we achieve CNT film thickness of 3 mm in just 20 minutes along with rapid (100°C/s) control of the substrate temperature and optically image the film during growth (Figure 1).

By placing a weight on the catalyst-coated substrate, we measure the force which can be exerted by a growing CNT film and demonstrate that the film thickness after a fixed growth time and the alignment of CNTs within the film decrease concomitantly with increasing applied force [2]. We utilize this principle to fabricate three-dimensional structures of CNTs (Figure 2) that conform to the shape of a microfabricated template. This technique is a catalytic analogue to micromolding of polymer and metal microstructures; it enables growth of nanostructures in arbitrarily-shaped forms and does not require patterning of the catalyst.

Finally, we perform combinatorial flow studies of CNT growth using an array of parallel microchannels fabricated by KOH etching of silicon [3]. We observe transitions in CNT yield and quality along the microchannels, grow CNT structures that are aligned by gas flows in the microchannels, and fabricate CNT-filled microchannels for applications such as microfluidic filters.
Prediction of Variation in Advanced Process Technology Nodes

N. Drego, A.P. Chandrakasan, D. Boning

Sponsorship: MARCO C2S2

As Moore’s Law forces the semiconductor industry into the sub-50-nm regime, process variability is proportionally becoming larger. Design cycles must simultaneously accommodate this increase in process variability and are thus being extended in order to ensure product robustness in the face of such manufacturing uncertainty. To facilitate the designer’s need to accurately model and simulate circuits in the face of variation, we seek to provide predictive statistical models for advanced technology nodes and/or novel transistor architectures. Coupled with predictive technology models (PTM) [1], these statistical models will allow designers to simulate designs in a robust manner during, or even prior to, the development phase of a new process technology.

As a basis for providing such models, we are developing simple digital circuits that ease the measurement and extraction of parameters. An example of such a circuit appears in Figure 1. This circuit employs a delay-based measurement to measure the current drive of each of the transistors highlighted in red. If the transistor is biased in the sub-threshold regime and the DIBL coefficient of the process is small enough, we can use the delay measurement as a proxy for threshold-voltage ($V_T$) variation (i.e., variations in time to charge the capacitor and disable the counter are dominated by $V_T$ variation as Figure 2 shows). However, if the DIBL coefficient is large enough, $\Delta V_T$ is no longer as dominant a source of current variation due to the increasing effect of channel-length variation ($\Delta L$). As a result, the circuit no longer functions as a proxy for $V_T$ variation. Nevertheless, the same circuit can be used to determine $I_{ON}/I_{OFF}$ and the sub-threshold slope of a given transistor. In advanced process technologies, a primary factor in determining the viability of the process will be the performance of the process with regard to short-channel effects (SCE), among which the $I_{ON}/I_{OFF}$ ratio and subthreshold slope are extremely significant. Furthermore, the SCE performance of novel transistor architectures such as the FinFET depends heavily on new critical dimensions such as body (fin) thickness [2]. The ability to efficiently measure variability due to such critical dimensions will enable quick determination of process feasibility. Future work includes fabrication of the aforementioned circuit on both mature as well as novel processes such as a FinFET process. Additionally, we would like to identify other circuits capable of such variation measurement to enable us to build complete statistical models.

REFERENCES


Parameterized Model Order Reduction of Nonlinear Circuits and MEMS

B. Bond, L. Daniel
Sponsorship: MARCO GSRC, NSF

The presence of several nonlinear analog circuits and micro-electro-mechanical (MEM) components in modern mixed-signal system-on-chips (SoC) makes the fully automatic synthesis and optimization of such systems an extremely challenging task. Our research is the development of techniques for generating parameterized reduced-order models (PROM) of nonlinear dynamical systems. These reduced-order models could serve as a first step towards the automatic and accurate characterization of geometrically complex components and subcircuits, eventually enabling their synthesis and optimization. Our approach combines elements of an existing non-parameterized trajectory piecewise linear method [1] for nonlinear systems with an existing moment matching parameterized technique [2] for linear systems. By building on these two existing methods, we have created four different algorithms for generating PROMs for nonlinear systems. The algorithms were tested on three different systems: a MEM switch, shown in Figure 1, and two nonlinear analog circuits. All of the examples contain distributed strong nonlinearities and possess some dependence on several geometric parameters.

The reduced-order models can be constructed to possess strong local or global accuracy in the parameter-space, depending on which algorithm is used. Figure 2 shows the output of one PROM created for the example in Figure 1 and compared to the field solver output of the full nonlinear system. In this example the system was parameterized in the width of the device and simulated at a parameter value different from the values at which the model was created. We found that in general the best algorithm is application-specific, but the PROMs are very accurate over a practical range of parameter values. For further details on parameter-space accuracy and cost of the algorithms, see [3].

Figure 1: Application example: MEM switch realized by a polysilicon beam fixed at both ends and suspended over a semiconducting pad and substrate expansion.

Figure 2: Center point deflection predicted by our parameterized reduced model of order 40, compared to a finite difference detailed simulation.

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The performance of several mixed-signal and RF-analog platforms depends on substrate effects that need to be represented in the library model with critical field solver accuracy. For instance, substrate-induced currents in RF inductors can severely affect quality and hence RF filter selectivity. We have developed an efficient approach to full-wave impedance extraction that accounts for substrate effects through the use of two-layer media Green’s functions in a mixed-potential-integral-equation (MPIE) solver. In particular, we have developed accelerated techniques for both volume and surface integrations in the solver.

In this work, we have also introduced a technique for the numerical generation of basis functions that are capable of parameterizing the frequency-variant nature of cross-sectional conductor current distributions. Hence skin and proximity effects can be captured utilizing many fewer basis functions in comparison to the prevalently-used piecewise-constant basis functions. One important characteristic of these basis functions is that they only need to be pre-computed once for a frequency range of interest per unique conductor cross-sectional geometry, and they can be stored off-line with a minimal associated cost. In addition, the robustness of these frequency-independent basis functions is enforced using an optimization routine.

