The CNF is a member of the National Nanotechnology Infrastructure Network (www.nnin.org) and is supported by:

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Introduction

This year marks the 29th anniversary for the Cornell NanoScale Science and Technology Facility (CNF) and its predecessors. Throughout this period, CNF has had one mission: To support a broad range of nanoscale science and technology projects by providing state-of-the-art resources coupled with extensive staff support. In this mission, CNF has been singularly successful.

Our success is measured by the success of our users resulting from the services of our extensive staff and equipment resources—through their publications and products. Each year, over 700 users take advantage of CNF’s resources. In this publication, we try to summarize the extensive work carried out in CNF in the prior year. I believe that you will find this collection of work impressive, and even more impressive in realizing that only a fraction of the projects are reported in this volume.

CNF is supported through funding to the National Nanotechnology Infrastructure Network (NNIN). NNIN is an integrated network of user facilities, established March 2004 and supported by the National Science Foundation, serving the needs of nanoscale science, engineering and technology. NNIN provides users across the nation—in academia, small and large industry, and government—with open access, both on-site and remotely, to leading-edge tools, instrumentation, and capabilities for fabrication, synthesis, characterization, design, simulation, and integration. The NNIN also has extensive education, training and outreach activities. The thirteen partners in the network are facilities and laboratories at Cornell, Stanford, Michigan, Georgia Tech, Washington, Penn State, UCSB, Minnesota, New Mexico, Texas, Harvard, and Howard, with NCSU as an affiliate. Management of NNIN is from Cornell, with Prof. Sandip Tiwari, Director of NNIN, and Dr. Lynn Rathbun, NNIN Program Manager. Additional information about NNIN can be found at www.nnin.org.

Our participation in the network is of tremendous benefit to CNF. Interactions with our partners, common user projects, joint instrument acquisition proposals, research experience for undergraduates (REU) programs, joint workshops, educational efforts, back-up support, and the higher visibility of the network are all beneficial to our effective operation.

New People

Since the end of Sandip Tiwari’s tenure as CNF Director, the facility has been under the interim leadership of John Silcox, the David E. Burr Professor of Engineering. Many of you will remember John as part of the team who wrote the proposal that started CNF (or, the National Research and Resource Facility for Sub-Micron Structures as it was originally called). Senior Research Associate Dr. Jurriaan Gerretsen served as CNF’s interim Associate Director. Both John and Jurriaan have earned our gratitude for their leadership during the past year.

We are very fortunate to have recruited Donald Tennant as Director of Operations. Don, who started at CNF on August 15th, left Lucent Bell Labs after a 27 year career.
At Bell Labs, Don was a Distinguished Member of Technical Staff where he managed the Advanced Lithography Group. Since 1979 he worked at Bell in the area of high resolution electron beam lithography and related nanostructure technology. His work has had significant impact on a wide range of disciplines, including: soft x-ray imaging and extreme ultraviolet lithography (EUVL), high precision grating production for DWDM (dense wavelength division multiplexing), and gate technology for both high performance circuit applications and the exploration of the practical limits of silicon technology. His collaborative efforts with SUNY Stonybrook and Brookhaven National Laboratories have resulted in important advances in the field of x-ray optics and microscopy. Don has a broad technical background and is well known in the community. We look forward to having his experience help guide the technical direction for the CNF.

I am also pleased to announce that Edward Camacho is CNF’s newest Research Support Specialist in Lithography. Originally born in the Dominican Republic, Edward grew up in New York City. He received a BS in Microelectronic Engineering from the Rochester Institute of Technology in NY. Edward worked as a student technician for RIT’s Semiconductor & Microsystems Fabrication Laboratory, and as a process engineer for the same organization. He also worked at the General Electric Global Research Center in Niskayuna, NY, on state-of-the-art medical sensors for X-ray imaging technology.

I have the honor of being the new L.B. Knight Director of CNF, an appointment that started on August 15th. I am an Associate Professor in the Department of Materials Science and Engineering, and my research interests are in the area of organic electronics. I received a BS in Physics from the Aristotle University in Greece and a PhD in Mathematics and Physical Sciences from the University of Groningen in the Netherlands. It is a pleasure and a privilege to be associated with CNF and I look forward to working with CNF staff and users to make this facility an even better place for state-of-the-art research.

New Equipment

During the last year, CNF continued to upgrade its equipment resource base in Duffield Hall. To enhance our extensive ebeam lithography capability we acquired the Proxecco Proximity Correction software. This acquisition was funded by a generous semiconductor equipment grant from Intel. This software tool calculates adjusted ebeam exposure doses to compensate for electron scattering, assisting in the exposure of very small, closely backed features.

Intel continued its strong support of the CNF scientific computation initiative with an award to cover eight more nodes for our cluster along with Infiniband network fabric. These new nodes each contain two dual-core Xeon™ processors, providing users with effectively four processors per node. In addition, the Infiniband fabric...
will help accelerate parallel calculations that require high data throughput between nodes. These additions will be installed in October. At that point we will have a total of 56 nodes in support of nanoscale science calculations.

Through a special arrangement with Trion Technologies, CNF was able to acquire a new ICP etch tool configured for a variety of chlorine etch process. In particular it is currently being set up for dry etching of chrome mask blanks. The tool was delivered in August and is under installation. Using this new etch capability along with our existing ebeam lithography facilities, we intend to establish a complete process for production of masters for imprint lithography.

As a continued part of our restructuring plan for our etch capabilities, we converted our Plasma Therm 770 from a Bosch™ silicon etch tool to a III-V etch tool and retired both the Plasma Quest and the Plasma Therm 720. A used dual chamber Plasma Therm 720/720 RIE was acquired and is currently being installed. This tool will be configured for chlorine etching of silicon and aluminum.

After a long wait, CNF is nearing replacement of its ion implantation capability. Our old ion implantation capability was retired when we moved into Duffield. Several years ago we obtained a used Eaton Nova ion implanter which has been in storage. This summer renovations on a portion of Phillips Hall began to allow installation of this tool. This renovation will be complete in October and our new ion implantation capability will be available by January.

In 2006, we also welcomed the Applied Materials Instructional Laboratory operated by the College of Engineering. While not directly part of CNF, these facilities are located in the Duffield Hall clean room. Capabilities include two Karl Suss contact aligners, a sputtering tool and a Plasma Therm RIE system. The first course was taught in this facility in Spring 2006.

In addition to these major new installations, we continued to make progress on bringing additional capabilities on line as a result of the move to Duffield Hall. In particular, additional furnace tubes have been brought on line to support a broader variety of MEMS and electronics processes.

