*Opposite page:* 

(Top) Microfluidic cell-adhesion assay device that is fabricated using rapid prototyping techniques; (Middle) Cell electroporation lysis device that contains arrays of electrodes; (Bottom) Organelle sorting device that uses miniaturized isoelectric focusing field flow fractionation technique.

*Courtesy of H. Lu, S. Gaudet, L. Koo, P. Sorger, D. Lauffenburger, and L. Griffith (K. Jensen and M. Schmidt)* 

Sponsor: DARPA Bio-Info-Micro

# **Microelectromechanical Devices**



# **Microelectromechanical Devices**

- Microspectrometer Using Electrostatic or Piezoelectric Tunable Gratings
- Micromachined Structures for Microphotonics
- Electronic Detection of DNA by its Intrinsic Molecular Charge
- Development of Microfluidic Device for Study of Chemotaxis
- Integration of Heterogeneous Microfluidic Separation
- Platforms for Rapid Screening and Analysis of Biological and Chemical Processes
- Microfabricated Fluidic Devices for Cell Adhesion Assay, Cell Lysis, and Subcellular Component Separations
- Vapor Microbubbles for Cell Actuation
- Microfluidic Logic
- A Differential Microcantilever Stress Sensor for Biomolecular Detection
- Self Activated Microcantilever-based Gas Sensor
- Gas-Liquid Flow, Separation and Mass Transfer in MicroChemical Systems
- Microreactors for Catalyst Testing
- Scalable Multiphase Microchemical Systems for Direct Fluorination
- MicroChemical Systems for Separations
- Three Dimensional Integration of Microfluidic Devices
- Low Leakage Micro Switch Valves for Gas Chromatography
- Fuel Processing in Microfabricated Chemical System
- Microchemical Systems for Fuel Processing and Conversion to Electrical Power
- Piezoelectric Micro Power Generator (PMPG): A Novel MEMS Based Electrical Power Source
- Development of Hydrocarbon-Fueled Silicon Combustors for Micro Gas Turbine Engines
- Thermophotovoltaic Micro-Generators
- Micro-Fuel Cell
- Lateral RF MEMS Switch with PZT Actuation
- MEMS Tunable Capacitor and LC Tank
- DRIE-Fabricated Curved-Electrode Zipping Actuators with Low Pull-In Voltage
- MEMS Amplification of Piezoelectric Strain for In-Plane Actuation
- Water-Immersible Micromachined Pb(ZrTi)O<sub>3</sub> Thin Film Actuator

# continued **Microelectromechanical Devices**

- Wafer Bond Alignment and Strength Characterization
- Microelectromechanical (MEMS) Thin Film Stress Sensors
- 3D Nanomanufacturing via Folding of 2D Membranes
- Nano-Scale Machining with Femtosecond Laser Pulses

# Microspectrometer Using Electrostatic or Piezoelectric Tunable Gratings

#### Personnel

W.-C. Shih, C. Hidrovo, C. W. Wong, and Y.-B. Jeon (S.-G. Kim and G. Barbastathis)

# Sponsorship

n/a

This project seeks to build a "true" miniature optical spectrometer utilizing analog tunable gratings. Miniaturization is desirable for portable devices and applications which parallel processing benefits. However, in optics there are two fundamental problems when systems are small: the decrease of numerical aperture and optical path length. The combination of these two problems degrades the resolution of such a system. Existing technologies have tried to maintain performance through maximizing numerical aperture and optical path length under constraints miniaturization superimposes. However, we believe this is not the way if true miniaturization is desired. Instead we confront the aperture and path length deficiency issues and try to resolve them with the aid of computational imaging and optical diversity.

Computational imaging is widely used in the context of image processing for deblurring or restoration purposes. Since every image is formed through convolving a system function with the object, restoration is essentially the inverse process. The system function can be generated by motion blur, diffraction, or aberration, etc. If one can model the convolution process properly and use appropriate numerical algorithms, the result of the inverse problem may be better than the original image. For our case, the object is a spectral intensity distribution and the convolver is the Point Spread Function (PSF) of the system. Optical diversity is essentially a method which allows us to change the system transfer function at minimal cost, in our case, actuation of the tunable grating. Various transfer functions not only increase the robustness of inversion by over-constraining the system, but also give us adaptive spectral resolution capability.

The electrostatic grating is shown in Figure 1. An SOI wafer with 25  $\mu$ m thick device layer is etched with DRIE until the buried oxide is exposed. The buried oxide is removed successively with wet etching. The period of

the grating is 12  $\mu$ m, and the grating can be actuated by two comb-drives on both sides. The piezoelectric grating is shown in Figure 2. The grating structure is made of platinum and silicon nitride. The actuators are made of two platinum electrodes sandwiching the thin film PZT.

We set up a grating monochromator with the tunable grating. Due to the cross-leakage and unavoidable noise in the measurement, the system is ill-conditioned. There are many methods to resolve this such as Wiener filtering and maximal-likelihood. We used Tikhonov regularization with positivity constraint to extract the real spectrum out of the "blurred" spectrum due to miniaturization. The real spectrum and the recovered spectrum are shown in Figure 3. Optical diversity utilizes the fact that the grating is tunable, and we can change the system transfer function upon actuation. Figure 4 shows an Improved recovered spectrum using Computational Imaging with Optical Diversity (ICIOD).



Fig. 1: Electrostatic grating.



Fig. 2: Piezoelectric grating.



*Fig. 3: Spectrum recovered using deconvolution VS real spectrum of an interference filter.* 



*Fig. 4: Spectrum recovered using ICIOD VS real spectrum of an interference filter.* 

# Micromachined Structures for Microphotonics

# Personnel

D. Seneviratne and Y. Avrahami (H.L. Tuller), with M. Watts, H. Haus, and G. Nielson (G. Barbastathis)

# Sponsorship

Multidisciplinary program supported by Pirelli and administered by the Microphotonics Center

Binary-switchable and dynamically-tunable micromachined structures are being designed and fabricated to operate in conjunction with optical resonators and photonic band gap structures to achieve optical switching, multiplexing/de-multiplexing, and wavelength adddrops in Si integrated photonic systems. The work is being performed in conjunction with a multidisciplinary group that is working on the integration of microphotonic devices onto silicon wafers. Novel, multi-functional, optical material systems that can be readily integrated with Si platforms are also being investigated.

# **Electronic Detection of DNA by its Intrinsic Molecular Charge**

#### **Personnel** E. Cooper and J. Fritz (S. Manalis)

# Sponsorship

DARPA Bio-Info-Micro and AFOSR

A wide range of techniques for detecting nucleic acids is based on their hybridization to DNA probes on a solid surface. In the methods used most routinely, the physical nature of the readout requires the attachment of reporter molecules such as fluorescent, chemiluminescent, redox or radioactive labels. Although labeldependent methods achieve the highest sensitivities, eliminating the labeling steps has the advantage of simplifying the readout and increasing the speed and ease of nucleic acid assays. This is especially desirable for characterizing infectious agents, scoring sequence polymorphisms and genotypes, and measuring mRNA levels during expression profiling. The development of label-independent methods that can monitor hybridization in real-time and that are simple and scalable is still in its infancy.

We have demonstrated the selective and real-time detection of label-free DNA using an electronic readout (Fritz et al. PNAS 2002). Microfabricated silicon field-effect sensors were used to directly monitor the increase in surface charge when DNA hybridizes on the sensor surface (See Figure 5). The electrostatic immobilization of probe DNA on a positively charged poly-L-lysine layer enables rapid hybridization at low ionic strength where field-effect sensing is most sensitive. Nanomolar DNA concentrations can be detected within minutes, and a single base mismatch within 12mer oligonucleotides can be distinguished by using a differential detection technique with two sensors in parallel (See Figure 6). The sensors were fabricated at the MTL and show promise for future electronic DNA arrays and rapid characterization of nucleic acid samples. This approach demonstrates the most direct and simplest translation of genetic information to microelectronics.



Fig. 5: (a) and (b) Electrolyte - insulator - silicon interface of a n-type field-effect sensor. DNA exhibits one intrinsic negative charge per base at its sugar-phosphate backbone. Probe DNA is electrostatically bound to a layer of PLL on the surface. (a) Binding of negatively charged target DNA to its complementary probe DNA at the sensor surface extends the depletion region (black arrow) in the silicon portion of the sensor compared to (b) where no binding occurs to non-complementary probe DNA (blue). (c) Optical image of a device consisting of field-effect sensors at the terminus of two cantilevers. The cantilevers are 500  $\mu$ m long, 75  $\mu$ m wide and 3  $\mu$ m thick. (d) Cross section of a cantilever field-effect sensor. The sensing area at the terminus of the cantilever is electrically connected to a metal contact on the substrate by a layer of highly doped silicon inside the cantilever.



Fig. 6: Differential surface potential from two sensors which were functionalized with probe oligonucleotides A and Am which differ only in a single base. Control solutions with non-complementary target oligonucleotide cB show no differential signal, while injection of 80 nM of complementary sequences cA and cAm both show a distinctive hybridization signal.

# Development of Microfluidic Device for Study of Chemotaxis

**Personnel** M. Shur (C. F. Dewey)

#### Sponsorship DARPA

Cells exhibit a highly complex and integrated response to chemical gradients that requires quantitative explanation. Several theoretical models of a cell's ability to sense a gradient have been formulated. However, the inability to produce a linear gradient of chemoattractant and maintain it at steady state has prevented researchers from obtaining reproducible, quantitative results and validating the models.

Currently, the Dunn chamber assay is widely used for studying chemotaxis. In order to study slow-moving cells such as fibroblasts, a gradient must be maintained at steady state for four or more hours, which is impossible to do in the static Dunn chamber.

The multilayer application of soft lithography allows for peristaltic pumping and valving modules to be built into the device (See Figure 7). The overall goal is to manufacture a device on a chip with integrated modules for pumping fluids and cells, mixing various concentrations of reagents, placing cells in designated areas, and controlling chamber temperature to provide a viable environment for the cells. Ultimately, the chemotaxis chamber will be a universal "lab on a chip" that can be used to study different types of cells on various substrates and to probe potential chemoattractants at a range of concentrations.