We have shown in [2] that the cost of solving a complex interconnect system using our new basis functions can be reduced by a factor of 170 when compared to the use of piecewise-constant basis functions over a wide range of operating frequencies. Furthermore our volume and surface integration routines result in additional efficient improvement by a factor of 9.8 as shown in [1]. Our solver accuracy is validated against measurements taken on fabricated devices.

REFERENCES
This work proposes an optimization-based model-order-reduction (MOR) framework. The method involves setting up a quasi-convex program that explicitly minimizes a relaxation of the optimal H-infinity norm MOR problem. The method generates guaranteed stable and passive reduced models and it is very flexible in imposing additional constraints. The proposed optimization approach is also extended to the parameterized model reduction problem (PMOR). The proposed method is compared to existing moment-matching and optimization-based MOR methods in several examples. For example, a 32nd order parameterized reduced model has been constructed for a 7-turn RF inductor with substrate (infinite order) and the error-of-quality factor matching was less than 5% for all design parameter values of interest.

**REFERENCES**

Amorphous Zinc-Oxide-Based Thin-film Transistors

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Sponsorship: Hewlett-Packard

Recently, RF-sputtered zinc oxide-based field effect transistors (FETs) have been demonstrated with higher mobilities and performance than amorphous silicon, the dominant material used for display backplanes [1,2]. The low temperature processing possible for zinc oxide-based FETs [3] makes these materials compatible with flexible polymer substrates, but patterning with shadow masks limits feature size and accuracy. This project aims to develop a low-temperature, lithographic process for zinc oxide-based FETs, similar to one developed for organic FETs [4].

Our initial work focuses on two issues: determining optimal conditions for (1) growing the oxide semiconductor and (2) depositing high-quality oxide semiconductor and contact films on an organic polymer, parylene. For the former, top-contact, bottom gate ZnO FETs were fabricated on Si/SiO\(_2\) substrates, using SiO\(_2\) as the gate dielectric, Si as the gate, and sputtered indium-tin-oxide (ITO) as source/drain contacts. The RF sputtering power, total chamber pressure, and annealing temperatures were varied in a series of experiments; Figure 1 shows the current-voltage characteristics of a device from one set of conditions.

To determine optimal conditions for depositing high-quality films on an organic polymer, ITO films were deposited on an organic polymer dielectric, parylene, at different sputter rates. High stress in the oxide films on top of the soft organic polymer dielectric, parylene, may cause cracking and discontinuities in the film. Figure 2a shows a microscope photograph of the cracked surface of an ITO film sputtered at 80W on parylene; Figure 2b shows a continuous ITO film sputtered at 15W on parylene.

REFERENCES

Magnetic Rings for Memory and Logic Devices

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Sponsorship: Cambridge-MIT Institute, Singapore-MIT Alliance, Outgoing Marie Curie Fellowship, NSF

We use fabrication processes combining electron-beam lithography and photo-lithography to create prototypes for magneto-electronic devices based on small ring- and bar-shaped multilayered magnetic elements with widths in the deep-sub-micron regime. These small structures have potential uses in magnetic-random-access memories (MRAM), magnetic logic devices, and other magneto-electronic applications. Current MRAM devices rely on bar-shaped multilayered magnets, containing two magnetic layers separated by a thin layer of either a non-magnetic metal (spin-valves) or an isolator (magnetic tunneling junctions). The resistance of such elements depends on the relative orientation (parallel or anti-parallel) between the magnetization in the read-out (free) and storage (pinned) layers. On switching back and forth the magnetization direction of the free layer two different resistance levels can be detected, allowing for a non-volatile bit of data (“0” or “1”) to be stored in each element. As an alternative bit shape, MRAMs based on ring-shaped multilayered magnets have been recently proposed due to their robust magnetization reversal and the existence of flux-closure or “vortex” states.

We recently fabricated ring devices made from NiFe/Cu/Co/Au pseudo-spin-valves (PSVs) with non-magnetic contact-wires (see Figure 1). In these structures magneto-transport behavior is dominated by giant magnetoresistance (GMR). While the resistance of a PSV bar-shape element displays two resistance levels on cycling the free (NiFe) layer (Figure 2a), the rings display additional intermediate resistance levels [1] reached through abrupt transitions (Figure 2b). Additionally, the storage (Co) layer can be cycled into different states [1], allowing for profoundly different device responses on switching the free layer (Figure 2c). We have explored the switching mechanisms of PSV ring structures using micromagnetic simulations, as well as the effect of the contact configuration on the device response [2]. The sharp, low-field resistance changes in these PSV rings, which can be tailored by choice of ring dimensions and multilayer stack, will make them attractive for magnetoelectronic applications such as memories or logic devices that require multiple stable resistance levels. Most recently we are pursuing operating these devices using current pulses, rather than with an applied magnetic field.

Figure 1: Scanning electron micrographs corresponding to elliptical and circular ring devices made from NiFe/Cu/Co/Au multilayers and Ta/Cu non-magnetic contact wires. The outer dimensions of the rings ranges from 930 nm to 20 µm and the widths from 80 nm to 350 nm.

Figure 2: (a) Resistance versus applied field measurements on switching the free (NiFe) layer of a 200-nm-wide NiFe (6 nm)/Cu (5 nm)/Co (6 nm)/Au (4 nm) bar-shaped device. (b-c) resistance versus applied field corresponding to a 120-nm-wide NiFe (4 nm)/Cu (6 nm)/Co (8 nm)/Au (4 nm) elliptical ring, on switching the free layer with the storage layer in different states.