Broader Impacts

CNF conducts and participates in numerous educational outreach activities, both alone and as part of NNIN. As part of the NNIN Research Experience for Undergraduates (REU) program, 63 undergraduate students participated across NNIN in a 10-week program of focused research in nanotechnology. Ten of these spent the summer at CNF. We were most fortunate to receive supplemental support from the Intel Foundation to support five of these CNF REU students.

Nanotechnology is extremely popular with the undergraduate population. NNIN received well over 400 applications for our 63 openings in this program. As a result, the students this year and their resulting research projects were of exceptional quality. The research projects of the students reflect the breadth of disciplines, consist of an experimental project that can be completed within the limited time period, and provide a sound introduction to research. The student presentations at the end-of-program convocation held here at Cornell and their research reports are available through the NNIN web site.
This completes our 10th year of NSF site funding for REU. We believe our program is of extremely high quality and makes a significant contribution to the national human resource pool in nanotechnology. As per the program goals, a high percentage of these participants continue on to graduate studies in science and engineering. We are seeking funds to continue our program next year.

CNF continues to offer a number of workshops and special events. In April 2006, CNF presented a special 3 day hands-on workshop on nanofabrication as part of a course at Clarkson University. We are open to offering this workshop as a laboratory activity for integration into courses at other universities.

In October 2005, CNF conducted its first fall workshop on computation nanoscience, “Modeling the Nanoscale World” that focused on codes for nanomaterials, nanochemistry, and nanophotonics. This three day event consisted of morning lectures that addressed the underlying theory behind different approaches, while afternoon hands-on sessions gave participants the chance to try out codes, adapt input files, and in some cases learn directly from code developers. Participants attended from Canada, Japan, and seven different states, bringing a wide range of interests and backgrounds. Use of the CNF computational cluster has increased dramatically as a result of this effort and the CNF cluster currently provides computing time to over 40 users.

Preparations are underway for the second annual CNF Fall Workshop, “Building Nanostructures Bit By Bit” which will be held from October 23rd-25th. This workshop will include molecular dynamics techniques that can be used in microfluidics and biological studies, real space density functional techniques for clusters and nanowires, open source alternatives for modeling photonic structures, and first principle approaches for determining acoustic properties of materials. Details are available on the CNF web site.

The CNF has offered the CNF Short Course: Technology & Characterization at the Nanoscale (CNF TCN) for two years now. This lecture based short course is offered at the beginning for each summer, for new students and users. Because of considerable demand for this type of course, the CNF TCN is also offered in January. Each TCN is open to participants from academia, industry, and government.

Over the past year, CNF has hosted visits from hundreds of interested people; from prospective graduate students and incoming faculty members, to visiting dignitaries and corporate executives. We enjoy meeting and working with high school students — introducing them to the nano-world we live in and getting them into “bunny” suits for a tour of our clean room. We even have events specifically designed for the many elementary school students who visit us.

The possibilities of science open to them as they discover vegetable batteries and sound/wave dynamics.
In this past year CNF has been actively engaged in exploring and informing the community on the social and ethical issues (SEI) of nanoscience and nanotechnology endeavors. To this end we have organized a number of lectures with both local and international speakers, and collaborated with several Cornell departments to make their SEI events available online. These talks have ranged from introductions, catering both to technical and non-technical audiences, to more specialized subjects, such as the media coverage of nano-related research. All these multimedia resources, as well as a growing number of articles, reports and pointers to other meaningful organizations and initiatives, are available on our SEI website.

In an effort to increase the accessibility and ease-of-use of this site, we are currently working, under the auspices of NNIN, on a new and improved portal. This new portal will function as a repository for SEI resources—useful for both specialized audiences and those looking for a place to start their inquiries—and also as a place to find unique information on research projects being conducted at any of NNIN’s thirteen sites.

We are currently in the process of developing two research projects that look to explore the social and ethical dimensions of the fields of nanomedicine and ‘nano-military’. These two studies will contribute much needed empirical findings to the larger intellectual discussions on nano-SEI. Finally, in this past year we produced, within the REU program, a short video that provides the community with an introduction to the social and ethical dimensions of doing research at the CNF.

The many activities of CNF that address the broader impacts of nanotechnology are available both through the CNF and NNIN web sites.

30th Anniversary

We plan to celebrate the upcoming 30th anniversary with a major symposium, to be held in the summer of 2007. The symposium will include three tracks. The first one will explore the future of nanotechnology and will be organized by Don Tennant. The second track will highlight applications of nanotechnology in medicine, and will be organized by Prof. Harold Craighead. Last but not least, a track on ethical and societal implications of nanotechnology will be organized by Prof. Ronald Kline. Several plenary talks and a major poster session will give researchers the opportunity to present their work. Keep an eye on our webpage (www.cnf.cornell.edu) for more information.

On behalf of our staff, I present to you the 2005-2006 CNF Research Accomplishments. As always, we welcome your comments about CNF and its operations, as well as suggestions for improvements.

George Malliaras
L.B. Knight Director of CNF
A Selection of CNF-Related Patents, Presentations & Publications


“Decoherence in Phase Qubits”, K. Cooper, R. McDermott; invited talk APS March meeting (March 2005).


“Decoherence in Superconducting Qubits”, J.M. Martinis, invited talk; Conference on Macroscopic quantum coherence and computing, Italy (May 2006).


“Directional Microphone Array Fabrication”; Ron Miles; Cornell NanoScale Facility (CNF) External User Representative, National Nanotechnology Infrastructure Network (NNIN) Board and NSF review panel, University of Texas at Austin, Feb 26-27, 2006.


“Microwell Array Fabrication using Photolithography to Use As A Template for Biosensors”, ND Kalyankar, Cornell Nanoscale Science and Technology Facility (CNF) annual meeting, September 2005.


“Optofluidic Particle Manipulation and Characterization in Individually Addressed Nanowell Sensors”; B. Cordovez, S. Tung, D. Erickson, IEEE/LEOS Summer Topical on Optofluidics, Quebec City, July 2006.


“Pierce’s disease of grapevine: evaluating the role of pili in Xylella fastidiosa cell motility and colonization.” H. C. Hoch, Plant Pathology Department, University of Georgia, Athens. April 24 2006.

“Pierce’s disease of grapevine: new insights into Xylella fastidiosa cell motility and colonization.” H. C. Hoch, Department of Plant Pathology, Cornell University, Ithaca. April 5. 2006.


“Probing Electrostatic Potentials in Solution with Carbon Nanotube Transistors,” L. Larrimore, APS March Meeting (March 2006).