The geometry of the current design allows for several experiments to be run simultaneously. The diverging flows of chemoattractant and buffer allow for several discrete gradients to be established.



*Fig. 7: Two layer mask for the chemotaxis chamber containing valves and peristaltic pumps.* 

# Integration of Heterogeneous Microfluidic Separation

**Personnel** Y. C. Wang and M. H. Choi (J. Han)

#### Sponsorship DARPA

One of the most important separation methods in the field of proteomics is 2-D protein electrophoresis separation. It is the only method to separate over 1,800 proteins in a single run with satisfying reproducibility now. The first dimension separation is IsoElectric Focusing (IEF) which can focus proteins into different groups by their pI values. Then, Sodium Dodecyl sulfate polyacrylamide gel electrophoresis (SDS-PAGE) is performed to separate these protein groups by their molecular weight.

Our goal here is to build an on-chip multidimensional separation system for separation of biomolecules, especially proteins. We have developed IEF and SDS-PAGE techniques on a microfluidic chip format. Miniaturization of these techniques allows us to perform these processes in very short microchannels (several millimeters or less) within a minute, which is an order of magnitude improvement in speed. The methods and chip architecture for these separations have been selected specifically for the eventual goal of their integration to two-dimensional 2D protein separation. IEF of proteins was achieved with commercially available ampholyte in a microchannel (1cm or shorter), fabricated on either poly(dimethylsiloxane) (PDMS) or glass substrates. Several naturally fluorescent and labeled proteins were analyzed with this technique (See Figure 8). We also demonstrated SDS-PAGE in a chip by photopolymerizing and photopatterning polyacrylamide gel in a microchannel. A protein marker sample (20.1 ~ 205 kDa) was analyzed, and the peak movement was monitored by video microscopy (See Figure 9).

Miniaturizing the length of separation channel in IEF and SDS-PAGE techniques leads to a number of improvements over their conventional counterparts. 1) Separation of protein peaks could be achieved very quickly, typically within 30s. 2) The electric potential to be applied across the channel was greatly decreased, which is another engineering constraint in future integrated systems. 3) Instead of mobilizing or eluting focused peaks, a microscope optic could be used for imaging of the channel in real time for a faster analysis. These advantages can be exploited for

constructing future microfluidic 2-D protein separation systems, which is highly desirable for proteomics research.

Our next goal in this project is to develop technologies to combine these heterogeneous separation systems, and to solve any issues arising from such integration. The success of this project could lead to a generic solution for other integrated microfluidic systems for (bio)molecule analysis, where several fluidic components should be interfaced without compromising the function of each components.



Fig. 8: Isoelectric focusing in 4mm channel. Electric field was 35.7 V/cm. Carbonic anhydras.e II was labeled by cysteine-specific labeling (rhodamine-maleimide), while R-phycoerythrin and EGFP are naturally fluorescent. The focusing was achieved within 30 s after applying the electric field. Wider channel regions at both ends are filled with catholyte and anolyte, respectively, and pH gradient was established within 4mm channel.



blue dye added to the sample. The width of dye band is much larger than other protein bands, suggesting no stacking effect for small dye molecules.

# Platforms for Rapid Screening and Analysis of Biological and Chemical Processes

## Personnel

P. Boccazzi, H. Lee, P. Lessard, N. Szita, A. Zanzotto, and B. Zhang (K. F. Jensen, P.E. Laibinis, R.J. Ram, M.A. Schmidt, and A.J. Sinskey)

# Sponsorship

DuPont MIT Alliance

This is a multidisciplinary research program aimed at developing new platforms for bioprocess discovery and development, specifically banks of miniaturized, automated bioreactors, each with integrated bioanalytical devices, and all operating in parallel (see Figure 10). Such systems will address the continuing demand in bioprocess science and engineering for fast and accurate analytical information that can be used to rapidly evaluate the interactions between biological systems and bioprocess operations. Moreover, the microbioreactors will provide the platforms for efficiently incorporating modern tools of biology (genetics, enzymology, bioinformatics) to improve bioprocess screening and development. Applying microfabrication technology to bioprocess development should result in rapid screening of strains and metabolic pathways as well as speed up of transfer of cell cultures in production. In order to realize the microfermentors, we are addressing the following critical issues: (i) design and fabrication strategies for microfermentors; (ii) integration of novel optical sensors; (iii) sensitivity of the analytical devices; (iv) biocompatibility of the materials; (v) appropriate biological systems for evaluating performance of the microfermentors; and (vi) benchmarking of microfermentation against traditional bioprocessing methodologies.



*Fig.* 10: (Top) *Schematic of microbioreactor with optical, on-line monitoring of optical density (cell concentration), Dissolved Oxygen* (DO), and pH. (Bottom) *Example of data obtained with on-line sensors.* 

# Microfabricated Fluidic Devices for Cell Adhesion Assay, Cell Lysis, and Subcellular Component Separations

# Personnel

H. Lu, S. Gaudet, L. Koo, P. Sorger, D.Lauffenburger, and L. Griffith (K. F. Jensen and M. A. Schmidt)

# Sponsorship

DARPA Bio-Info-Micro

This project is part of the DARPA Bio-Info-Micro program at MIT aimed at understanding signal transduction pathways in cell decision processes, in particular apoptosis (programmed cell death) and cell-biomaterial interactions. We focus on the development of advanced micro-scale analytical devices for efficient analysis of small populations of cells, their subcellular components, and intracellular proteins of interests.

Cell adhesion is important in many fundamental biological phenomena and medical applications. We miniaturized a shear flow chamber to perform cell adhesion assays that probe the cell-substratum interactions. The advantages include much larger dynamic range of shear forces, ease of analysis, and small sample requirement (in terms of both cells and biomaterials). These devices were fabricated using rapid prototyping techniques and were demonstrated for both short-term and long-term cell culture, biochemical stimulation, and adhesion analysis.

In proteomics and signal transduction studies, the demand to identify the location and amount of proteins poses challenges to subcellular separation and sample preparations. Current technologies involve laborious and time-consuming procedures that require large sample volumes (>10<sup>6</sup> cells). For protein profiling, the organelle separation needs to be fast, parallel, and automated to match the great number of experiments needed. We developed microfluidic systems that handle small number of cells ( $\sim 10^3$ ), lyse them, and separate out the organelles of interest (See Figure 11). Cell lysis by electroporation is accomplished in a device containing multiple metal saw-tooth electrodes and flow channel. Alternating electric field and appropriate voltages are used to minimize water electrolysis and to maximize cross cell membrane potential while minimizing impact on the organelles. Once cells are lysed, organelles of interest, such as mitochondria and nuclei, are separated from the resulting lysate by isoelectric focusing in a microfabricated device containing parallel electrodes and fractionation collection channels. The performance of the

device has been modeled to provide understanding of isoelectric focusing on the micron scale with particles that are amphoteric (such as organelles). Current research extends this method to more separations on the molecular level and the devices are used to address fundamental biological questions underlying signal transduction pathways.



Fig. 11: (Top) Microfluidic cell-adhesion assay device that is fabricated using rapid prototyping techniques; (Middle) Cell electroporation lysis device that contains arrays of electrodes; (Bottom) Organelle sorting device that uses miniaturized isoelectric focusing field flow fractionation technique.

# Vapor Microbubbles for Cell Actuation

**Personnel** R.A. Braff and A.L. Gerhardt (M.L. Gray, M.A. Schmidt, and M. Toner)

#### Sponsorship

AfCS (Alliance for Cellular Signaling)

Microfluidics is becoming increasingly important to the success of a wide variety of micromachined devices, particularly those with biological applications. However, many obstacles still exist in the production of a robust and simple microfluidic device. One area that is in need of improvement is microfluidic actuators, valves, and pumps.

We are developing guidelines to use thermally formed microbubbles as a means of fluidic actuation. The use of microbubbles is attractive due to the simple fabrication and operation of such devices. However, prior work in this area was hindered by several issues inherent to vapor bubble formation that severely limited the reliability of bubble-based devices. We have shown that it is possible to control the location at which bubbles form, to control the size of the bubbles, to make formation temperature more repeatable and reduced, and to have bubbles that collapse completely in less than 10 seconds (See Figure 12).

The achievement of controllable microbubbles makes possible many microfluidic applications, one of which we have demonstrated. We have built a device that is capable of capturing, holding, and selectively releasing single bioparticles using microbubble actuation (See Figure 13). The next generation bioparticle actuator is in the development stages of being scaled into an array for the analysis of a large population of individual cells.

The arrayed bioparticle actuator features the ability to observe cells from both vertical positions, two cellular medium channels to facilitate experimental and control fluid flows simultaneously, facile four-cell observation in one microscope view, and a pogo-pin contact scheme for short chip replacement times between assays (See Figure 14).



*Fig. 12: A platinum heater with machined bubble nucleation sites, before and after bubble formation.* 



Fig. 13: Schematic of microbubble bioparticle actuator. (a) A small backflow draws particle into capture well. (b) A bulk flow sweeps uncaptured particles away. (c) The platinum heater is turned on, and a vapor bubble begins to form in the bubble chamber. (d) The volume expansion of the bubble creates a jet of fluid that releases the particle from the capture chamber and is entrained in the bulk flow.

0	-	
(A)	(B)	(C) T=0 seconds
Direction of flow		
(D) T=0.77 seconds	(E) T=0.87 seconds	(F) T=2.66 seconds

Fig. 14: Microbubble bioparticle actuator in operation. In (A) and (B), a  $10\mu$ m polystyrene bead is drawn into the capture well where it is held against a bulk flow in (C). In (D), the bead is released by a jet created by a bubble in the chamber below. In (E) and (F), the bead is entrained in the bulk flow.