REFERENCES
Studies of Field Ionization Using PECVD-grown CNT Tips
B. Adeoti, A.I. Akinwande
Sponsorship: DARPA

The Micro Gas Analyzer project aims to develop the technology for portable, real-time sensors intended for chemical warfare and civilian air-purity control. For the analyzer, we are developing a field ionizer array based on gated CNTs. We plan to use arrays of CNTs because their small tip radii and high aspect ratio yield high fields at low voltage. One possible configuration for the device is to bias the CNTs to the highest potential and the collecting anode to the lowest potential. The electrons in the outer shell of the molecules tunnel out due to the ambient high electric fields, which serve to lower the unperturbed potential barrier seen by the electrons (Figure 1). The tunneling effect is a purely quantum-mechanical process whose probability of occurrence is strongly dependent on the applied electric fields [1]. We optimize the electron current by varying structural parameters in our device. The most relevant parameters include the radius of curvature, height, base radius and base angle of the grown tip; height and thickness of the tip; and the gate aperture. Varying the gate (or oxide) height without updating the height of the CNT yields the derivable result that the electric field is maximized with the tip peaks at about the same height as the gate. When the tip height is varied in sync with the height of the gate (or oxide), we see that an independent optimum height exists (Figure 2). The value of this height will depend, among other variables, on the electrostatic properties of the insulating material and the actual dimensions of the rest of the structure. These simulation results are being verified by experiment.

Figure 1: Representative picture of potential barrier faced by electron in a single molecule when the molecule is close to a tip biased at a high potential.

- Field applied at Tip = 2 x 10^8 V/cm
- Fermi sea (Tip)
- Unperturbed potential
- Perturbed Potential Well
- Distance of molecule from tip / Angstroms

Figure 2: Field factor β for various heights of gate. Tip height was varied in sync with gate height so that the tip remained within the range of the gate width. A field ionization configuration was used. [CNT] : roc = 5.01 nm | Base: angle=85°, radius=101 nm [GATE] : Aperture=1.01 μm | Thickness=0.301 μm | V_CNT = 10 V, V_GATE = 0 V, V_ANODE = -150 V

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Growth of Carbon Nanotubes for Use in Origami Supercapacitors

H.J. In, S. Kumar, Y. Shao-Horn, G. Barbastathis
Sponsorship: MARCO IFC, ISN, NSF SGER

The Nanostructured Origami™ 3D fabrication and assembly process can be used to create nanostructured, three-dimensional (3D) devices exclusively through existing 2D micro- and nano-fabrication techniques. Previously, the folding method was used in fabricating an origami supercapacitor with carbon electrodes [1]. The carbon electrode material was composed of 99 wt% Super P carbon black and 1 wt% polyvinylidene fluoride binder in N-methyl-2-pyrrolidone. This mixture was highly porous with a very high specific surface area and resulted in microscale supercapacitors with a high specific capacitance. However, the material had to be deposited manually using a probe tip, and its exact properties, e.g., surface area and variations in pore size could not be determined precisely. To enable in situ deposition of the electrode material, multi-walled carbon nanotubes (CNTs) were grown through a plasma-enhanced chemical-vapor deposition (PECVD) method in place of the manually deposited carbon paint. Moreover, as Figures 1 and 2 partially illustrate, growth conditions of the nanotubes determine their density, length, diameter, etc. Being able to control the nanotube properties will allow us to more carefully characterize the electrochemical properties of the origami supercapacitors. All nanotubes were grown using S. Kim group’s PECVD CNT deposition equipment.

![Figure 1: Low-density growth of carbon nanotubes. Each tube is approximately 2 microns long.](image1)

![Figure 2: High-density growth of carbon nanotubes.](image2)

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Self-Alignment of Folded, Thin-Membranes via Nanomagnet Attractive Forces

A. Nichol, W.J. Arora, F.J. Castaño, G. Barbastathis
Sponsorship: ISN, MARCO IFC

We are developing a thin-membrane self-alignment technique based on the attractive force between arrays of nano-patterned magnetic material (nanomagnets). The alignment scheme shown in Figure 1 will be applied in the Nanostructured Origami™ fabrication method [1], which involves first nano-patternning membranes using 2D lithography and then folding the membranes in a 3D configuration. We have shown that the scaling of the attractive force between magnets is favorable for this application. The research is moving towards a completely self-assembling, 3D nanofabrication method with better than 50-nm accuracy of feature placement for use in 3D photonics, 3D integrated circuits, and other 3D hybrid devices.

Nanomagnets with preferred magnetization in-plane can be patterned on thin-membranes with nano-scale precision using e-beam lithography, e-beam evaporation for the metallization steps, and liftoff processing. The alignment system uses a large array of nanomagnets to increase force and to average out the local errors in magnetic pole positions. A dynamic model of the system has shown a significant dependency on the number of magnets for the precision of the final alignment.

The magnetic alignment scheme is most effective when the folding takes place in an external magnetic field that holds a common magnetization direction for the nanomagnets. The addition of an external field allows the use of soft magnetic material operating at saturation. This reduces processing constraints because hard permanent nanomagnets at this scale are more difficult to fabricate than soft magnets. Furthermore, the torque due to the external field provides alignment about two axes of rotation. Using a dipole approximation along with finite element magnetic modeling (FEMM) [2], we have determined the alignment forces that can be achieved with this method. A plot of the forces between two 100 nm x 1 µm x 3 µm saturated iron nanomagnets appears in Figure 2. We are characterizing the alignment force experimentally using sensitive 300 nm-thick silicon nitride flexures that have been e-beam-patterned with iron nanomagnets.

![Figure 1: Schematic of the alignment system of nanomagnets. The two membranes are patterned with magnetic material that is magnetized in-plane by using an external magnetic field. When the coarse alignment brings them close enough, self-alignment occurs.](image)

![Figure 2: The force exerted on a single 100 nm x 1 µm x 3 µm magnetically saturated iron nanomagnet by similarly-sized magnets on the other membrane. The force is highly dependent on the separation between the membranes.](image)

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Nanostructured Origami™ is the 3D nanomanufacturing process that folds patterned membranes into useful 3D structures. Since the origami segments must be accurately aligned in the 3D folded state, the actuation mechanisms for Nanostructured Origami must be both controllable and repeatable. Accurate analytical models of the actuation are therefore necessary to expedite the design of complex origamis. We borrow techniques from the robotics community to simulate the motion of the origami as it folds from 2D to its 3D shape. The dynamics of the folding process are computed using wrench calculus [1] and are used to design a control scheme for folding the segments in the laboratory. A PD position control scheme is briefly described here.