“Spin torque, tunnel-current spin polarization and magnetoeresistance in MgO magnetic tunnel junctions”;


“Spin-Transfer-Driven Ferromagnetic Resonance of Individual Nanomagnets”;


“Understanding and controlling the step bunching instability in aqueous silicon etching.” H. Bao, (Cornell Univ., 2005)


CNF  
Research  
Accomplishments  
2005-2006
A Micro/Nano Engineered Evolution Landscape

CNF Project # 398-91
Principal Investigator: Robert H. Austin

Abstract:
We have constructed fitness landscapes to study the interplay between the structure of habitat and the biology of life-cycle propagation. Since the structure and amount of habitat patches, as well as the regime of patch dynamics, have deep implications for metapopulation persistence and global extinction, we use these features to implement fitness landscapes for the directed adaptation and evolution of microorganisms.

Summary:
Park et al. showed how metabolic stress can induce a “population collapse” of E. coli into very small confining volumes.

In order to move beyond the original single patch experiment of Park et al. [1], we have now constructed a one-dimensional array of coupled micro-environmental patches (MEPs). “Coupling” means that the organisms are allowed to move between the MEPs through narrow channels. The channels are designed to be narrow enough that each MEP can be viewed as a local spatial-temporal niche in a much larger fitness landscape.

There are three fundamental parameters which characterize this coupled array of MEPs: (1) the local density of bacteria in the MEP; (2) the coupling between adjacent MEPs which allows bacteria to move between discrete MEPs; (3) the coupling between the MEP and some source of food for the ecological niche community which allows food to diffuse into a given MEP.

We have designed these to create fitness landscapes that can be designed to test evolutionary dynamics in a quantitative way, and have carried out initial experiments.

References:
A Micro/Nano Engineered Evolution Landscape

CNF Project # 398-91
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Primary Funding: NSF
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Web Site: http://feynman.princeton.edu/~austin/

No pictorial report was submitted for this project
Towards Electronic Nanodetection of DNA Molecules

CNF Project # 398-91
Principal Investigator: Robert H. Austin

Abstract:
DNA analysis has traditionally concentrated either on the sizing of long molecules or the functional analysis of short molecules. The integration of functional analysis and the capability of dealing with long molecules poses a formidable challenge—a single-molecule technique is needed. Nanochannel analysis, coupled with nanoelectrodes, bridges the gap between functional analysis and large DNA by analyzing single molecules [1].

Summary:
Using electrophoresis or hydrodynamic flow, a single double-stranded DNA molecule is introduced into a nanochannel with a cross-section which is much smaller than the radius of gyration, and a length greater than its contour length. We are now interfacing these highly confined nanochannels with single wall carbon nanotubes in order to reach down to single basepair nanodetection of elongated DNA molecules.

A major challenge is construction of sub-20 nm nanochannels. We are testing using PMMA to define the channels. We are currently two steps away from integrating carbon nanotubes with nanochannels.

At the same time we have been measuring and analyzing the dynamics of elongated DNA molecules in such nanostructures and we have shown how the statistical mechanics of the polymers change as the diameter of the polymer becomes less than the persistence length of DNA, in our case 50 nm.

References:
Towards Electronic Nanodetection of DNA Molecules

CNF Project # 398-91
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No pictorial report was submitted for this project
Wetting Dead-End Nanochannels

CNF Project # 398-91
Principal Investigator: Robert H. Austin

Abstract:

We have developed a method for wetting micro- and nanofluidic devices with water or any other pure liquid. The process is performed by enclosing the fluidic device in a liquid-filled cell, heating the cell to a temperature above the critical point of the liquid, and subsequently cooling the cell to room temperature. Because the process liquid is essentially a gas during wetting, arbitrary shapes can be wetted.

We demonstrate wetting of micro- and nanostructures in a fused-silica device with only a single inlet [1]. The process is low-cost, fast, safe, and very reliable.

Summary:

A growing variety of bioanalytic devices based on micro- and nanofluidic structures has been developed over the recent years. They have been used for capillary electrophoresis, DNA separation in synthetic gels, size-deterministic particle sorting, and complete self-sufficient analysis systems. Recently, nanofluidic devices have been used for single-molecule DNA sizing and restriction mapping.

However, with decreasing size and increasing complexity of the fluidic structures, the ability to wet using capillary action has become one of the limiting factors in the design of these devices. In particular, junctions, networks, and regions with decreasing channel dimensions are prone to formation of gas bubbles during wetting. Previous approaches toward complete wetting have concentrated on decreasing the free energy of wetting, forcing liquid through the device using pressure, or removing bubbles from the device by outgassing under reduced pressure.

Here we introduce a drastically different method, in which the device is wetted using supercritical water. The critical point of water is at 374°C and a pressure of 3212 psi. The liquid form of water is then obtained later by lowering the temperature. The process is thus conceptually related to critical point drying, which is often utilized in the fabrication of microelectromechanical systems.

References:

Wetting Dead-End Nanochannels

CNF Project # 398-91
Principal Investigator: Robert H. Austin
User: Robert Riehn

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• Wet dead-end nanostructures.
• Works with water.
• Simple and safe critical point wetting.

A

B

• Wet dead-end nanostructures.
• Works with water.
• Simple and safe critical point wetting.
A Nanochannel Switchyard for DNA

CNF Project # 398-91
Principal Investigator: Robert H. Austin

Abstract:
We have constructed a two-dimensional meta-material made of an asymmetric lattice of nanochannels, and have shown that in two dimensions, DNA can be transported in a fashion that is similar to propagation of light in a birefringent crystal.

Summary:
The meta-material consists of a square lattice of nanochannels that was fabricated into the surface of a fused silica wafer. A closed fluidic system was formed by sealing the structure with a fused silica cover slip. The width of the nanochannels was 100 nm and 200 nm, with channels running in one direction wider than in the other. The nanochannels were oriented at a 90° angle to each other and 45° to the sides of the lattice.

The transport direction under a d.c. bias is not parallel to the applied external field, but parallel to one principal axis of the lattice. The transport direction can be switched by 90° by applying an a.c. modulation to the d.c. bias. This effect arises from an interplay of entropic and dielectrophoretic energy contribution.

Our results imply that programmable DNA transport in nanofluidic devices is possible without the need for moving parts, external pumps, or an inflationary number of local electrodes.

References:

A Nanochannel Switchyard for DNA

CNF Project # 398-91
Principal Investigator: Robert H. Austin
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No pictorial report was submitted for this project
Measurement of Stepwise Length Changes in Single Invertebrate Thick Filaments by Using Nanofabricated Cantilevers

CNF Project # 486-93
Principal Investigator: Gerald H. Pollack

Abstract:

We investigated the dynamics of isolated *Mytilus* ABRM (anterior byssus retractor muscle) thick filaments manipulated by nanofabricated silicon-nitride levers. Single thick filaments were suspended between the tips of two microbeams oriented perpendicular to the filament’s axis: a deflectable cantilever and a stationary beam. Axial stress was applied by translating the base of the deflectable nanolever away from the stationary beam, which bent the nanolever. Tips of flexible nanolevers and stationary beam were imaged onto a photodiode array to track their positions. Filament shortening and lengthening traces, obtained immediately after the motor had imposed stress on the filament, showed steps and pauses. Step size was 2.7 nm and integer multiples thereof. Steps of this same size paradigm have been seen both during contraction of single sarcomeres [4] and during active interaction between single isolated actin and myosin filaments [3], raising the question whether all of these phenomena might be related.