# **Microfluidic Logic**

# Personnel

T. Thorsen

# Sponsorship

n/a

Advanced image analysis tools are used in the Thorsen group in Hatsopolous Microfluids Laboratory at M.I.T. to complement core research structured around addressable silicone microfluidic networks. Monolithic multilayer elastomeric devices, which use multiple thin polymer layers to create three-dimensional microfluidic networks, are an important platform from which simple valve and pump structures can be used to build complex dynamic microfluidic networks. These elastomeric devices have the advantage of low cost, rapid prototyping, scalability, and highly flexible design constraints.

We developed high-density microfluidic chips that contain plumbing networks with thousands of micromechanical valves and hundreds of individually addressable chambers (See Figure 15). These fluidic devices are analogous to electronic integrated circuits fabricated using Large Scale Integration (LSI). A key component of these networks is the fluidic multiplexor, which is a combinatorial array of binary valve patterns that exponentially increases the processing power of a network by allowing complex fluid manipulations with a minimal number of inputs.

The simple, modular fabrication technology has made the project an attractive platform for the Undergraduate Research Opportunities Program (UROP) as an educational tool providing the students with hands-on research experience in design, manufacture, and fluid mechanics.



Fig. 15

# A Differential Microcantilever Stress Sensor for Biomolecular Detection

#### **Personnel** C. Savran and T. Burg (S. Manalis)

#### Sponsorship

DARPA Bio-Info-Micro, NSF Center for Bits and Atoms, and Media Lab TTT Consortium

Conventional procedures based on two-dimensional gel electrophoresis for profiling the concentrations of specific proteins and their byproducts are time-consuming, laborintensive, and require significant technical expertise to obtain quantitative information. One approach for circumventing these limitations is to develop the equivalent of a DNA microarray for identifying proteins.



Fig. 16: Concept for measuring displacement with InterDigitated (ID) fingers. When the fingers are illuminated with a laser, the reflected light produces a diffraction pattern composed of several modes. The intensities of these modes depend on the relative out-of-plane distance between the ID finger sets.

Recently, Fritz et al. (*Science 2000*) demonstrated that a microcantilever with immobilized receptor molecules can translate the binding of the target protein to a mechanical bending of the cantilever. Although the physical origin of the stress-induced bending has not yet been elucidated, an investigation by Wu et al. (*PNAS 2001*) suggests that the bending results from a combination of steric crowding of the target molecules and the free energy change between the receptor and receptor – target complex.

We have developed a differential microcantilever stress sensor that translates the binding of molecules on the sensor surface into a mechanical displacement that modulates the intensity of visible light (*Savran et al., JMEMS 2002*). The shape of the cantilevers is defined such that when they bend, the fingers of one cantilever are vertically displaced relative to the interdigitated fingers on the other. When illuminated with coherent light, the fingers form a phase-sensitive diffraction grating, and the out-of-plane displacement is determined by measuring the intensity of a diffracted mode. The concept of modulating coherent light with a diffraction grating is shown in Figure 16 and the application of this concept to a differential pair of cantilevers fabricated at the MTL is shown in Figure 17.

The differential microcantilever offers two improvements over the conventional cantilevers used in previous experiments. First, the cantilever bending is measured by interferometry which is one of the most sensitive methods for measuring position. The power spectral density of the differential deflection (See Figure 18) reveals that the resolution is limited by the cantilever's thermomechanical noise of 0.008 Å/Hz<sup> $\frac{1}{2}$ </sup> for frequencies above 40 Hz. Second, the design of the interferometer inherently measures the relative bending between two identical cantilevers. The relative, or differential, measurement is critical for eliminating unwanted signals resulting from changes in temperature, pH, ionic strength, and to some degree, nonspecific binding. Acquiring a differential measurement before the output of the detector is amplified can also improve the overall resolution by eliminating unwanted signals from background fluctuations and nonspecific binding. We are currently using the differential cantilever to detect the concentration of proteins that are difficult to detect via fluorescent labels.

# Self-Activated Microcantilever-based Gas Sensor

**Personnel** Y.K. Min (H.L. Tuller)

#### Sponsorship

NSF and Daewoo Electronics Fellowship

We are studying a Self-Activated Microcantileverbased (SAM) gas sensor providing features useful in control devices. The SAM gas sensor has a Sibased micromachined structure with gas sensing and piezoelectric components. The influence of crystallinity, microstructure and deposition conditions on gas sensor response of sputtered ZnO thin films is under investigation. The sensor response to a number of gases is being investigated and modeled. Efforts to integrate sensor and actuator functions are being pursued.



Fig. 17: Scanning electron micrograph of a differential microcantilever stress sensor.



*Fig.* 18: Power Spectrum Density (PSD) of the differential cantilever deflection.

# Gas-Liquid Flow, Separation and Mass Transfer in MicroChemical Systems

#### Personnel

A. Guenther, M. Jhunjhunwala, T. Kraus, and N. de Mas (M. A. Schmidt and K. F. Jensen)

## Sponsorship

ARO MURI and MIT Microchemical Systems Technology Center

We characterize the dynamics of gas-liquid flow in microchannels and apply such information for a systematic design of gas-liquid microreactors with a significantly increased throughput. The inherently transient nature of gas-liquid flows in microchannels is represented in slug, wavy annular, and bubbly flow regimes. The design of multichannel gas-liquid systems with desired flow and mass transfer characteristics across tens to hundreds of reaction channels requires understanding of the underlying multiphase fluid flow phenomena. We use single, silicon-based channels with hydraulic diameters between 40 and 400 µm that are fabricated by deep reactive ion etching and coated with a 0.5 µm oxide layer. Pyrex is anodically bonded to the silicon, providing optical access for flow visualization. Characterization is done using fluorescence and confocal microscopy, and integrated concentration and flow regime sensors. Superficial gas and liquid velocities were varied between 0.01-10 m/s and 0.001-10 m/s, respectively.

*Gas-liquid separation.* Using a microfabricated capillary array, we are able to conduct a complete "on-chip" gas-liquid separation, Figure 19, that can be integrated into gas-liquid microreactors. Such a methodology did not exist previously, limiting the development and usefulness of

microfluidic gas-liquid systems whenever good contacting and subsequent separation of gas and liquid streams was important or unavoidable. Our concept allows manipulation of any pattern, steady or transient, gas-liquid mixtures in microchannels, their reliable separation into individual phases at high velocities and for altering gas and liquid fractions. It allows the introduction of a gas/liquid stream into the flow channel and their contacting in any desired way and separation into individual streams, in precise amounts and at well defined locations along the flow path. With alternate methods, this is presently only possible for steady liquid-only systems or for steady and segregated gas-liquid flows.

*Mixing.* Mixing on microscale has previously been done either by focusing the streams or by patterning the channels. Both require additional features or fabrication steps. Figure 19 shows fluorescence micrographs illustrating the effect an inert gas phase has on an otherwise quiescent, laminar co-flow of two differently colored miscible liquid streams,  $L_1$  and  $L_2$ . The degree of liquid mixing is quantified using confocal microscopy where an ensemble of planar scans is obtained. We demonstrate that the transient nature of gas-liquid flows can be used to significantly improve mixing of miscible liquids compared with existing methods.



*Fig.* 19: (a) *Microfabricated capillary array in silicon. Annular (b), bubbly (c) and slug flow (d) into the separator.* 

*Flow regime and concentration sensing*. We developed a lowcost sensor (See Figure 21) for gas/liquid and liquid/liquid flows in microchannels that does not require direct optical access. It allows detection of individual slugs, bubbles, or drops and can be used to continuously determine their number and velocity. Monitoring the flow regimes in multiphase microchemical systems is important since the reactor performance is directly linked to it. The sensor can be integrated in vertically stacked and horizontally expanded multichannel systems without requiring design changes. It can be used in highly corrosive, purified or toxic media, and comprises of standard, mass-produced components. It can be encapsulated, allowing its use in industrial environments. The analysis of the signal is simple and robust.

*Mass transfer measurements.* For the model system of oxygen absorption in an oxygen-water flow, mass transfer is linked to the flow regime predictions. Sensing ports are integrated at different streamwise positions on the chip (See Figure 20). They allow a fraction of the liquid phase to be drawn out and passed over an oxygen sensitive foil. In combination with fluorescent microscopy, the local liquid concentration can be determined. A systematic investigation of gas-liquid flow combined with quantitative measurements of mass transfer rates provides a fundamental framework for design and operation of multichannel gasliquid systems.









Fig. 20: Left: Mixing of two liquid streams (L1 and L2) by introducing a passive gas stream (G) into a 400 µm channel through one side inlet of cross-section 10x40 µm at 0.67m/s. Streamwise locations (top to bottom) are: gas inlet, 1mm, 3mm downstream of the gas inlet. Right: Schematic of on-chip concentration sensing for gas absorption in an annular oxygen-water flow. The intensity of red light reflected from the sensor film is measured with fluorescence microscopy and represents the oxygen concentration in the liquid.

# Microreactors for Catalyst Testing

#### Personnel

S. K. Ajmera, C. Delattre, and C. D. Baertsch (K. F. Jensen, and M. A. Schmidt)

# Sponsorship

MIT MicroChemical Systems Technology Center

We are exploring the use of microfabricated chemical reactors for the improved testing of heterogeneous gas phase catalytic processes. In order that results from the microreactor are relevant to macroscale processes, we are designing microfabricated packed-bed reactors that utilize standard catalyst particles. Through the use of novel flow geometries and fluid distribution mechanisms directly integrated into the reactor design, we have designed a silicon cross-flow micro packed-bed reactor that approaches a gradientless reactor for quantitative kinetics determination. The cross-flow reactor achieves uniform flow distribution over a wide (25.5 mm) but shallow (400  $\mu$ m long × 500  $\mu$ m deep) catalyst bed to realize differential conversions with sufficient reaction to allow monitoring with conventional analysis techniques. A set of shallow microfabricated channels maintains a spatially uniform pressure drop irrespective of variations in catalyst packing. Experiments and finite element simulations confirm the bed is isobaric with even distribution of flow and a pressure drop ~1600x smaller than traditional micro packed-bed designs. Quantitative analysis of transport effects indicates that the microreactor length scale suppresses thermal and mass gradients in the catalyst bed. These characteristics make the cross-flow microreactor a superior tool to obtain kinetics and optimize reaction conditions.