To date, we have modeled the dynamics of two classes of origamis: accordions and single-vertex origamis [2]. In this abstract, we describe the analysis of a single-vertex origami referred to as the corner cube. The creases are modeled as revolute joints, which represent the generalized coordinates of the systems. The segments of the origami are assumed to remain rigid throughout the origami’s motion. This allows us to use screw calculus [1] to describe the geometry and kinematics of the corner cube. Since the corner cube has a closed chain kinematic topology, the motion of the segments is interdependent. The dependencies are manifested through holonomic equality constraints. The corner cube is then virtually cut at an unactuated joint, and the dynamics of the resulting one degree-of-freedom system are computed. The equations of motion of the system are then formed according to the principles outlined in [1]. These nonlinear equations can be integrated in time to simulate the trajectory of the origamis. The dynamic response of the corner cube subject to a PD controller is shown in Figure 1, and its trajectory is illustrated in Figure 2. The final folded state of the corner cube is also guaranteed to be stable for a stress actuation method since the stiffness matrix is positive definite.

![Figure 1: Dynamic response of driving joint of corner cube when subject to an input torque following the PD control law.](image1)

![Figure 2: Trajectory of corner cube as it folds from the flat to the folded state.](image2)

**REFERENCES**


Measuring Thermal and Thermoelectric Properties of Single Nanowires and Carbon Nanotubes

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Sponsorship: JPL, NSF

Knowledge of nanowire and carbon nanotube thermal and thermoelectric properties will be important for the thermal management of nanoscale devices that have recently been demonstrated (optoelectronic, sensing, and computing) and essential for the design of nanostructured thermoelectric materials. For nanowire diameters smaller than the bulk mean-free path of heat carriers (electrons and/or phonons), theory predicts that the thermal conductivity of these structures will be reduced when compared to similar bulk materials [1]. In order to experimentally verify these predictions, we are exploring several systems to measure the properties of single nanowires and carbon nanotubes.

Current work includes a basic platform to measure the thermal conductivity and specific heat of electrically conductive nanowires, such as the silicon nanowire shown below. Electron-beam lithography was used to pattern the leads of a four-point probe aligned to the ends of the nanowire. Joule heating of a suspended nanowire with thermally clamped ends results in a temperature rise of the nanowire due to its finite thermal resistance. This temperature rise can be measured by resistance thermometry (again using the nanowire) and used to calculate its thermal conductivity and specific heat. This technique is being adapted for an in situ TEM measurement, to enable high-throughput physical property measurements of many nanowires of various geometries and morphologies, and allow correlations with their atomic structure as determined by TEM.

Microfabricated metal lines can also be employed to measure electrically insulating nanowires. Using electron beam lithography, a metal heater line is fabricated such that a target nanowire crosses the center of the line. With the ends of the nanowire and heater thermally anchored, the nanowire removes a fraction of heat from the heater line, reducing the heater’s temperature rise, and thus making it possible to calculate the thermal resistance of the nanowire.

Figure 1: Contacts for a four-point probe measurement of a single silicon nanowire.

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Nanocomposites as Thermoelectric Materials


Sponsorship: NASA, Intel, Nanolab SBIR

Direct energy conversion between thermal and electrical energy based on thermoelectric effects is attractive for potential applications in waste heat recovery and environmentally-friendly refrigeration [1-2]. The energy conversion efficiency depends on the dimensionless figure of merit of the thermoelectric materials, ZT, which is proportional to electrical conductivity, square of the Seebeck coefficient, inverse of the thermal conductivity, and absolute temperature. At the current stage, the low ZT values of available materials restrict the efficient applications of this technology. Recently, significant enhancements in ZT were presented through the use of nanostructures such as superlattices. Previous works done by our group show that such improvement in superlattices is mainly attributed to the increased interfacial diffuse phonon scattering [3-4]. These studies lead us to pursuing the nanocomposite approach as a cost-effective alternative in developing high ZT materials.

Previously, we reported thermal conductivity reduction in SiGe nanocomposites. Through collaboration with Boston College group and Jet Propulsion Lab, we realized significant improvement in the ZT over that of SiGe used in the past NASA flights (Figure 1). We are also working on Bi$_2$Te$_3$ and PbTe nanocomposites. Our preliminary results on Bi$_2$Te$_3$ nanocomposites also show reduced thermal conductivities. Work is in progress to optimize the structures for further improving the ZT values.

| Figure 1: Measurements of temperature-dependent ZT. Nanostructured sample SGMA04 shows higher ZT than typical bulk alloy, “RTG SiGe.” |
| Figure 2: Temperature-dependent thermal conductivities of Bi$_2$Te$_3$ nanocomposite samples. Data of bulk materials are plotted for comparison. |

REFERENCES


CNT Assembly by Nanopelleting
S.D. Gouda, S. Kim, S.-G. Kim
Sponsorship: Intelligent Microsystems Center

We have developed a novel method of manufacturing and assembling process termed nano-pelleting [1-2], which refers to large-scale handling and long-range order assembly of individual carbon nanotubes (CNTs). The nano-pelleting concept overcomes the limitation of very small-scale order by embedding carbon nanotubes into micro-scale pellets. This technique includes vertically growing single strand CNTs, embedding a CNT into a polymeric pellet, separating a pellet, and transplanting a CNT. The CNTs are grown vertically, both individually and in bunches, on the patterned catalytic metal using a plasma enhanced chemical vapor deposition (PECVD) machine (Figure 1) built by us at MIT. The machine’s key feature is the control of the substrate temperature during the growth process. At the bottom of the ceramic heater, three thermocouples are connected to measure the temperature, which is controlled by the heater controller. Plasma is formed between anode and cathode by applying a DC voltage, which then decomposes acetylene into carbon that deposits below the Ni catalyst and leads to the formation of carbon nanotubes. The process sequence to make pellets is the following: coating PMMA on the silicon wafer, exposing the photo-resists using Raith 150 to obtain the desired patterns by varying the aperture size, dose, electric field, developing the photo-resist, depositing Ti/Ni (25nm), and lifting-off the resist to obtain Ni-catalyst nano-dots. Single stranded CNTs are grown in the PECVD machine with optimized process conditions as shown in Figure 2. On these isolated CNTs, SU-8 is spin-coated to form a thickness of 25 micro-meter. This SU-8 layer is exposed to UV light using an appropriate mask and then developed to form nano-pellets. The nano-pellets are released from the silicon substrate by manually breaking them with a spark needle. We are developing an in-plane AFM probe [3] with mechanically assembled CNT tips.