Summary:

In approaching the mechanism of motility and contraction, considerable attention has turned toward the elastic properties of the muscle filaments themselves. Early studies of muscle [2] attributed almost all of the sarcomere’s elasticity to cross-bridges, rather than to filaments. However, recent investigations on isolated thick filaments from *Mytilus* and *Limulus* have shown that physiological stresses produce filament-length changes of up to 23% and 66% respectively [5]. The ability to change length has also been demonstrated in synthetic vertebrate thick filaments during imposed physiological stress [1], although the magnitude was considerably smaller.

In this study we employed novel nanofabricated cantilevers to manipulate single myosin filaments and report for the first time dynamic length-change measurements on isolated thick filaments. We find that isolated thick filaments from the anterior byssus retractor muscle (ABRM) of the blue mussel *Mytilus edulis* change length in steps. Steps were observed consistently, and their size was indistinguishable from that found both during contraction and stretching of intact activated sarcomeres, and of isolated activated actin-myosin filament pairs [3, 4].

References:

Measurement of Stepwise Length Changes in Single Invertebrate Thick Filaments by Using Nanofabricated Cantilevers

CNF Project # 486-93
Principal Investigator: Gerald H. Pollack
User: Yekaterina Nagornyak

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Primary Funding: The National Institutes of Health
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- The dynamics of thick filaments were investigated with nanofabricated silicon-nitride cantilevers.
- Filament shortening and lengthening occur in a stepwise manner.
- Step size was 2.7 nanometers and integer multiples thereof.
- This 2.7-nm paradigm also appears in various contractile systems.

Figure 1, top left: Schematic of apparatus with thick filament attached between deflectable and stationary nanolevers.

Figure 2, bottom left: Traces of length change.

Figure 3, above: Histograms of shortening- and lengthening-step size.
Biomolecular Applications of Entropic Traps

CNF Project # 551-95
Principal Investigator: Harold G. Craighead

Abstract:
We have made nanoscale constrictions in fluid channels of varying size and orientation as entropic traps to investigate the dynamics of individual DNA molecules as they probe and subsequently escape through the trap. Previous researchers have made or modeled entropic traps in multiple configurations for separating DNA molecules or for studying the motion of DNA molecules through entropically restrictive spaces [1-3,5]. This work has focused on obtaining the time that individual molecules spend at the boundary between entropically favorable and unfavorable regions when driven electrophoretically.

Summary:
We fabricated entropic traps of various size and orientation in fused silica substrates. Typical structures were defined through a two-layer lithographic process in which the deep region of the device (entropically favorable) was patterned and etched with one layer of lithography, and the shallow region of the device (entropically unfavorable) was patterned and etched with a second layer of lithography. This is the configuration that others have used and modeled in the past. We have also made sideways-oriented devices using a one layer electron beam lithography process to define both the deep and shallow regions of the device. The devices are sealed by press bonding and annealing a 170 µm thick fused silica cover wafer to the substrate. Using both processes, we have fabricated and tested devices with shallow regions as small as 20 nm.

Other researchers have examined the trapping of DNA at entropic traps by averaging over many thousands of traps or by modeling the behavior of DNA in similar geometries [1-5]. Still others have examined DNA in two dimensional arrays of cavities separated by entropic barriers [4]. For investigating the dynamics of single molecules as they move through an entropic trap, the devices described herein offer a number of advantages over the aforementioned devices. The experimental setup and device geometry allow individual molecules to be observed throughout the trapping process with fast time resolution. Furthermore, the geometry used in these experiments allows for easy control of the length of the shallow region. This is important as the dynamics of the trapping-escaping process should depend upon the degree of confinement of the molecule throughout the process. The degree of control over the experimental parameters and the ability to observe individual molecules as they escape through entropic barriers yield information regarding the physical dynamics of the trap-escape process.

Some data are presented in Figure 3 for a sideways-oriented device with a 59 nm wide shallow region. The data are fit to the theory presented by Han [5]. Devices with shallow regions of 20 nm have been used to trap DNA molecules as short as 400 base pairs. With these dimensions, it should be possible to trap biomolecules with significant secondary or tertiary structure. Future work will focus on studying and separating large RNA complexes and proteins.

References:
Biomolecular Applications of Entropic Traps

CNF Project # 551-95

Principal Investigator: Harold G. Craighead
Users: Joshua Cross, Kevan Samiee, Elizabeth Strychalski

Affiliation: Applied & Engineering Physics, Cornell University
Primary Funding: Princeton-NIH
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- Entropic traps made in both top-down and sideways configurations for studying single molecule trapping dynamics.
- 20 nm traps tested with 400bp DNA.
- New applications: DNA denaturation, protein and RNA trapping.

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**Figure 1, top left:** Experimental schematic. To measure the DNA trapping time, the DNA is illuminated and photons are collected.

**Figure 2, bottom left:** A SEM showing a typical “sideways” entropic trap.

**Figure 3, above:** A graph of the trapping time versus the inverse electric field at the trap interface.
Compression and Free Expansion of Single DNA Molecules in Nanochannels

CNF Project # 551-95
Principal Investigator: Harold G. Craighead

Abstract:
Our goal is to understand the mechanical properties of single DNA strands under high compression forces in confined environments. Characterization of these properties is important for an understanding of DNA packing into chromatin or bacteriophage heads as well as the verification of theoretical biopolymer models. We are currently investigating the compression and subsequent free expansion of DNA molecules in artificial nanofluidic devices [1]. Our method stands in contrast to established techniques for investigating single DNA strands [2], for example the stretching by an external force, which was used to study the elasticity of DNA molecules [3].

Summary:
Our microfluidic devices consist of parallel fused silica nanochannels (diameter 90-130 nm) which have a narrow constriction for blocking electrophoretically driven DNA. The structures were fabricated by sealing a structured wafer (500 µm) with a thin cover wafer (170 µm). The nanochannels were built by deep reactive ion etching using a chromium or aluminum mask which was patterned by electron beam lithography.

In our experiments, single λ DNA multimers were first driven into the nanofluidic channels by an electric field. Since the radius of gyration of the biomolecules was bigger than the channel diameter, their equilibrium state was no longer a sphere but instead an elongated cylinder [4]. Forcing the stretched DNA into a channel constriction led to a compression of the molecule into a tight conformation. When the electric field was turned off, the DNA molecule slowly expanded back to the energetically favorable stretched out conformation.