Reaction experiments with CO oxidation and acetylene hydrogenation on supported noble metal catalysts confirm the ability of the microreactor to obtain quantitative and accurate information such as turnover frequency (activity), reaction order (mechanistic), selectivity, activation energy, and deactivation that compares well with parameters previously determined in macro-scale systems. Reactor modeling indicates that the catalyst bed operates differentially even at total conversions that would be considered large in traditional reactors adding to the utility of the cross-flow microreactor as an efficient laboratory tool. In addition, CO pulse chemisorption methods have been developed for *in-situ*  analysis of catalyst metal surface area within the microreactor, providing a fully functional chemical reactor/ analysis tool for catalyst testing (See Figure 22). High temperature/high pressure fluidic interconnects have also been developed for microreactor packaging. This complete technology will allow the operation of multiple reactors in parallel which will enable multi-reactor high-throughput quantitative catalyst testing as well as the development of mini-chemical plants for chemical production utilizing multi-step synthesis.



Fig. 22: Photograph of cross-flow microreactor for catalyst testing (Felice Frankel) and electron micrograph of catalyst channel detail.

# Scalable Multiphase Microchemical Systems for Direct Fluorination

#### Personnel

N. de Mas, A. Günther, and T. Kraus (K.F. Jensen and M.A. Schmidt)

#### Sponsorship

Novartis Foundation and MIT MicroChemical Systems Technology Center

Miniaturization and integration of chemical synthesis with chemical analysis to realize stand-alone microchemical systems could ultimately revolutionize chemical research and development by providing flexible tools to rapidly screen and optimize reactions, catalysts, and materials synthesis. In addition, small reactor volumes enhance the control of fast, exothermic reactions and allow new reaction chemistries deemed too difficult to control in conventional macroscopic reactors to be carried out safely. One example of such type of chemistry is the direct fluorination of organic molecules.

We have developed a microfabricated gas-liquid reactor using standard silicon processing and metal deposition techniques that enables efficient and safe direct fluorination reactions (See Figure 23). Gas and liquid reagents are contacted concurrently at room temperature in the microfabricated reactor and flow distribution patterns are systematically characterized by flow visualization (See Figure 24). To increase the reactor throughput we operate a large number of replicated microfluidic channels in parallel with controlled flow distribution (See Figure 24). Gas-liquid flow regime and temperature in the channels are monitored in situ. Additional separation and detection units could be integrated on chip to create platforms for automated and efficient chemical synthesis.



*Fig. 23: Cross-sectional scanning electron micrograph of the reaction microchannels.* 



Fig. 24: Gas-liquid flow in a single microchannel with a field of view (FOV) of 435  $\mu m x 1.3 mm$  (left) and multiple microchannels, FOV of 10 mm x 12 mm (right).

# **MicroChemical Systems for Separations**

**Personnel** B. Wilhite, J. Kralj, and E. Murphy (K.F. Jensen and M.A. Schmidt)

## Sponsorship

ARO MURI and MIT MicroChemical Systems Technology Center

Several unit operations are used on the macro scale to separate chemical species; examples include membrane separation, extraction, distillation, and chromatography. We have identified separation methods that take advantage of enhanced performance created by the small length scales of microfabricated chemical systems.

*Palladium Micromembranes.* High hydrogen purity is required in a variety of processes, from the microelectronics industry to PEM fuel cells. Microfabricated Palladium Membranes (See Figure 25) have been shown to enable hydrogen purification at high fluxes and high selectivity.

Work is currently underway to refine our fabrication processes to maximize device lifetime and stability. We are also investigating the use of alternate substrates for improved performance and ease of packaging.

*Liquid-Liquid Extraction.* The production of fine chemical or pharmaceutical compounds requires multistep synthesis, including the mixing, reacting, and separating of chemical compounds. Since multi-step syntheses typically involve reactive intermediates, it is desirable to run a separation immediately following reaction to reduce product degradation and the overall time required for synthesis. We have designed and fabricated a first generation extractor that utilizes electrocoalescence to separate two emulsified immiscible liquids. Electrical phenomena in general are advantageous because they are non-invasive and can readily be implemented in microfluidic devices. Current work involves characterizing the performance of the extraction system. *Chromatography.* This technique is widely used on the macroscale for highly selective separations such as those required for analytical chemistry and pharmaceutical production. A method for performing continuous chromatographic separations on the microscale would open the possibility of fabricating multistep designs requiring highly selective separations. The use of simulated moving bed chromatography as a separation tool on microscale is currently under investigation.



Fig. 25: Palladium micromembranes for hydrogen purification. Membrane device, 200 nm palladium film supported on silicon nitride and oxide membrane with 4 micron holes, image of membrane structure and integrated heater, actual device components.

# Three Dimensional Integration of Microfluidic Devices

# Personnel

T. Inoue, E. Murphy, and N. Szita (K.F. Jensen and M.A. Schmidt)

# Sponsorship

Institute for Soldier Nanotechnology and Microchemical Systems Technology Center

The objective of this project is to develop general schemes that enable flexible integration of microfluidic components. In particular, general strategies are being developed to integrate microfluidic devices made by different microfabrication methods with mixed fluidic, electrical, and optical circuit boards as well as hollow fiber platforms compatible with textile technology. Laser direct write techniques are being developed for rapid prototyping of microfluidic and packaging components. The team uses standard microfabrication techniques in silicon and glass as well as new polymer based approaches allowing for flexible, low cost interconnection schemes. Interconnection strategies based on flip chip technology concepts are being developed. UV-based direct laser writing is being pursued as a tool for rapid prototyping of microfluidic devices and packaging components. Different laser direct write approaches and laser wavelength have been surveyed. The use of UV lasers (e.g., ArF excimer laser at 193 nm) allows for laser ablation of commonly used polymers, such as polyimide, PMMA, and polycarbonate as well as glass and ceramics. The technique can be operated as a direct write procedure where the scanning beam ablates material to form microfluidic channels and vias. Alternatively, the UV beam can be used with a mask to ablate an entire section, or a subsection, of a microfluidic design. The latter approach has the advantage of producing smoother surfaces, an important issue for microfluidics, and offering higher throughput than direct write, but at the expense of having to develop a mask. We are exploring the characteristics of both writing approaches in developing the technique for fabrication and integration of microfluidic components.

# Low Leakage Micro Switch Valves for Gas Chromatography

**Personnel** J. Sihler (A. H. Slocum and J. H. Lang)

#### Sponsorship ABB

Microvalves for gas chromatography have high requirements in terms of low leakage, reliability, and chemical inertness. We are designing and building a symmetrical 3-way micro gas valve with an electrostatic actuator that has been chosen for low energy consumption. Figure 26 shows a cross section of the valve. If the plate is electrostatically pulled downwards, the fluid will flow through outlet A; if the plate is pulled up, the fluid will escape through outlet B. The fundamental sealing mechanism is a flat plate on a flat surface, where the width of the valve seat is orders of magnitude larger than the mean free path of the gas. Smooth surfaces are in general easily available (i.e. the surface of a regular silicon wafer is smooth to within 10 Angstroms). Utilizing these surfaces in a microvalve design has the potential of economically creating very well-fitting valve surfaces capable of sealing with a very low leakage. If the valves are to be arranged in an array, the very center silicon layer as well as the back side of the base wafers can be used to create the fluid and electrical interconnections. In this case, the valve chip is packaged with an anodically bonded Pyrex wafer on either side.

Thus far, one-sided prototype valves have been built and are currently under test. Figure 27 shows the test results of the electrostatic actuator. The displacement of the valve plate from its initial relaxed position in the center is plotted with respect to the applied voltage. Pull-in has been observed at 41 Volts where the valve plate displaces by its full stroke of 10 micrometers. Upcoming tests will examine the sealing capability of the closed valves using a He-leakage tester as well as the flow rate dependence on the pressure drop across the open valve.

# Fuel Processing in Microfabricated Chemical Systems

L.R. Arana, C.D. Baertsch, B. Blackwell, A. Mitsos

(K. F. Jensen, and M.A. Schmidt)

Personnel

Sponsorship

ARO MURI and DARPA



Fig. 26: Switch valve cross section





Chemical fuels store substantially more energy per unit weight and volume than the most advanced batteries currently available. For this reason, portable fuel cell systems operating on chemical fuels have received a great deal of attention in hopes of outperforming batteries in low-power, portable electronics. Given the current state of the art in fuel cell technology, a feed of pure hydrogen is desirable to reach optimal fuel cell efficiencies. However, hydrogen storage presents a challenge due to safety concerns and its low compressibility. Point-of-use hydrogen generation from a conveniently stored liquid fuel (e.g., butane) is a desirable albeit challenging solution to this problem. Our research program investigates the potential of MEMS (Micro-Electro-Mechanical Systems) microfabrication technology to make safe, portable, efficient chemical fuel processors for point-of-use hydrogen generation.

We have developed a fuel processor (See Figure 28) comprised of an integrated high-temperature chemical reactor and heat exchanger. Thin-walled silicon nitride tubes enable a high degree of thermal isolation of the high-temperature reaction zone, and integrated silicon slabs enable heat exchange between process streams. Electrical and combustion heating of the reactor to temperatures over 900°C have demonstrated its high degree of thermal isolation and mechanical stability. In addition, net hydrogen production has been demonstrated through autothermal (no electrical power input) ammonia and butane combustion coupled with ammonia decomposition. Our ongoing research effort can be divided into four major thrusts: fuel processor design and modeling, fabrication and packaging technology, heterogeneous catalysis in microchannels, and reactor testing/overall system assessment.

*Fuel processor design and modeling*. The optimization of the reactor design is a valuable and challenging aspect of the research effort. One major consideration in reactor design is thermal management, which in this

continued

application means being able to integrate exothermic combustion and endothermic hydrogen-producing reactions such that they exchange heat with each other but lose little heat to the environment. Detailed modeling of fluid dynamics, i.e. heat and mass transfer, and chemical reactions is an important component of the reactor design effort.