**REFERENCES**


In this project, an effective technique for site-selective, multicomponent assembly at the nano- and micro-scale has been created and quantitatively modeled. This approach offers great promise for assembling arbitrary (not necessarily periodic) systems of multiple different types of nanoscale components, such as electronics (memory, logic, interconnects, displays) and sensor systems.

The key elements of the approach follow. First, the topography of the substrate is modified to match the components’ 3D shapes. Then the substrate and components are coated with an adhesion promoter, such as a hydrophobic SAM for adhesion in a water-based environment. The components and substrate are placed in a fluid environment for the assembly process, and megahertz frequency ultrasound is applied to the fluid bath. Components contact the substrate randomly and adhere wherever they land; however, components that are not in shape-matched sites are removed by fluid forces initiated by the high frequency ultrasound. Components in shape-matched sites are selectively retained because their adhesive force is stronger than the removal forces.

Figure 1 is an optical micrograph showing the successful assembly of 600 nm and 2 µm diameter silica microspheres into designated sites on the substrate. Figure 2 shows how measured assembly yield of spheres into holes of slightly different sizes increases with the contact area between spheres and substrate.

This approach to assembly is inherently selective; since each component will adhere only in a shape- and size-matched site, geometrically distinct components will assemble only into their designated assembly sites. This allows the organizing information to be stored in the template initially, and permits components that may not be compatible with top-down manufacturing techniques to be added to the system later, with high positional precision. Work is underway to demonstrate this approach at smaller size scales and to create practical systems using this technology.
Nanostructured Origami™ describes a method of manufacturing three-dimensional (3D) nanostructures on a silicon wafer. Nanometer-scale structures are best fabricated with various two-dimensional (2D) lithography techniques. This project addresses the problem of building 3D structures using only 2D lithography. The general method of the Nanostructured Origami™ approach involves three steps: lithographically define micrometer-scale membranes and hinges; lithographically pattern nanostructures on these membranes; and release the membranes and actuate the hinges to fold into a 3D shape. Presently, we envision this technology to be well suited for fabrication of 3D photonic crystals and 3D diffractive optical devices.

We have developed a process to fold thin membranes of silicon nitride using stressed chromium hinges [1]. To date, these membranes have been patterned with gratings prior to being folded into 3D. However, arbitrary nanoscale features can be etched into or deposited onto the membranes. Experimental results are shown in Figures 1 and 2.

Current work focuses on reducing the radius of the folds. With the stressed chromium metal approach, we have demonstrated 30-µm-radius folds and by thinning the membrane at the hinge area, we predict that it is possible to achieve 5-µm-radius folds. However, it is desirable to have fold radii on the order of the membrane thickness, usually 1 µm or less. We are developing a new folding approach that uses the stress gradient created by ion implantation. The maximum strain generated is proportional to the implanted ion concentration, which can be much larger than that generated by the stressed chromium. Additionally, as the ion implantation takes place, the hinge area is sputtered and can be controllably thinned. The combination of these effects results in a much smaller radius. Preliminary experimental results are shown in Figure 2.

**Figure 1:** a) A 200-nm SiN membrane folded to 45° with a grating etched into the center. The grating was patterned by interference lithography and the period is 200 nm. The membrane is folded near the base by a strip of chromium. The fold radius is 34 µm. b) Three identical hinges (63-µm long) each folded to 120° to form a triangular shape out of a long SiN strip. The membranes are patterned with interference lithography inside the circular regions. The fold radius is 32 µm.

**Figure 2:** Single pixel lines written with a focused ion beam source (Ga+ ions, 30keV) folded a 200-nm-thick SiN membrane with a 500-nm radius. The folding strongly depends on the implanted dose; the line on the right had half the dose of the folded line on the left.

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Sponsorship: AFOSR, DARPA

This work [1] explores a method to precisely assemble two planar MEMS components. Our intended application is the assembly of the extractor electrode of an electrospray thruster, in which holes in the extractor must be aligned precisely with emitter needles or ridges (see [2] in this volume). In this method, the components can be accurately assembled by hand. Moreover, the assembly is made using a system of flexures, allowing considerable flexibility in the choice of materials and coatings for the components.

Figure 1 shows a diagram of our device. The extractor electrode (red) needs to be assembled in a recess on the base of the electrospray thruster (blue). To do this, the extractor is placed by hand in its recess. This step is easy as there are a few hundred micrometers of slack. The extractor is then rotated. As it rotates, features around its periphery force it to align its center to within 50 micrometers of its final position. As it continues the rotation, flexible fingers on the base part get flexed by the extractor, until the fingertips fall into notches in the sidewalls of the extractor.

Our devices, shown in Figure 2, were initially made out of Silicon using deep reactive-ion etching (DRIE). To show the flexibility of the method, we have also produced laser-cut polyimide extractors. The polyimide extractors have allowed us to achieve electrical insulation between the extractor and the rest of the device, which is vital for our intended application.

We have measured front-to-back alignment on all our silicon devices and found that they are within 9 micrometers RMS of their intended location. However, multiple assembly/dissassembly cycles on a specific device show that the position is repeatable to within 1.5 micrometers of standard deviation. This measurement suggests that much of the misalignment we are observing occurs due to misalignments during the various photolithography and bonding steps.

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REFERENCES
A Fully Microfabricated Planar Array of Electrospray Ridge Emitters for Space Propulsion Applications

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Sponsorship: AFOSR, DARPA

Electrospray thrusters work by extracting ions or charged droplets directly from a liquid surface using an electrostatic field and accelerating them in that field to produce thrust. This method could lead to more efficient and precise thrusters for space propulsion applications. The propellant liquid is generally placed at the tip of a needle to enhance the electrostatic field. The electrospray process limits the thrust from a single emitter needle. To get into the millinewton range will require an array with thousands of emitters. Batch microfabrication is well suited to making such an array.