This expansion can be interpreted with the help of a simple polymer model based on self-avoidance effects.

References:
Compression and Free Expansion of Single DNA Molecules in Nanochannels

CNF Project # 551-95
Principal Investigator: Harold G. Craighead
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- We have investigated compression and ensuing relaxation of single DNA molecules.
- Compression is performed at constrictions inside nanochannels.

Figure 1: Schematic of a DNA strand entering into a nano-channel (1-3), its compression (4-6) and its relaxation (7-9).

Figure 2: Constriction inside an open fused silica 130 nm channel.
Development of Novel PDMS-Based Microfluidic Devices for Blood Analysis

CNF Project # 551-95
Principal Investigator: Harold G. Craighead

Abstract:
We are developing novel microfluidic devices for biological applications. Specifically, we are using microfluidic technology to examine anticoagulated whole blood and its components.

Channels are cast in polydimethylsiloxane (PDMS) using well-known fabrication methods. The inner surfaces of the devices are chemically modified with covalently bound surface treatments to improve biocompatibility. Devices require inexpensive and uncomplicated clean room fabrication. Rapid assembly removes the need to clean channels for reuse: a new device can be used for each experiment, eliminating cross-contamination between runs. Device disposability is another feature of our chips that is desirable for clinical use as a biomedical diagnostic device.

Summary:
Microfluidic devices have been constructed from glass and PDMS using well-known PDMS casting and bonding techniques. Microchannels cast in PDMS are irreversibly bonded to PDMS spun onto glass cover slides using an oxygen plasma. Polymer tubing is inserted into the side of the device in plane with the channel, or an open plastic tube is inserted perpendicular to the channel to allow pressure drive of fluid through the channel. Depending upon the specific application, a device may be modified with a surface treatment, such as polyethylene glycol. The materials, device structure, and pressure drive systems used are designed to maximize biocompatibility.

Devices are mounted on an inverted fluorescence microscope to visualize flow. Samples are driven through devices using pressure, which is typically provided by a syringe pump or hydrostatic pressure from a column of liquid. Samples for interrogation are often incubated with fluorescent labels to identify biological components of interest and characterize their flow behavior through the channels.

Biomedical diagnostic devices using microfluidic technology for the examination of whole blood and its components would improve upon conventional techniques for blood analysis and take advantage of the flow characteristics of blood through micrometer-sized channels and apertures. Conventional blood testing can require tens of milliliters of whole blood, while miniaturized testing using microfluidic geometries call for as little as a few microliters.

Using point of care microfluidic diagnostic devices to examine single cells in addition to populations of cells, without the need for expensive and time-consuming sample preparation and equipment, would allow more frequent testing of more patients. The small size of microfluidic chips and their inexpensive fabrication in PDMS would give a disposable device for blood analysis at a dramatically reduced cost and improved reproducibility over conventional testing techniques.
Development of Novel PDMS-Based Microfluidic Devices for Blood Analysis

CNF Project # 551-95
Principal Investigator: Harold G. Craighead
User: Elizabeth A. Strychalski
Affiliation: Applied & Engineering Physics, Cornell University
Primary Funding: Gift Funding
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- Simple fabrication techniques lend themselves to rapid prototyping and quick assembly of many disposable devices.
- Microfluidic channels cast in PDMS provide biocompatible surfaces for biological applications, including analysis of anticoagulated whole blood.
- PDMS can be readily modified with chemical surface treatments to improve biocompatibility.

Figure 1: Glass-capped PDMS microfluidic device assembled and ready for biological applications (Euro coin shown for scale).
Zero-Mode-Waveguide (ZMW) for Surface-Cell Interaction Study

CNF Project # 599-96
Principal Investigator: Harold G. Craighead

Abstract:

Sub-wavelength metallic apertures have been used for optical single molecule studies. These devices are referred to as zero-mode waveguides (ZMWs). Because of their small observation volume, ZMWs allow single-molecule fluorescence correlation spectroscopy (FCS) at micro-molar concentration and microsecond temporal resolution [1]. Recently ZMWs have found further use in studies of diffusion of single lipids within a cell membrane [2]. Because ZMWs confines fluorescence excitation to zeptoliter volumes, they have become an effective tool in observation of cell functions with high spatial resolution. However, because the excitation volume is confined to the bottom ~ 50 nm of the holes, the question of how far cells probe into the waveguides still remains to be resolved [3]. In our project, we fabricated closely spaced waveguides allowing more sampling points in a given illumination area. These structures were used to study cell and surface interactions. We also compared cell adhesion to aluminum and SiO₂ surfaces by recording the fluorescence of Dii-labeled cell membranes that are excited inside the ZMW holes.

Summary:

Fused silica substrates are used in fabricating zero mode waveguides. A 100 nm thick aluminum film was patterned using electron-beam lithography. The substrate is subsequently diced into individual dies for experiment. For comparison, a few dies are further coated with a thin (3 nm) layer of SiO₂, by electron beam evaporation. Rat basophilic leukemia (RBL)-2H3 cells that contained Dii C16 suspended in solution were applied to these surfaces, incubated for 30 minutes, and fixed with formaldehyde. Fluorescent images are taken to compare the adhesion of cells to surfaces, and actively probe into the waveguides while the cells are still alive. The affinity difference of cells to SiO2 and aluminum surfaces remains to be quantitatively determined. In addition, further experiments are planned to test the investigation of cells into ZMWs.

References:


Zero-Mode-Waveguide (ZMW) for Surface-Cell Interaction Study

CNF Project # 599-96
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- ZMWs allow single molecule study at micro-molar concentrations.
- ZMWs can be used to monitor diffusion of single lipids in cell membranes.
- How far into the ZMW holes a cell membrane would investigate remains to be determined.

Figure 1: SEM image of ZMW aluminum surface, showing developed e-beam resist patterns.

Figure 3: (a) Trans-illuminated ZMWs showing four different hole sizes, each at 1 µm spacing, and epi-fluorescence of RBL cells fixed to (b) SiO<sub>2</sub> surface, (c) Aluminum surface, showing qualitatively more cells adhering to SiO<sub>2</sub> surface than to Al surface.