*Fabrication and packaging technology.* The improvement of fabrication and packaging technology is another key aspect of the research program. While a working fabrication process has been developed, improvements to and optimization of the process are still actively being investigated. This includes, but is not limited to, the development of a process for vacuum packaging of the fuel processor.

*Heterogeneous catalysis in microchannels.* Heterogeneous catalysis, specifically as it applies to reactions in the micro fuel processor, is an essential component of the research effort. Our catalysis research focuses on (1) investigation of methods for the controlled deposition of high-surface-area porous catalyst films (washcoats) into the microchannel structures, (2) development of accurate techniques to characterize these catalyst washcoats in-situ (for measurement of active surface area, adhesion to substrate, etc.) before and after carrying out high-temperature chemical reactions, (3) examination of catalyst performance (rate, selectivity, deactivation, etc.) for high temperature fuel reforming and H<sub>2</sub> production reactions, and (4) implications of catalysis (catalyst selection, reaction scheme, etc.) on the optimization of the fuel processor system.

*Reactor testing and overall system assessment.* Testing of the fuel processor in a variety of hydrogen production schemes is part of the effort to properly assess its potential in portable fuel cell systems. Fuels include butane, methanol, and ammonia, and reaction pathways include partial oxidation, and combustion coupled with thermal decomposition or steam reforming. The need for separations systems and auxiliary equipment, such as pumps, valves, sensors, control systems and plumbing is also being evaluated.



Fig. 28: Micro fuel processor. Photograph of device (top) with close-up of suspended tube/slab structure. Electron micrograph of reactor/heat exchanger section (lower left). Glowing reactor during autothermal combustion and ammonia cracking (Note, that the tubes and surroundings remain at low temperatures).

# Microchemical Systems for Fuel Processing and Conversion to Electrical Power

## Personnel

L. Arana, C. Baertch, J. Cui, J. Hertz, A. Ie, C. Lee, A. Mitsos, O. Nielsen, T. Stefanik, K. Turner, and S. Weiss (K.F. Jensen, P. Barton, S.M. Spearing, M.A. Schmidt, H. Tuller, and J.Y. Ying)

# Sponsorship

ARO MURI

This program aims to develop a fundamental understanding of the different physical phenomena underlying fuel processing at millimeter to micron scale as well as to establish the engineering principles needed to realize portable electrical power generation from hydrocarbon fuels.

Portable, high density power sources have been identified as an enabling technology. A continued reliance on batteries, combined with their relatively low projected energy densities, create serious logistical mission constraints. Taking advantage of the high energy density of chemical fuels to generate power becomes an attractive technological alternative to batteries. However, development of fuel processors capable of chemical/electrical conversion with a net power output on a portable scale represents a significant technological challenge.

Microreactors as miniature fuel processors represent an emerging technology that could significantly impact our ability to produce high density power sources in the future. The program aims to develop a fundamental understanding of the many different physical phenomena underlying fuel processing at millimeter to micron scale as well as to establish the engineering principles needed to realize portable electrical power generation from hydrocarbon fuels based upon advances in microfabrication, fuel cells, catalysis, materials characterization, and systems engineering. Competing approaches to fuel conversions are addressed with particular emphasis on two basic strategies:

- Conversion of hydrocarbons to hydrogen for use in a hydrogen fuel cell by partial oxidation, reforming, and product separation steps.
- Direct conversion of hydrocarbon fuels in a microfabricated solid oxide fuel (SOFC) cell system integrated with microfluidic controls.

In order to address the many different aspects of microfabricated fuel processing systems, we have a multidisciplinary research team that combines the necessary expertise, specifically in the areas of thermomechanical properties of materials, materials synthesis, microfabrication, chemical reaction engineering, heat transfer, catalysis, simulations, and systems engineering. This multidisciplinary research program has set the following goals for the proposed effort:

- Development of design and microfabrication strategies for microchemical systems capable of operating at elevated temperatures and being rapidly cycled between low and high temperatures. This will entail the development of novel fabrication strategies involving high temperature materials such as oxides not currently used in standard, mainly silicon-based, microfabrication methods.
- Synthesis and characterization of novel catalytic materials for low temperature partial oxidation and reforming as well as for novel SOFC electrodes. In particular, tailored heterogeneous catalytic surfaces for microchemical systems will be generated by forming nanostructured features on surfaces of micron-scale flow channels.
- Fundamental understanding of and engineering approaches to integration of materials with different thermophysical properties into systems undergoing large spatial and temporal temperature variations.
- Fundamental understanding of transport and reaction processes in microchemical systems. Development of engineering design principles and simulation tools for microreactor systems.

# **Piezoelectric Micro Power Generator** (PMPG): A Novel MEMS-Based Electrical Power Source

**Personnel** R. Sood and Y.B. Jeon (S.G. Kim and S. Sarma)

# Sponsorship

Auto-ID Center

#### Systems engineering concepts and tools for understanding and predicting the performance of integrated microchemical systems at steady state and during transients, i.e., dynamic behavior.



Fig. 29: Different elements of the fuel processing program; fuel processor, microcombustor, solid oxide fuel cell schematic, crystal structure of catalysts, energy integration diagram.

#### Concept and Key Idea

A MEMS-based energy harvesting device is designed and fabricated that converts ambient, acoustical energy to electrical energy via the piezoelectric effect. The electrical energy is subsequently stored within the device to act as a constant, electrical power source in replacement of a common battery or wired supply.

#### **Design and Fabrication**

The PMPG (Piezoelectric Micro Power Generator) is a device currently in the microfabrication stage. It converts ambient acoustical and/or vibrational energies to electrical energy via the piezoelectric effect. Unlike conventional batteries, the PMPG will provide power for an infinite duration of time, provided there is ambient energy available. It consists of a composite cantilever beam with top interdigitated electrodes and an added proof mass.

The ambient energy is coupled into the first resonance mode of the mechanical structure. When the device is in resonance, the generated electrical signal will be sinusoidal in nature. The signal must, therefore, be rectified before the resulting charge can be stored. The PMPG is designed to deliver a high open circuit voltage and electrical power specification due to its exploitation of the  $d_{33}$  excitation mode of the piezoelectric material (PZT). The  $d_{33}$  mode has been calculated to provide up to 20 times the open circuit voltage value of a  $d_{31}$  device with the same beam dimensions. The converted electrical energy is then stored, effectively creating a portable, electrical power source capable of driving low power digital circuits among other applications. These devices are, therefore, suitable for powering small wireless sensors or Auto-ID tags. The PMPG would replace chemical batteries in these low power applications and would, in fact, be better because the PMPG will have a virtually infinite operation lifetime.

Figure 30 shows a picture of the ideal, released PMPG. The device employs a surface micromachining process using XeF<sub>2</sub> vapor etcher in order to create the released micro-cantilever structure. The composite beam includes a  $SiO_2/ZrO_2$  membrane layer that acts as an electrical diffusion barrier, a PZT piezoelectric layer and a Pt interdigitated electrode layer. Figure 31 is an electrical equivalent circuit model of the PMPG, along with the rectifying circuitry necessary before charge storage. Figure 32 contains two polarization curves of a sample PMPG device. After poling, the electric dipoles within the PZT are aligned, allowing for device operation. A small array of these devices is expected to permit a power density of approximately  $10\mu W/mm^2$ . The final PMPG is released from the bulk silicon by way of a XeF<sub>2</sub> isotropic etch step. Acoustic operation frequency is set between 20 kHz and 40 kHz, outside of the audible range. Figure 33 shows an overhead view of an actual, pre-released device.



Fig. 31: Electrical equivalent circuit model of PMPG with full-wave rectifier



*Fig.* 32: *PE hysteresis curves of the PMPG device before and after poling* 



Fig. 33: Top View of interdigitated PMPG fabricated



Fig. 30: Surface micromachined PMPG device

# **Development of Hydrocarbon-Fueled Silicon Combustors for Micro Gas Turbine Engines**

**Personnel** C. M. Spadaccini, J. Peck, N. Miki, and L. Ho (I.A. Waitz)

# Sponsorship

ARL, ARO, and DARPA

Recent advances in the field of silicon micro-fabrication techniques and silicon-based MicroElectroMechanical Systems (MEMS) have led to the possibility of a new generation of micro heat engines for power generation and micro air-vehicle propulsion applications. The design for a silicon-based, micro gas turbine generator capable of producing 10-50 Watts of power in a volume less than 1 cm<sup>3</sup> while consuming 7 grams of fuel per hour has been developed.

An engine of this type will require a high temperature combustion system to convert chemical energy into kinetic and thermal energy. To accomplish this, a unique set of challenges must be overcome:

- 1. Shorter residence time for mixing and combustion.
- 2. Heat loss due to high surface area-to-volume ratio.
- 3. Material and structural constraints of silicon.
- 4. Rudimentary 3-D geometry due to limits of micro-fabrication techniques.
- 5. Micro-engine thermodynamic cycle constraints.

All of the above impact the design and development of a suitable micro-combustion system.

The baseline micro-combustor device is comprised of all the non-rotating functional components of the micro gas turbine engine. The device measures 2.1 x 2.1 x 0.38 cm and is aligned-fusion bonded from 6 silicon wafers. Figures 34 and 35 show a schematic and a SEM of this micro-combustion system. Fabricated largely through Deep Reactive Ion Etching (DRIE), the structure required anisotropic dry etching through a total thickness of 3,800 µm. Complete with a set of fuel plenums, fuel injector holes, pressure ports, and compressor and turbine static airfoils, the design of the six-wafer structure required a multi-disciplinary approach that accounted for all the chemical, structural, and fluidic interactions as well as engine system considerations.