We have designed and built a prototype thruster that consists of two silicon parts (Figure 1) made using deep reactive ion etching (DRIE) and SF6 plasma etching. The thruster base holds the electrospray emitters. Its surface is treated to control the areas where propellant can go. The extractor produces the electric field, which generates the electrospray. It is equipped with slits to allow the accelerated particles through. The two parts are positioned relative to each other using a kinematic mount, in which alumina balls rest in holes on the silicon dies (Figure 1). Alumina screws hold the assembly together.

In this design, we have replaced the needles that are typically used in electrospray thrusters by ridge emitters: vertical slabs with sharp tips spaced along their length (Figure 2). We have shown that our process for needles [1] can be extended to ridge shapes, and a modeling effort is underway to better control the shapes of the emitters.

Our thruster has been fired with the ionic liquid EMI-BF4. This experiment shows successful electrical insulation, even in the presence of the liquid. Challenges we now face are reducing the amount of emission that is intercepted by the extractor and determining where on the ridges the emission is coming from.

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**REFERENCES**

Thermal Management in Devices for Portable Hydrogen Generation

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Sponsorship: ARO MURI

The development of portable-power systems employing hydrogen-driven solid oxide fuel cells continues to garner significant interest among applied science researchers. The technology can be applied in fields ranging from the automobile to personal electronics industries. This work focuses on developing microreaction technology that minimizes thermal losses during the conversion of fuels – such as light-end hydrocarbons, their alcohols, and ammonia – to hydrogen. Critical issues in realizing high-efficiency devices capable of operating at high temperatures have been addressed, specifically, thermal management, the integration of materials with different thermophysical properties, and the development of improved packaging and fabrication techniques.

A new fabrication scheme for a thermally insulated, high-temperature, suspended-tube microreactor has been developed. The new design improves upon a monolithic design proposed by Arana et al.[1]. In the new modular design (Figure 1), a high-temperature reaction zone is connected to a low-temperature (~50°C) package via the brazing of pre-fabricated, thin-walled glass tubes. The design also replaces traditional deep reactive ion etching (DRIE) with wet potassium hydroxide (KOH) etching, an economical and time-saving alternative. A brazing formulation that effectively accommodates the difference in thermal expansion between the silicon reactor and the glass tubes has been developed.

Figure 1: Suspended-tube microreactor showing 2 pre-fabricated SiO$_2$ tubes, a microfabricated Si reaction chip, and a microfabricated Si frame. The modular design is assembled via a glass braze.

REFERENCES

Autothermal Catalytic Micromembrane Devices for Portable High-Purity Hydrogen Generation

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Sponsorship: ARO MURI

The high efficiency and energy density of miniaturized fuel cells provide an attractive alternative to batteries in the portable power generation market for consumer and military electronic devices [1-3]. The best fuel cell efficiency is typically achieved with hydrogen, but safety and reliability issues remain with current storage options. Consequently, there is continued interest in reforming liquid fuels to hydrogen. The process typically involves high temperature reforming of fuel to hydrogen combined with a low temperature PEM fuel cell, which implies significant thermal loss. Owing to its high hydrogen content (66%) and ease of storage and handling, methanol is an attractive fuel. However, partial oxidation of methanol also generates some CO, which may poison the fuel cell catalyst.

Previously [4] we successfully demonstrated hydrogen purification using thin (~200 nm) Pd-Ag membranes using electrical heating. Further, integration of these devices with LaNiCoO$_3$ catalyst allowed methanol reforming at 475°C with 47% fuel conversion [5]. Since microreactors possess high surface area to volume ratio, minimizing heat loss is important. Hydrogen flux across the Pd membranes is an equilibrium controlled process. Thus to achieve thermal management, the unextracted hydrogen, generated CO, and unreacted methanol can be completely oxidized in a separate reactor.

In the current work, we explore the realization of autothermal hydrogen generation by fabricating silicon-based reactors using bulk micromachining techniques. The hydrogen generation unit comprises a 200-nm palladium-silver membrane coated with a reformer catalyst while the combustor is loaded with platinum catalyst. High thermal conductivity of silicon ensures autothermal operation. Upon thermal isolation using vacuum packaging [6], we characterize the performance of this integrated, autothermal hydrogen generation system in terms of energy efficiency and hydrogen production.

REFERENCES

Self-powered Wireless Monitoring System Using MEMS Piezoelectric Micro Power Generator

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Sponsorship: NSF, Korean Institute of Machinery and Materials

A thin-film lead zirconate titanate Pb(Zr,Ti)O$_3$ (PZT), MEMS Piezoelectric Micro Power Generator has been integrated with a commercial wireless sensor, Telos, to simulate a self-powered RF temperature monitoring system (Figure 1). Such a system has many important applications, ranging from structure to rotary system monitoring. Telos consumes 2270 µJ for 221 ms per measurement. The PMPG and power management module are designed to satisfy such power requirements.

The first prototype of PMPG provides an average 1 µW, with a natural frequency of 13.9 kHz (Figure 2). It has an energy density of 0.74 mW-h/cm$^2$, which compares favorably to lithium ion batteries [1]. The second generation PMPG is designed to provide 0.173 mW of power at 3 V with a natural frequency of 150 Hz and maximum strain of 0.12% [2]. We increased the effective mass of the PMPG by adding a Si substrate with thickness of 525 µm to the beam structure. The increase in the effective mass increases the energy store in the device and its power output. The beam length is also increased to achieve a low resonant frequency. The third generation PMPG will use a serpentine structure, which can achieve a low frequency with minimum volume.

Since PMPG offers limited power, a storage capacitor and a power management module are implemented to power the sensor node at discrete time intervals [3]. The PMPG is first connected to a rectifier that converts AC to DC voltage. Each cycle consists of a charging interval, in which PMPG charges the capacitor, and operation intervals, in which Telos uses the energy from capacitor. We developed a test bed, which mimics that of a liquid gas pipe used in the Alaska where the PMPG device will be used to generate power for temperature sensors. Scaling/dimension factors as well as cost and robustness are considered in the design.