Figure 2: SEM image showing ZMWs on aluminum surface after lifting off e-beam resist.
Patterned Biofunctional Polymer Brushes

CNF Project # 640-97
Principal Investigator: Christopher K. Ober

Abstract:

Protein patterning was carried out using a simple procedure based on photolithography wherein the protein was not subjected to UV irradiation and high temperatures, or contacted with denaturing solvents, and strongly acidic or basic solutions. Self-assembled monolayers of poly(ethylene glycol) (PEG) on silicon surfaces were exposed to oxygen plasma through a patterned photoresist. The etched regions were back-filled with an initiator for surface-initiated atom transfer radical polymerization (ATRP). ATRP of sodium acrylate was readily achieved at room temperature in an aqueous medium. Protonation of the polymer resulted in patterned poly(acrylic acid) brushes. A variety of biomolecules containing amino groups could be covalently tethered to the dense carboxyl groups in the brush region under relatively mild conditions. The PEG regions surrounding the poly(acrylic acid) brush prevented non-specific adsorption. Bovine serum albumin (BSA), tagged with specific ligands, were immobilized on the brushes. BSA enhanced the non-denaturing environment of the PAA brushes and also “blocked” non-specific interactions. Biotin-tagged BSA was used to demonstrate a strategy of physically immobilizing proteins through the strong streptavidin-biotin complex formation. Such a specific, physical immobilization is expected to better retain the protein functionality than covalent binding.

Summary:

The presentation of a protein or biochemical ligand of interest against a protein-resistant background is of importance in several areas of biotechnology [1]. Much work has been done on patterning of biological molecules [2] and the patterned surfaces are used to study cell-surface interactions [3]. Most of the patterned substrates are modified with self-assembled monolayers. Recently, polymer brushes attract a lot of interest in modifying surfaces for biological applications [4].

The spontaneous reaction of amine containing biomolecules with activated carboxylic acids makes silicon surfaces functionalized with carboxyl groups a versatile platform for microarray technology. A variety of biological molecules can be covalently immobilized on such substrates under mild conditions of pH and temperature.

Here, we prepared poly(acrylic acid) brush patterns with PEG as background on a silicon wafer. First, PEG was vapor deposited onto a silicon wafer which was cleaned using oxygen plasma cleaner. Subsequently a photoresist was spin-coated onto the wafer and patterned using photolithography. The uncovered PEG was removed from the oxygen plasma cleaner and the remaining photoresist was removed. The PEG patterned surface was backfilled with a silane surface initiator. Surface initiated atom transfer radical polymerization (ATRP) was used to grow a poly (sodium acrylate) brush at room temperature. The carboxyl groups on the polymer brush were activated by 1-ethyl-3-(3-dimethylaminopropyl)-carbodiimide (EDC)/N-Hydroxy succinimide (NHS) and then reacted with amino groups on target proteins. The poly(acrylic acid) brushes can be functionalized with proteins in different ways. First, protein molecules can be directly immobilized by covalent linkage. Second, a protein such as avidin can be attached, which could then be used to bind different biotinylated molecules through the almost irreversible avidin-biotin interaction. In a third approach, biotinylated BSA can be tethered to the surface, followed by binding of streptavidin through the biotin-streptavidin interaction. Such a surface can be further functionalized with a variety of biotinylated proteins [5].

AFM was used to examine the patterned surface. Fluorescence microscopy was used to study the protein immobilization process. The patterned proteins showed a sharp contrast against a protein resistant PEG background when observed under a fluorescence microscope.

References:

Patterned Biofunctional Polymer Brushes

CNF Project # 640-97
Principal Investigator: Christopher K. Ober
Users: Rong Dong, Sitaraman Krishnan

Affiliations: 1. Department of Chemistry; 2. Material Science and Engineering; Cornell University
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Figure 1: AFM height image of 2 µm wide alternating stripes of PEG SAM and PAA brush on silicon substrates.

Figure 2: Fluorescence image of BSA-FITC immobilized on 2 µm wide PAA brush patterns in a background consisting of PEG SAM. The darker regions are the PEG covered substrate where there was no covalent binding or nonspecific adsorption of BSA-FITC.
Retinal Implant Project

CNF Project # 657-97
Principal Investigators: Dr. Douglas Shire and Dr. John Wyatt

Abstract:
The purpose of the Retinal Implant Project is to restore useful vision to patients who are blind with degenerative retinal diseases. The primary illnesses we hope to treat are retinitis pigmentosa (a primary cause of inherited blindness) and age-related macular degeneration (the leading cause of blindness in the developed world). Both these diseases cause the eventual destruction of the photoreceptor cells—rods and cones—in the retina, leaving intact the ganglion cells which transmit electrical impulses (and hence visual information) to the brain. The ganglion cells may be stimulated, however, with biphasic current pulses from a microfabricated electrode array. Blind surgical volunteers have consistently described visual percepts that resulted from such stimuli, and this has led our team to develop a wireless, implantable retinal prosthesis.

Summary:
The implanted portion of our device consists of power and data secondary receiving coils, a small number of discrete components, and a custom-designed integrated circuit which consists of a delay locked loop for clock and data recovery, current drivers for each of the 15 electrodes in the stimulating electrode array, and a programmable function generator capable of stimulating with a wide range of pulse widths and amplitudes. The current outputs drive iridium oxide stimulating electrodes, which in turn give rise to the visual percepts mentioned above.

The CNF-fabricated components of this system have been the flexible circuit on which the remaining components of our prosthesis are assembled, and the stimulating electrode arrays themselves. Si wafers serve as carriers for these freestanding films during processing. The electrode leads are fabricated inside of a ‘sandwich’ of polyimide or parylene-C, while the IrOx electrodes themselves are fabricated by reactive sputtering.

Assembly of the intraocular components of the prosthesis is accomplished by flip chip stud bumping of the IC onto the host flex circuit, and epoxy die attach of the discrete components and coils. An external patient interface unit, under development by our team, will consist of a video camera for capturing images in the patient’s environment, a digital signal processor, and an RF transmitter and coil to relay power and data to the implanted device. The patients will also be offered the ability to adjust the electrical stimulation parameters to optimize their perception, in much the same manner as modern hearing aids and cochlear implants.

A major scientific challenge, however, remains in deciphering the neural code by which action potentials in the central nervous system (of which the retina is a part) are transformed into meaningful percepts. To this end, we have also developed a flexible, transparent retinal recording electrode array which may be affixed to the inner surface of the eye without significantly blocking or dimming the ambient light passing through the lens on its way to the retina. This device may be connected in vitro or in vivo to perform chronic retinal recording experiments while the subject animal roams freely in its environment. Our goal is to record the naturally occurring pattern of action potentials which result from various visual stimuli, and to adaptively develop a pattern of electrical stimulation which results in a similar response by the host tissue.

Further details on our program of research may be found at http://www.bostonretinalimplant.org.

The authors wish to acknowledge the financial support of the Veterans Administration Rehabilitation Research and Development office, the NIH, and NSF.