For the propulsion and power generation applications of interest, the principal figure of merit is power density. The baseline device achieved power densities in excess of 1100 MW/m<sup>3</sup> with hydrogen-air combustion. This corresponds to exit gas temperatures over 1700 K and combustor efficiencies greater than 95%. In an attempt to achieve higher power densities, a staged combustor has also been developed. With this device, a 100% increase in power density was achieved for some operating conditions. These power densities are about two times larger than those produced by a conventional gas turbine combustor and are an order of magnitude larger than other power MEMS devices. Hydrocarbon fuels, such as ethylene and propane, have been burned in these devices at lower power densities,  $500 \text{ MW}/\text{m}^3$  and  $150 \text{ MW}/\text{m}^3$ respectively. Data from both the staged and baseline combustors has been used to develop a non-dimensional operating space which can be used as a design tool for gas phase combustors.

A combustor which utilizes heterogeneous catalysis to improve hydrocarbon-air reaction rates has been identified as a means of increasing power density for hydrocarbon fueled micro-combustors. A six-wafer catalytic microcombustor similar to that shown in Figure 34 was fabricated. During the fusion bonding process, the combustion chamber was fitted with a piece of platinum coated foam to serve as the active catalytic surface. With propane fuel, this device operated at significantly higher mass flow rates than its gas-phase counterpart and achieved an ~8fold increase in power density. However, this combustor did not achieve high exit gas temperature and overall efficiency. As a result, low order models have been used to identify the device as diffusion controlled, and design recommendations for a second-generation catalytic microcombustor have been made.

continued



Fig. 34: Schematic of six-wafer combustion system.



Fig. 35: SEM cross-section of six-wafer combustion system.

Personnel

O.M. Nielsen and C.D. Baertsch (K.F. Jensen and M.A. Schmidt)

#### Sponsorship DARPA

Based on a suspended-tube micro chemical reactor developed at MIT, this project aims at converting heat released from the reactor by radiation into electrical power by the use of low-bandgap photovoltaic cells. The devices are ultimately likely to be operated at around 1000°C, at which temperature the radiation is mostly in the infrared regime, calling for low-bandgap converter materials such as GaSb or GaInAsSb. The cells are obtained commercially or from other research programs, while the emitter structure, photon recycling scheme, thermal management, and packaging (including vacuum) are developed within the project.

The ThermoPhotoVoltaic (TPV) micro-generator has been successfully operated with a net 1 mW (32 mW/cm<sup>2</sup>) electrical power output, at an efficiency of 0.08%. The emitter temperature was approximately 770°C during operation, and one GaSb photocell was used for energy conversion. According to conservative estimates for a TPV micro-generator with some basic improvements (two photocells, vacuum packaging, and 1000°C emitter temperature), 16 mW (250 mW/cm<sup>2</sup>) net power and 2.4% efficiency should be obtainable with the current structure. The advantage of increasing the emitter temperature cannot be over emphasized, as is clear from the test results shown in Figure 36.



*Fig. 36: Photocell power output and system efficiency, as a function of temperature, during electrical heating of the*  $S\mu RE$ .

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# **Micro-fuel Cell**

#### Personnel

J. Hertz and T. Stefanik (H.L. Tuller)

## Sponsorship

DoD Multidisciplinary University Research Initiative (MURI) administered by the Army Research Office.

The feasibility of integrating a solid oxide fuel cell structure into a micromachined silicon structure is being investigated as part of a larger multidisciplinary group. The performance of thin film electrolytes and electrodes is being studied as a function of composition, microstructure, and device structure. Processing procedures are being developed which enable the functional solid state ionic films to be incorporated into a micromachined silicon structure.

Among the photons emitted, only those that have energies above the photovoltaic cell bandgap can be converted into electricity. This combined problem of emitter characteristics and photon recycling will be approached in different ways. Since radiation is a surface property, the reactor (emitter) surface can be tailored to yield more desirable radiation spectra. Many different material and structural systems are possible candidates (e.g. SiC, photonic crystals), but fabrication complexity must be weighed heavily in making choices. Photon recycling schemes include filters and back-side reflectors on the photovoltaic cells. Most likely, a combination of these different approaches will be pursued.

A new, optimized reactor structure for TPV purposes is currently under fabrication. Packaging of the entire structure, including the photovoltaic cells, will be investigated. Vacuum packaging will be essential, and some work has already been done. The ultimate goal is to demonstrate a device that can convert chemical energy to electricity with an overall efficiency in the 10-15% range. Power density goals are on the order of 1 W/cm<sup>2</sup>. The work is targeted at developing a replacement technology for batteries in certain applications based on higher energy conversion efficiencies and energy densities.

# Lateral RF MEMS Switch with PZT Actuation

**Personnel** Y. Shi and T. M. Lee (S.-G. Kim)

# Sponsorship

KĪMM

#### **Concepts and Key Idea**

A new RF MEMS switch with lateral contacts is being designed and developed for wireless mobile communication applications. The objective of this project is to develop a RF switch with much improved robustness, compactness, reliability, and low cost comparing to the existing RF switches. The major advantages of the piezoelectric lateral contact RF switch will be very low driving voltage (less than 5 Volt), very low contact resistance (less than 0.1  $\Omega$ ), and very high reliability (more than 100 billion cycles), which are essential for real applications without scarifying the benefits of MEMS switches, such as low insertion loss, near zero power consumption, and very high isolation, etc. The device is enabled by strain-amplified PZT actuators which provide the mechanical movement creating the open or short circuit in the RF transmission line.

#### **Design and Fabrication**

To achieve the major objectives of very low contact resistance and very high reliability, maintaining a large and real electric contact area is critical, which requires the elastic-plastic deformation of the metalmetal contact layers. PZT actuator is chosen for the large force and displacement it can provide. Lateral contact switch configuration is adopted to take full advantages of the PZT actuators. The switch function of the device is achieved through the lateral contact of two switching components. The mechanical movement and force required for the contact is realized through large-strain thin film piezoelectric actuators which use PZT ( $Pb(Zr,Ti)O_3$  as the active material. The displacement from PZT is amplified by more than 10 times by the amplification mechanism for each individual actuator. Actuators are also combined in parallel or series to provide larger displacement or bigger force requirements. Lateral displacement of 10  $\mu$ m can be achieved with less than 5 volts actuation in a footprint of less than 500  $\mu$ m x 500  $\mu$ m. Larger

contact area (> 10  $\mu$ m<sup>2</sup>) and larger gap (about 5  $\mu$ ) are maintained between the two switching components which provide low insertion loss, but high isolation and high power handling capacity. Su-8 has been chosen as the switch structure material for its unique property of making near vertical sidewall structures and also its good mechanical properties. Mathematical modeling for the contact behavior and FEM simulation of the device design is being done to optimize the overall performance of the switch. A schematic view of the device with e-beam Au layer on the side and folded Au layer are shown in Figure 37 and Figure 38 respectively.

PZT is deposited using Sol-gel method, and several approaches have been investigated to fabricate the displacement amplification mechanism of the actuator. Spin-on Su-8 has been chosen as the structure materials for its unique properties and compatibility with the actuator. The most critical part of the switch fabrication is the deposition of contact metal on the contact areas on the sidewall of the Su-8 structure. We have chosen Gold layer for our initial tests. The electrical contacts are crucial for the performance of the switches. Two methods have been investigated to create the metal contact layers. The first one is to fold a thick metal film already deposit on the bottom surface of the structure into the gap between the two contact components, while the second is to deposit the metal film on the contact surface using e-beam evaporation or sputtering process. The Gold layer on the side wall of a Su-8 structure with negative slope using e-beam evaporator is shown in Fig. 39. An initial test shows the resistance across the side wall is less than 0.4  $\Omega$ , which can be improved further by having a relatively vertical or positive side wall slope. The sidewall slope due to fabrication will have significant influence not only on the metal layer quality, but also on the ohmic contact. Efforts have been made to ensure high quality metal-metal contacts required. Fabrication of the device is still ongoing.

# **MEMS Tunable Capacitor and LC Tank**

#### Personnel

X. Yang (A. H. Slocum, J. H. Lang, and M. A. Schmidt)

## Sponsorship

Deshpande Center

Currently there is a great demand, especially in the wireless communication area, for frequency agile devices that are capable of frequency tuning and selection in the ultra-high and super-high frequency. Furthermore, integration of such devices with the transceiver circuit is highly desirable in order to reduce total costs and power consumption. The goal of this research project is to use MEMS technology to develop a high Q, highly tunable LC oscillator that is suitable for frequency agile tuning from 2 to 5 GHz or higher.

The design of the capacitor originates from the ideas of "Nanogate" and "Zipper Actuator", two devices being developed in the group, and uses the fact that two smooth, perfectly matched surfaces can be pried apart with an external actuator, and the gap between the surfaces can be accurately controlled. An LC tank is devised by incorporating a cavity inductor with the capacitor design.

A 2-D cross section of the tunable capacitor is shown in the Figure 40. It comprises three wafers anodically bonded together: the top and the bottom wafers are Pyrex wafers while the device wafer is a SOI (Silicon-On-Insulator) wafer. The device wafer contains a membrane that is supported on tethers from the sidewall and the fulcrum (with a ring shape) that's anchored to the top Pyrex wafer. The fulcrum is the pivot of rotation for the membrane. The outer rim of the membrane forms the zipper actuator that pins down to the bottom Pyrex wafer when a voltage is applied. Using the fact that gold does not bond to oxide, we can obtain a perfect match of the dielectric of the capacitor with its bottom electrode upon bonding. The capacitance at this position corresponds to the maximum. When voltage is applied to the zipper actuator, the center of the membrane bows up and hence changes the gap of the capacitor that's formed between the membrane and the bottom aluminum electrode. The change in the gap results in the change in the capacitance, and hence, a tunable capacitor is formed.

continued

# Au Contacts PZT Actuator

Fig. 37: A schematic view of the switch.







Fig. 39: SEM picture of side wall Au



There are many challenges in the project in terms of fabrication, RF design, packaging, and integration. The fundamental contributions expected include: to demonstrate a high tuning ratio capacitor that has not been reported before; to create innovative fabrication solutions that could benefit other users; to develop a frequency agile MEMS LC tank; and to explore the potential of RF MEMS in fulfilling RF communication needs.

Fig. 40: 2-D schematic of the design of a tunable capacitor.