**REFERENCES**

An Integrated Multiwatt Permanent Magnet Turbine Generator

Sponsorship: ARL Collaborative Technology Alliance

There is a need for compact, high-performance power sources that can outperform the energy density of modern batteries for use in portable electronics, autonomous sensors, robotics, and other applications. Previous research efforts on a micro-scale, axial-flux, permanent-magnet turbine generator [1-2] culminated in a spinning rotor test stand that delivered 8 W DC output power through a diode bridge rectifier with an overall generator system efficiency of 26.6%. In these experiments, the generator rotor was mounted via a steel shaft to an air-driven, ball-bearing supported spindle and spun to the desired operational speed.

Current research efforts aim to fully integrate the permanent-magnet (PM) generator design into the silicon micro-turbine engine fabrication process and create devices that can deliver 10 W DC output power when driven by compressed air. The integrated generator will couple energy from the compressed air to the rotor through microfabricated turbine blades attached to the backside of the rotor. One important challenge in this integration process is the structural integrity of the magnetic rotor spinning at a tip speed near 300 m/s, or equivalently 450 krpm.

Based on power requirements, a 300-µm thick circular NdFeB PM with an inner radius of 2.5 mm and an outer radius of 5 mm must be embedded into the silicon rotor on top of a 150 µm FeCoV back iron. FEA analysis shows that the maximum principle stress at 450 krpm in the silicon rotor, 900-µm thick and 12 mm in diameter, with bonded annular PM and back iron pieces, will be approximately 180 MPa through the entire structure. This stress is well below the tensile strength of silicon and FeCoV. However, because the PM is brittle and has a typical tensile strength around 83 MPa, it is unclear whether the material will fracture. Tests are currently underway to characterize the reference strength and Weibull modulus of the PM, and from these results, a working rotor design will be proposed.

Figure 1: Conceptual schematic of the fully integrated surface-wound permanent magnet turbine generator. The bottom two wafers constitute the stator and coil winding of the generator while wafers 3, 4, and 5 form the magnetic rotor. A center-fed journal-bearing design is shown in the schematic, but an axial-fed design is also possible.

Figure 2: An FEA simulation for the magnetic rotor structure spinning at 450 krpm. Because of the fully-bonded boundary conditions, most of the load is carried by the silicon hub. The maximum principal stresses in the silicon, PM, and back iron are 176.7 MPa, 188.0 MPa, and 184.5 Mpa, respectively.

REFERENCES
Micro-scale Singlet Oxygen Generator for MEMS-based COIL Lasers

Sponsorship: DARPA, MDA

Conventional chemical oxygen iodine lasers (COIL) offer several important advantages for materials processing, including short wavelength (1.3 µm) and high power. However, COIL lasers typically employ large hardware and use reactants relatively inefficiently. This project is creating an alternative approach called microCOIL. In microCOIL, most conventional components are replaced by a set of silicon MEMS devices that offer smaller hardware and improved performance. A complete microCOIL system includes micro-chemical reactors, micro-scale supersonic nozzles, and micro-pumps. System models incorporating all of these elements predict significant performance advantages in the microCOIL approach [1].

Initial work focuses on the design, microfabrication, and demonstration of a chip-scale singlet oxygen generator (SOG), a micro-chemical reactor that generates singlet delta oxygen gas to power the laser. Given the extensive experience with micro-chemical reactors over the last decade [2-4], it is not surprising that a micro-SOG would offer a significant performance gain over large-scale systems. The gain stems from basic physical scaling; surface-to-volume ratio increases as the size scale is reduced, which enables improved mixing and heat transfer. The SOG chip demonstrated in this project, shown in Figure 1, employs an array of micro-structured packed-bed reaction channels interspersed with micro-scale cooling channels for efficient heat removal. Production of singlet oxygen has been confirmed via spontaneous emission (as shown in Figure 2) and mass spectrometry techniques. The yield (or fraction of singlet oxygen produced) is estimated at 70%, making the micro-SOG competitive with macro-scale alternatives.

![Figure 1: Photograph of completed microSOG device.](image1)

![Figure 2: The IR spectrum measured at the µSOG gas outlet. The peak at 1268 nm indicates the spontaneous decay of singlet oxygen into its triplet state.](image2)

REFERENCES:


For a number of years, batteries have not kept up with the fast development of microelectronic devices. The low energy densities of even the most advanced batteries are a major hindrance to lengthy use of portable consumer electronics, such as laptops and of military equipment that most soldiers carry today. Furthermore, battery disposal constitutes an environmental problem. Hydrocarbon fuels exhibit very high energy densities in comparison, and micro-generators converting the stored chemical energy into electrical power at even modest levels are therefore interesting alternatives in many applications. This project focuses on building thermophotovoltaic (TPV) micro-generators, in which photocells convert radiation from a combustion-heated emitter into electrical power. TPV is an indirect conversion scheme that goes through the thermal domain and therefore does not exhibit very high efficiencies (10-15% max). However, because of its simple structure and because the combustor and photocell fabrication processes do not need to be integrated, the system is simpler to micro-fabricate than other generator types, e.g., thermoelectric systems and fuel cells. It is also a mechanically passive device that is virtually noiseless and less subject to wear than engines and turbines. In this TPV generator, a catalytic combustor, the suspended micro-reactor (SuRE) (Figure 1), is heated by combustion of propane and air, and the radiation emitted is converted into electrical energy by low-bandgap (GaSb) photocells. Net power production of up to 1 mW has been achieved [1], constituting a promising proof of concept. A new version of the SuRE is currently under fabrication. This new design (Figure 2) aims to address several problems existing in the earlier version, including fabrication difficulties, low burst pressure of the tubes, and low emitter surface area.

**REFERENCES**

MEMS Vibration Harvesting for Wireless Sensors
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Sponsorship: Cambridge-MIT Institute, NSF

The recent development of “low power” (10’s-100’s of µW) sensing and data transmission devices, as well as protocols with which to connect them efficiently into large, dispersed networks of individual wireless nodes, has created a need for a new kind of power source. Embeddable, non-life-limiting power sources are being developed to harvest ambient environmental energy available as mechanical vibrations, fluid motion, radiation, or temperature gradients [1]. While potential applications range from building climate control to homeland security, the application pursued most recently has been that of structural health monitoring, particularly for aircraft.