References:
Retinal Implant Project

CNF Project # 657-97
Principal Investigators: Dr. Douglas Shire¹, Dr. John Wyatt²
Users: Marcus Gingerich¹, Douglas Shire¹

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Web Site: http://www.bostonretinalimplant.org

- Overall project goal: Restore useful vision to patients blind with degenerative retinal diseases.
- Figure 1: Intra-ocular components of chronically implantable prosthesis assembled on flexible circuit.
- With 15-electrode iridium oxide stimulating electrode array fabricated at CNF.
- Figure 2: Transparent retinal recording electrode array.
- With CNF-fabricated indium tin oxide leads (center) used for in vitro studies; goal is to develop chronic in vivo recording system.

Figure 1

Figure 2
Improving the Mixing Uniformity of Microfluidic Mixers

CNF Project # 692-98
Principal Investigator: Lois Pollack

Abstract:
This project continues to focus on the development of microfluidic mixers for use with a variety of experimental probes of macromolecular folding, including x-ray scattering and fluorescence microscopy. Much of our experimental work is carried out using x-ray beams with dimensions on the order of 10’s of microns. Thus the width of the microfluidic channel must be on the order of 50 µm to allow passage of the x-rays through the through-etched channel without touching the walls. This constraint can seriously affect the mixing uniformity of hydrodynamic focusing mixers, first proposed in [1], as the length scale for focusing is comparable to the width of the channels and can be significant when the channels are large. The non-uniform mixing that occurs during focusing contributes in a non-negligible way to the uncertainty of the measurement, as it contributes to the error in mixing time.

We have constructed, tested and simulated the flow conditions within a re-designed mixer. The use of two additional channels allows us to decouple the hydrodynamic focusing process from the diffusive mixing that triggers the conformational changes of interest. The sharpness of the mixing increases by an order of magnitude relative to earlier versions of this device. This improvement will enable sub-millisecond mixing reactions of ionic species with macromolecules, such as those triggering the folding of RNA [2].

Summary:
We have successfully characterized, fabricated and tested hydrodynamic mixers which separate the focusing process from the diffusive mixing process.

In previous designs, a thin jet containing macromolecules in unfolding buffer is created by hydrodynamically focusing the solution with folding buffer (buffer containing small ions that trigger the reaction). However, in devices with channels larger than 10’s of microns, significant contact between the solutions occurs during the focusing process and initiates mixing before the jet is fully formed. This premixing contributes significantly to the mixing uncertainty. This problem has been remediated by introducing flow through two additional channels that protect or sheath the macromolecule-containing solution during the focusing process. The folding buffer comes into contact with the macromolecules only after the thin jet is fully formed.

We have fabricated devices by imprinting soft plastic to create 50 µm wide channels, following a procedure described by Kameoka et al. [3]. The master was fabricated on a silicon wafer using contact photolithography and deep reactive ion etching. Full characterization by both experiment and simulation show more than an order of magnitude decrease in mixing time [4].

References:
Improving the Mixing Uniformity of Microfluidic Mixers

CNF Project # 692-98
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Figure 1 shows a light microscopy image of the five-inlet port, microfluidic mixer. The mixer is fabricated by imprinting plastic on a silicon master with raised features.

Figure 2 demonstrates the operation of the mixer shown in Figure 1. Unfolded macromolecules flow in the left port. Sheath flow from the diagonal channels (dotted) prevents contact between the macromolecules and the folding buffer (flows from top and bottom), until hydrodynamic focusing into a thin jet is complete. Uniform, rapid diffusive mixing across the thin jet triggers the folding reaction of interest.
Microfluidic Cell Culture Analog Devices to Mimic Animal Exposures to Toxins and Drugs

CNF Project # 731-98
Principal Investigator: Michael L. Shuler

Abstract:

Our group has developed microfluidic in vitro devices to mimic the response of humans or animals to drugs and toxins. Each device, or cell culture analog (CCA), contains an array of pseudo tissues that are interconnected by microfluidic channels. The recirculation of blood surrogate through the microchannels allows us to study tissue-tissue interactions, such as the break down of a parent compound in the liver and subsequent transport and reaction in the lung. We then combine these in vitro device experiments, which capture adsorption-distribution-metabolism-elimination-toxicity (ADMET) data, with physiologically-based pharmacokinetic model simulations to predict toxin and drug dynamics in humans.

Our original 4-chamber CCA devices were used to do a toxicity study of 1,2-naphthalenediol-diacetate, 1,4-naphthalenediol-diacetate, 1,8-naphthalenediol-diacetate and 2,3-naphthalenediol-diacetate. In an effort to improve cell culture conditions, a new CCA device was designed for use with alginate-based three-dimensional cell cultures.

We have utilized a microfabricated CCA device containing liver-marrow-normal tumor-resistant tumor cell compartments to study the efficacy of multidrug resistant (MDR) cancer therapeutics. With our device, we have found that the use of mixtures of helper drugs at reduced concentrations appears to have a better effect on resistant cancer cells with potentially reduced side effects to other tissues. We have developed a real time imaging system for the MDR CCA in conjunction with GFP expressing cell lines.

An independent GI tract CCA has been added to a lung and liver-chamber “body” CCA. The GI tract CCA consists of two chambers separated by a microporous membrane on which epithelial cells are cultured. Compounds of interest are subjected to an in vitro digestion then pumped through the top chamber, allowing drug to be absorbed through the epithelial layer and circulated into the “body” CCA. The CCAs have been used to recreate the toxic effects of acetaminophen. Preliminary results show that when ethanol, a cytochrome P450 2E1 inducer, is added to the culture medium, the toxic effects of acetaminophen are amplified.

A new CCA device was designed and fabricated to test fluorouracil-based drugs for colon cancer. It consists of separate chambers representing liver, colon, colon cancer, and bone marrow. The cytotoxic effect of 5-FU was tested with the constructed CCA system, and in a 48 hour experiment tumor, colon cancer cells (HCT-116) showed lower viability in response to 100 uM 5-FU.

New breast cancer cell lines, MCF7-61 and MCF7-16, with estrogen inducible expression of green fluorescence protein (GFP), were constructed to address the estrogen receptor (ER), estrogen respond element (ERE) and the ER-activating protein-1 pathway separately.

With these cells, we are now using the CCA device for screening endocrine disruptors, studying their activation pathways, and exploring possible synergistic effects.

Summary:

Our group has developed several cell culture analog devices to obtain ADMET data for predicting drug and toxin response in humans and animals. Thus far we have applied these devices to determine the route of toxicity of an environmental toxicant, naphthalene; to predict efficacy of cancer treatment strategies to resistant tumors and colon cancer; to model transport across the blood brain barrier and through the gut; and to screen for endocrine disrupters.
Microfluidic Cell Culture Analog Devices to Mimic Animal Exposures to Toxins and Drugs

CNF Project # 731-98
Principal Investigator: Michael L. Shuler
Users: Daniel Tatosian, Xinran Li, Gretchen McAuliffe, Jong Hwan Sung, Hui Xu, Jung Y. Chang

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- Microfabricated cell culture analog devices.
- ADMET studies in vitro.
- Closed system with metabolite recirculation and barrier (gut).
- Coupled experiments and PBPK math models.