Currently, fabrication of the tunable capacitor is underway. A major challenge was to form the cylindrical fulcrums with walls less than 50  $\mu$ m thick and 300  $\mu$ m tall. This has been done successfully using STS. Figure 41 is the SEM photo of the cross-sectional view of the fulcrum.



Fig. 41: SEM photo showing the cross-sectional view of fulcrum.

# DRIE-Fabricated Curved-Electrode Zipping Actuators with Low Pull-In Voltage

## Personnel

J. Li (J.H. Lang and A.H. Slocum in collaboration with M.P. Brenner)

# Sponsorship

n/a

Electrostatic curved-electrode zipping actuators have been developed to generate high force and large displacement simultaneously. Laterally-moving actuators fabricated with DRIE could provide greater stroke and force without a significant increase in actuator size. However, DRIE constraints limit the minimum electrodecantilever gap during a deep etch, and hence, result in actuators with larger starting voltages. The goal of this project has been to build a monolithic low pull-in electrostatic curved electrode actuator fabricated by Deep Reactive Ion Etching (DRIE).

We have designed and fabricated the actuators. Figure 43 shows two such curved-electrode zipping actuators with compliant cantilevers utilized to actuate a bistable MEMS relay. Figure 42a shows the relay beam (11) ready to be actuated by the actuators (3&7) through a T-bar (6). As shown in Figure 42b, the starting cantilevers (2&9) are attracted and pulled in to the corresponding actuator cantilevers when electrified. The gap is closed, and a very high electric field and force is created. The actuator beams then zip along the fixed electrodes (1&10) at relatively low voltage to actuate the bistable relay beam as shown in Figure 42c. Finally, an electrode (5) with its two compliant starting cantilevers (4&8) is used to pull the relay beam back to the initial position as shown in Figure 1d.

The device is fabricated using DRIE. After etching, 0.2micron of oxide is grown on both the surface and sidewalls. A shadow wafer is then used to etch the oxide off the top surface of the electrodes to achieve good electrical contact. The device wafer is bonded with a Pyrex handle wafer, and the electrodes are isolated with snap off tabs as a last step. Contact metalization makes low resistance relay contacts.

Figure 43 shows an optical micrograph of actuators with starting cantilevers fabricated into a bistable relay. Figure 43a shows the relay as fabricated. In Figure 43b the starting cantilever bends up at low voltage to close the gap and pull in. In Figure 43c the actuator cantilever zips completely to toggle the bistable relay beam as shown in Figure 43d. The actuators each measure 4.5 mm \* 100 µm \* 300 µm, excluding the contact electrodes and the relay beam. The pull-in voltages of the actuators were measured to be between 75 and 85 V. After pull-in, the actuator cantilever zipped along the electrode with increasing voltage, pushing the relay to its opposite bistable position. The voltage required to toggle the relay beam was measured to be between 100 and 140 V. During this actuation, the two actuator cantilevers developed a combined 8-mN force while translating 80 µm before toggling the bistable relay beam. Actuators and relay beams were operated using a bipolar drive at 100 Hz for over 120 hours through more than 40 million cycles without stiction or fracture. The devices have been switched with voltage pulses as short as 400 µs. The time taken for the actuator to close the relay was measured to be 3 ms.



Fig. 42



# MEMS Amplification of Piezoelectric Strain for In-Plane Actuation

**Personnel** N.J. Conway (S.-G. Kim)

#### Sponsorship KIMM

KIMM

## **Concepts and Key Idea**

Though MEMS piezoelectric actuators can provide very high force output at low driving voltages, they have a very small strain, typically only a couple of tenths of one percent, leading to very limited applications in real world. A new method of amplifying the small strain of in-plane piezoelectric actuators has been developed. Very compact in-plane piezoelectric actuators with a few percent of strain will enable very compact MEMS devices replacing the existing bulky electrostatic actuators. The entire device is designed and fabricated using batch micro-fabrication techniques, averting the need for costly micro-assembly of the actuator with a piezoelectric element (See Figure 44).

#### **Design and Fabrication**

The strain amplification is achieved through the fabrication of a compliant mechanism. A piezoelectric membrane, made of PZT, is situated in the middle of four parallel guiding linkages comprising the actuation mechanism. The pivot points are small length flexural pivots or living hinges designed to approximate an ideal pivot, but without any backlash associated with a real pivot. Because the angles the pivots subtend are small, the pivots behave close to ideal. By amplifying the membrane displacement, the compliant structure sacrifices some piezoelectric force, however, the PZT membrane provides sufficient force that the output force is still desirable (order  $100 \mu N$ ). Each actuator has a form factor of 500 µm X 500µm X 30µm, and the expected output displacement is 6 µm. The actuator can easily be arrayed in n-parallel to gain an n-times force advantage or in series to gain an n-times displacement advantage, with the drawback of increased form factor.

The compliant mechanism is made of SU-8, which allows the entire device to be surface micro-machined. The PZT membrane is fabricated first, followed by the SU-8, followed by a release. A process for single-crystal silicon-based compliant mechanism has also been devised. Batch-fabricated in-plane micro-piezoelectric actuators have not been done before to our knowledge. As this is an ongoing project, no experimental results of a final device are available.



Fig. 44: A Bow Actuator Design for Strain Amplification

# Water-Immersible Micromachined Pb(ZrTi)O<sub>3</sub> Thin Film Actuator

**Personnel** Y.B. Jeon and C.W. Wong (S.-G. Kim)

# Sponsorship

Department of Mechanical Engineering

#### Concept and Key Idea

There have been many efforts to overcome two major problems in SPM technologies: slow scanning speed and inability to measure living cells underwater. We demonstrate the water-immersible thin film PZT actuator; the whole PZT is sandwiched between the top and bottom metal electrodes to prevent water permeation and can be used for *in-vivo* or *in-vitro* SPM measurements of living cells under water or biological fluid. This can also be used for the direct actuation of microfluidic devices in various wet applications.

#### **Design and Fabrication**

In order to be water-immersible, the electrodes need to be electrically insulated and the piezoelectric layer needs to be waterproofed. This paper describes our design solution and process for a water-immersible piezoelectric device, which separates the bottom electrode from the top electrode by having a narrow ditch covered with PZT film. The whole PZT film is then encapsulated with the top metal electrode, which seals envelopes together with the metal layer left outside ditch of the lower electrode. In this structure, the whole PZT is sandwiched between the top and bottom metal electrodes to prevent water permeation. Figure 45 shows the water-immersible PZT cantilever beam designed.

The width of the ditch is the key design parameter for both electrical separation and good microstructure of PZT on top of the ditch. We tested several widths of the ditch and got good dense PZT microstructure and electrical insulation with the 2  $\mu$ m width of ditch. The surfaces of the PZT film on the 2  $\mu$ m separated Pt bottom electrode region ("P" region in Figure 45) were observed at three adjacent points by AFM (Figure 46). The piezoelectric constant, d<sub>31</sub> is about -100pC/N. The dielectric polarization and fatigue properties of the devices were measured in air and water. The spontaneous polarization, remnant polarization, coercive field, and dielectric constant are 54  $\mu$ C/cm<sup>2</sup>, 15  $\mu$ C/cm<sup>2</sup>, 60 KV/cm and 1200, respectively (Figure 47(a)) and the polarization.

ization property of the device was unchanged whether it was in the air or under the water until  $1 \times 10^9$  cycles (Figure 47(b)).



*Fig. 45: Device schematic of waterimmersible piezoelectric actuator.* 



Fig. 46: AFM images of the PZT film surface and intersection diagram pf the PZT film on the separated Pt bottom electrode region; (a) PZT surface exact on the separated Pt; (b) PZT surface near on the separated Pt; (c) PZT surface on Pt bottom electrode; (d) Schematic diagram of the regin "P" in Figure 1(a) which was observed by AFM.



*Fig.* 47: Polarization property changes of the PZT films. (a) P-E hysteresis curve deposition; (b) Fatigue properties against number of cycles in air or water.

# Wafer Bond Alignment and Strength Characterization

#### Personnel

C. Tsau and H. Verma (C.V. Thompson, M.A. Schmidt and S.M. Spearing)

#### **Sponsorship** SRC

We are exploring the property-process relationship of wafer-level thermocompression bonding process. This bonding technique relies on the simultaneous application of pressure and temperature to bring mating surfaces to close proximity and bond. In our study, we bond two silicon wafers together with thin, patterned gold. Gold is the preferred bonding material because it does not oxidize easily, which simplifies the surface treatment prior to bonding. The bond is performed between 260 and 300°C, permitting the bonding of MEMS and/or IC wafers together after the devices have been fabricated. In addition, because gold is conductive, parts of the bonding layer may also be used for local signal routing. Lastly, hermetic seals may be achieved, which is important in some inertial sensor applications.

We have established a wafer-level bonding protocol for 4" wafers using a commercial bond tool (Electronic Visions) and a mechanical testing technique for quantifying the bond toughness. The four-point benddelamination technique measures the critical load at which crack propagates at the bonding interface. The specimen geometry and the critical load then allow the critical strain energy release rate, or the input energy at which delamination is required, to be calculated. We have performed bonding at varying temperatures (260 and 300°C) and pressures on the gold (7 to 120 MPa) (See Figure 48). Our current effort is directed towards understanding the source of non-uniformity and variation in the bond toughness.



Fig. 48: Bond toughness data for specimens bonded at 300°C and 73 MPa (on gold).

# Microelectromechanical (MEMS) Thin Film Stress Sensors

**Personnel** R. Bernstein, A.L. Giermann, S.C. Seel, and D. Moore (C.V. Thompson)

# Sponsorship

CMI, NSF, and SMA

We have designed and fabricated several microelectromechanical devices for measurements of film stress during deposition and post-deposition processing.