This SHM application and the power levels required favor the piezoelectric harvesting of ambient vibration energy. Current work focuses on harvesting this energy with MEMS resonant structures of various geometries. Coupled electromechanical models for uniform beam structures have been developed to predict the electrical and mechanical performance obtainable from ambient vibration sources.

The uniform models have been validated by comparison to prior published results [2] and verified by comparison to tests on a macro-scale device [5]. Models of a uniform harvester with proof mass are currently undergoing macro-scale testing and validation. A non-optimized, uni-morph beam prototype (Figure 1) has been designed and modeled to produce 30 µW/cm³ [3]. A MEMS fabrication process for a prototype device is presented based on past work at MIT [4]. Dual optimal frequencies with equal peak powers and unequal voltages and currents are characteristic of the response of such coupled devices when operated at optimal load resistances (Figure 2). Design tools to allow device optimization for a given vibration environment have been developed for both geometries.

Future work will focus on fabrication and testing of optimized uni-morph and proof-of-concept bi-morph prototype beams. System integration and development, including modeling the power electronics, will be included.

\[\text{Figure 1: Illustration of MPVEH unimorph configuration (left) and SEM of a prototype device (right, courtesy of S.-G. Kim).}\]

\[\text{Figure 2: Power vs. normalized frequency with varying electrical load resistance [3].}\]

\[\text{REFERENCES}\]

Fabrication and Structural Design of Ultra-thin MEMS Solid Oxide Fuel Cells

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Sponsorship: ARO

Microfabricated solid oxide fuel cells are being investigated for portable power applications requiring high energy densities [1-2]. Reducing the thickness of the fuel cell stack (anode, electrolyte, and cathode) improves the electrochemical performance over that of traditional devices. This motivation for thinner structures, combined with significant temperature excursions during processing and operation (~600-1000 °C), leads to a major challenge of thermomechanical stability of such membranes. Figure 1 shows a buckled electrolyte/SiN thin film. To predict and control structural stability and failure, the structural characterization of thin films is being investigated.

Our group has characterized the residual stress and microstructure of the electrolyte layer. Complete studies were done on residual stress in sputter-deposited yttria-stabilized zirconia (YSZ) thin films (5 nm-1000 nm thickness) as a function of substrate temperature [3]. The results indicate variations in intrinsic stress from ~-0.5GPa to ~50 MPa as in Figure 2. Changes in microstructure are characterized using x-ray diffraction of as-deposited and annealed films and correlated with relevant mechanisms/models of residual stress evolution. Based on the design frameworks using the data above, a large-area full fuel cell stack (anode, electrolyte, and cathode) has been fabricated and tested to be thermomechanically stable at high operating temperatures. Tri-layers (Pt-YSZ/YSZ/Pt-YSZ, 50-200-µm wide, each 250-nm-thick) were sputter-deposited at high temperature (500-600°C). Devices are being tested for electrochemical performance and power generation. In addition, proton-conducting electrolytes, typically capable of significant power generation at temperatures lower than YSZ are also being investigated in ultra-thin film form. Crack-free barium cerium-yttrium-oxide (BaCeYO) films with uniform thickness (300-500-nm thick) have been successfully sputter-deposited. Electrochemical and residual stress characterization for this material is currently underway.

Additional ongoing work includes bulge-testing to determine the electrolyte’s elastic/thermal/fracture properties in ultra-thin membrane form, investigation of the mechanical and chemical properties of anode cathode materials, and nonlinear modeling of film postbuckling and failure.

- Figure 1: Postbuckled YSZ/SiN membranes on Si. Displacement contour plot.

- Figure 2: The YSZ electrolyte film stress as a function of film thickness.

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Tomographic Interferometry for Detection of Nafion® Membrane Degradation in PEM Fuel Cells

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Sponsorship: DuPont-MIT Alliance

At present, membrane degradation and failure are the main limitations on the lifetime of proton exchange membrane (PEM) fuel cell systems [1]. Nafion® membranes, produced by DuPont, have excellent proton conductivity and relatively good chemical stability. However, fundamental studies of the degradation mechanisms of Nafion membranes are needed in order to optimize membrane design. Our goal is to correlate indirect and ex-situ measurements with in situ monitoring of membrane microstructural changes in order to better understand how the membrane thins or forms pinholes and voids.

We are conducting atomic force microscopy (AFM) studies of both fresh and degraded ex-situ Nafion samples to study the changes in the microstructure of the Nafion as it degrades. These studies will be used to guide optical characterization of the Nafion® membrane in situ. The goal of the in situ optical studies is to construct a refractive index profile of the membrane while the fuel cell is in use. We place the fuel cell system inside one leg of a Mach-Zehnder interferometer (Figure 1). The membrane, which has an average refractive index of 1.35 (room temperature, 20% RH) [2], will act as a phase object, and the phase delay at any point in the x-y plane is proportional to the refractive index at that point. Thus, a 2D map of the refractive index in the membrane can be constructed from the interference pattern, which is recorded on a CCD camera (Figure 2). The refractive index is related to membrane density at each point by the Lorentz-Lorentz relation. We focus on density changes due to temperature, pressure, and humidity, which occur on different orders of magnitude. Simulations are used to solve the forward problem through the optical system and to compute the inverse problem, which can then be used to estimate the refractive-index profile of the membrane from the measured interference pattern.

In situ profiling of the Nafion membrane will allow us to observe exactly where and how the membrane degrades. Our approach is non-invasive and provides a 2D distribution of water uptake, stresses, and micropores in the membrane. The technique can be extended to 3D with tomographic techniques. A better understanding of the mechanisms of membrane failure will give insight into membrane and fuel cell design.

Figure 1: Experimental setup of PEM fuel cell within a Mach-Zehnder interferometer. Fringe patterns yield a map of the refractive index of the fuel cell membrane.

Figure 2: Measured fringe pattern (right) and the corresponding intensity vs. time plot for several specific points (left) for a Nafion® membrane as it cools. Chirped sinusoid is a result of decreasing temperature.

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