The operation of one class of devices is based on buckling of membranes and beams. Single-crystal Si membranes can be made through patterned anisotropic etching of bonded silicon-on-insulator wafers (See Figure 49a). Films can then be deposited on these membranes, and if the films are under a state of sufficiently high compressive stress, they will cause the membranes to buckle. The buckling state can be characterized using a variety of tools, but optical profilometry provides a particularly straightforward technique for characterization of the buckled membrane shape, with sufficiently high vertical spatial resolution to allow determination of film stresses through comparison with models for buckling. If the compressive insulating oxide film is left as part of the membrane, membranes within a size and thickness range will be buckled in the as-fabricated state (See Figure 49b). This state can be characterized before and after film deposition to measure the changes caused by the deposited film, thereby allowing characterization of the stress in the film. This technique allows determination of the stress states of both tensile and compressive films and can also be used when films are deposited on both sides of the membranes (e.g., by chemical vapor deposition). Buckling of composite, doubly-supported beams can also be used for film stress measurements.

The second class of devices under investigation is based on micromachined cantilevers, made of single crystal silicon alone, silicon nitride alone, or as part of a composite beam structure. The deflection of these cantilevers can also be characterized with very high vertical spatial resolution using an optical profilometer. The 'sample' cantilever can be 'actuated' by another cantilever, for example a rectangular AFM cantilever, as shown in Figure 50. Both cantilevers can be imaged concurrently. This second cantilever, also known as the 'sense' cantilever, is made of single crys-

tal silicon, so the mechanical properties are well known. Because of this, the force applied to the sample cantilever can be calculated and from this, the modulus of the sample cantilever can also be calculated. We are also using these simple devices for the study of thin film plasticity which is typically investigated by application of strains due to differential thermal expansion between a film and its substrate during heating and cooling. However, these cantilevers can be isothermally deflected through application of a known force at their tip using the sense cantilever, causing a known strain in films deposited on the sample cantilevers. The stress state resulting from deformation can be characterized using the optical profilometer measurements of the beam shape during and after release of the applied force. Studies of time-dependent changes in beam shapes also allow the study of time-dependent inelastic phenomena. Because micromachined cantilevers can be made very thin, Transmission Electron Microscopy (TEM) can be used to study the effects of inelastic deformation at the nano-scale. Continuous films will be studied, as will small structures (for example, sub-micron square dots) whose small dimensions limit dislocation formation and motion.

A third device has been developed for *in-situ* studies of stress evolution during film formation and during postdeposition processing. This device is made using a (110) Si wafer that is bonded to an oxidized (100) wafer and thinned to a thickness of 20µm. A cantilever beam with 4 resistors wired in a Wheatstone bridge structure is then fabricated in the (110) Si layer (See Figure 51). Three resistors are oriented so as to have no piezoresistance, and one is oriented so as to have high piezoresistance. When films are deposited on these piezocantilevers, forces exerted in the silicon cause stresses that can be characterized through measurement of the piezoresistance. In-situ stress measurements require only electrical feed-throughs in the UHV deposition system and can be made with a sensitivity of less than 1MPa-µm, even with these relatively thick beams. These piezocantilevers can be readily heated and cooled and can be used for measurement of stresses caused by films deposited via chemical vapor deposition (in which deposition occurs on both sides of the cantilever). These devices have been used for *in-situ* measurements of stress during formation and growth of polycrystalline films. We are currently investigating alternative, and simpler, fabrication processes.



*Fig. 50: Micromachined 'sample' cantilever being actuated by AFM 'sense' cantilever* 



*Fig. 51: (a) Top view and (b) Perspective view of a micromachined piezocantilever for* in-situ *stress measurements during film deposition* 



*Fig.* 49: (a) Micromachined single crystal Si membrane. (b) Pre-buckled composite Si-SiO<sub>2</sub> membrane

# **3D Nanomanufacturing via Folding of 2D Membranes**

## **Personnel** S. Jurga and C. Hidrovo-Chavez (H. I. Smith, and G. Barbastathis)

# Sponsorship

NSF and CMSE

Functional Three-Dimensional (3D) Nanostructures are necessary in numerous technological domains. The 3<sup>rd</sup> dimension promises to extend the pace of ever faster processors and higher-capacity memories beyond "the end of Moore's law," *i.e.*, when feature sizes of planar electronics reach their minimum practical limit. Commercial research in that direction is already in progress. In applications other than electronics, the need to conquer the 3<sup>rd</sup> dimension is even more urgent. Examples are: optical elements integrating sensing and processing for defense or commercial applications, miniature reactors for chemical and biochemical analysis in homeland security, drug delivery by miniaturized microfluidic implants, micromechanical and nanomechanical energy storage elements, environmental monitoring and industrial quality control applications, etc. However, 3D fabrication is not nearly as well understood and developed in the state of the art. Our CMSE seed grant research aims to take the 3D challenge with a specific method for 3D fabrication and assembly, which we refer to as "membrane folding." For a three dimensional technology to be successful and widely applicable to the worlds of solid-state electronics, MEMS, and nanomanufacturing, it must satisfy the functional requirement of sufficient connectivity between the micro and nano devices that compose the system (i.e. the transistors and capacitors of a microchip). Additionally, a winning technology must avoid fighting against the momentum of an existing industry with established tool-sets and large capital investments; a new 3D technology must be easily integrated and compatible with current methods of fabrication that remain planar in nature (i.e. photolithography, plasma etching and deposition, which are all 2D).

Our approach is a two step process designed to satisfy the following functional requirements: (a) integration of dimensional scales from the nano to the micro and beyond; (b) maximum utilization of existing fabrication tools; and (c) flexibility in achieving a large number of possible 3D configurations with minimum cost and maximum repeatability and yield that meets the connectivity constraint, provides an additional means for actuation, and allows for the seamless integration of existing 2D fabrication methods as well as new advances in the state of the art photolithography and nanopatterning. In the first step, all devices are fabricated on a planar substrate just as they are in today's semiconductor industry. In the second step an assembly technique is applied which the planar substrate is folded into a 3D or quasi-3D structure by appropriate actuation means. Imagine creating a microchip as a long ribbon, and then folding the ribbon over on itself many times as depicted in Figure 52. Designated compliant zones act as hinges between stiffer regions that contain micro and nano devices. By virtue of compliant circuitry that spans the hinge areas, full 2D connectivity is preserved across the entire length of the ribbon, even after folding. This is important for electronics as well as communication in MEMS sensors and actuators and integrated nano devices. An additional source of connectivity may also be achieved in the vertical, third direction by designing vertical connections to be formed when the planar folds are designed to contact each other at predetermined locations during folding.

Most connectivity remains within the plane and through the compliant hinges, while sparse connections permeate the vertical direction.

In our preliminary work, we have demonstrated a single 180 degree fold in a silicon-based device with magnetic actuation effecting induced folding (See Figure 53). The gold hinges are plastically deformed so that the folded membrane remains near 180 degrees. Electron beam evaporated gold was chosen for the compliant hinges due to its high ductility and comparatively small spring back angle. The hinges also complete a current loop around the perimeter of the membrane. By placing the device in a magnetic field and controlling the magnitude of current in this loop, a Lorentz force is generated that rotates the flap about its hinges. The Lorentz force is highly controllable and thus allows extensive experimental characterization of the mechanics of folding in our device. In the future, we will also explore different means of actuation such as stress and chemically induced folding. Some of these alternatives are more attractive than the magnetic method from the point of view of alignment and flexibility in 3D assembly. is an ideal means of actuation for the first round of prototypes because it is a highly controllable force that allows for good experimental analysis of the mechanics of folding.

The architecture achieved through various folding techniques is unique to the micro and nano realm because it creates a framework for building devices and structures that were previously impossible to imagine or too expensive to fabricate with other methods. For exampleAt present, our study aims to build 3D diffractive optical elements (3D-DOEs) as a case study in the technology development for foldedmembrane devices. Moreover, 3D-DOEs promise better performance in terms of light efficiency and angular selectivity than customary 2D diffractive optical elements that are customarily fabricated nowadays. Spacing multiple diffractive gratings or Ffresnel zone plates (perhaps as many as 50) vertically above one another establishes a matched filter with very high efficiency. The device could be fabricated using a sequence of bonding and etch-back steps, yet this would not only be costly, but very time consuming. Therefore, we plan to achieve the same results in a less expensive and more timely fashion by the folding scheme.

The first 3D diffractive device is displayed in its unfolded state, still attached to the substrate in Figure 54. Electrostatic combdrives tune the period of the binary grating, so as to change the angle of the diffracted orders. The fresnel Fresnel zone plate (essentially a diffractive lens) will be folded over and aligned to the grating as a demonstration of compound diffractive optics in 3D created through folding. Future work will focus on the final alignment and latching of the folds in addition to new actuation methods for folding that could be categorized as templated-self-assembly. Work in implementing multiple folds and studying their behavior of multiple folds is also underway. These steps are the early formative building blocks for establishing a multi-use platform. 3D assembly through folding lends itself to broader reaching goals such as combining discrete devices of varied functionality (optics, electronics, microfluidics, etc) into one cohesive, self-contained system capable of advanced sensing and response.



Fig. 52: Examples pre-fabricated thin 2D membranes folded into 3D structures in a two-step process. Black dots denote surface features nanofabricated on the membrane surface, and grey lines denote "hinges."



Fig. 53: Membrane flap before folding (left) and after folding to 180 degrees (right). Note the alignment tolerance achieved in folding (alignment fiducials are  $50\mu m$  wide).

continued

# Nano-Scale Machining with Femtosecond Laser Pulses

**Personnel** J.Y. Jia and M. Li (C.V. Thompson)

**Sponsorship** Panasonic Boston Laboratory

Researchers at the Panasonic Boston Laboratory have demonstrated the use of femtosecond laser pulses to drill 200nm-diameter holes in silicon films on insulating substrates. This technology has potential applications in microphotonics. We are collaborating in research on microstructural evolution in the vicinity of the drilled holes. This is expected to provide further insight into the drilling mechanism.

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*Fig.* 56: SEM image of holes drilled into a Si line on oxidized Si. A femtosecond laser was used.



*Fig.* 54: Top view of tunable grating (left) and static *Ffresnel zone* plate (right) before folding.



Fig. 55: An example of a heterogeneous system created through folding. Optics, microfluidics, nanopatterning, and circuitry are fabricated within the plane and then folded into one complete functional system.