Figure 1, top left: Viability results of 24 drug study of doxorubicin (DOX) with the modulators cyclosporine (CSP) and/or nicardipine (NCB) in CCA device.

Figure 2, bottom left: Fabricated GI tract to mimic transport through the gut wall into the blood stream of the “body” CCA.

Figure 3, above: “Body” CCA for use in evaluating efficacy of fluoracil drugs on colon cancer.
Nonspecifically Bound Protein Removal from a Microfluidic Channel with an Integrated Surface Acoustic Wave Device

CNF Project # 762-99
Principal Investigator: Harold G. Craighead

Abstract:
Unwanted protein adsorption to micro/nanoscale devices, commonly referred to as nonspecific binding, can clog fluidic channels, inhibit sensor function, and decrease signal-to-background ratios. In this work, we demonstrate ultrasonic nonspecifically bound protein removal from a sealed microchannel integrated with a Surface Acoustic Wave (SAW) device. Device operation is shown to increase nonspecifically bound protein desorption rates. The removal mechanism is the combined effect of hydrodynamic drag-induced reduction in the bound state potential energy well depth and temperature induced increase in the fluid-damped attempt frequency. Fluorescently tagged bovine serum albumin (BSA) was used as a model protein given its high concentration in plasma. Experiments were performed to quantify fluorescence intensity vs. time under inactive and activated device conditions. We first demonstrate negligible nonspecifically bound protein removal under low Reynolds’ number flow conditions. We then compare pressure-driven flow results to activate device desorption kinetics. Activated device channel areas where protein removal was optimal demonstrated intensity drops approaching 86% with 2.5 W delivered to the interdigital transducer. Cavitation was not observed during the experiment, though further work will be required to determine heat dissipation levels into the fluid.

Summary:
We hypothesized that ultrasound generated by a piezoelectric transducer driven at high-power relative to traditional gravimetric sensor applications could increase the successful bound-to-free transition probability for protein adsorbed to inorganic surfaces. To test this hypothesis and demonstrate integration with traditional photolithographic processes and materials, we fabricated SAW devices on 128° YX lithium niobate (LiNbO₃) wafers. Devices were diced and individually photopatterned with SU-8 2010. Figure 1(a) is a device photograph. A photodefined 100 µm channel, drawn in black and not to scale, was capped using a coverslip spin coated with poly(dimethylsiloxane) (PDMS) at 4000 rpm. The coverglass/PDMS cap was then pressed against the SU-8 channel to form a sealed channel with the lithium niobate acting as the substrate. Figure 1(b) demonstrates an edge-on view with the device sealed with fixture pressure applied via screws.

Fluidic channels were coupled through the chip by gluing silicone tubing to holes sandblasted through the lithium niobate after dicing and SU-8 photopatterning. Channel architectures developed in PDMS and SU-8 were tested. The difference in attenuation is clearly demonstrated by insertion loss values of 40 dBm for a SU-8 channel and 60 dBm for PDMS. Insertion loss for a device with no channel was 25 dBm. Further optimization using unidirectional transducers has been developed for cellular phone applications and could be easily applied to reduce input power requirements.

Taking integrated devices packaged as described, a 10X PBS buffer solution containing 55 micrograms/mL BSA fluorescently tagged with Alex Fluor 594 was flowed through the channel for 5 min at 3 microliters/min. Removal throughout the channel had a periodic spatial dependence as figure 1(e) demonstrates. It appears that a standing wave is generated in the channel resulting from complex diffraction patterns generated by the interdigital transducer design. Improved removal uniformity could be obtained by redesigning the transducer layout.

Results demonstrate proof-of-concept and elucidate areas requiring improvement. We conclude by suggesting broader applicability to systems where ultrasonically induced dissociation can disrupt van der Waals interactions (e.g. bead-based sorting and sensing).

References:
Nonspecifically Bound Protein Removal from a Microfluidic Channel with an Integrated Surface Acoustic Wave Device

CNF Project # 762-99
Principal Investigator: Harold G. Craighead
Users: Don Aubrecht, Grant Meyer

Affiliation: Applied & Engineering, Physics, Cornell University
Primary Funding: Cornell LIFE Program, NYSTAR
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- Ultrasound induced nonspecifically bound protein removal.
- Integrated microfluidic channel with Surface Acoustic Wave device.
- SU-8 defined microchannel minimizes acoustic losses.

Figure 1: (a) SAW device with channel placement drawn in black. (b) Edge-view with electrical connections and fluidic channel. (c) Control and Active device fluorescent intensity vs. time. (d) Raw image before device activation obtained with fluorescence microscopy. (e) Channel after SAW activation for 10 min.
**Micropatterned Substrates with Improved Uniformity of Deposition for DNA Microarrays**

**CNF Project # 762-99**  
**Principal Investigator: Harold G. Craighead**

**Abstract:**

Biomolecular arrays have become a core technology used in numerous fields for parallel analysis. However, it has become obvious that current microarrays are hindered by methodological and technological barriers that limit their sensitivity, reproducibility, and therefore their utility. A fundamental problem with DNA microarrays is the relative insensitivity in detecting weakly expressed transcripts; this is especially significant when examining gene expression in complex tissues. While it has been proposed that a marked improvement in the detection limit of microarrays could be achieved if a uniform DNA layer can be spotted [2], the issue of its implementation has not been fully addressed. In an effort to resolve this problem, we have fabricated micropatterned substrates for the uniform deposition of DNA with a polymer lift-off technique [3]. We pursued a methodology compatible with existing microarray technologies, which constrained the substrate area where the probe DNA were spotted. This yields a uniform layer of deposited DNA and improves the microarray data reproducibility between replicates on a single slide, and also across multiple slides.

**Summary:**

Micropatterned substrates were fabricated by vapor-deposition of a conformal polymer coating on glass substrates, followed by patterning with photolithography and oxygen plasma reactive ion etching (Figure 1). This process defined the exposed areas of the underlying substrate. The fabricated substrates were spotted with probe oligonucleotides in a conventional microarrayer. The polymer coating was then peeled off from the surface leaving behind only the material spotted within the openings. The substrates were later hybridized and proccessed in the same way as a conventional microarray glass slide.

To test deposition uniformity, fluorescently-labeled oligonucleotide probes were spotted on microfabricated substrates and control slides, and then imaged with confocal microscopy. It was observed that constraining the available area to the patterned openings facilitated the uniform deposition of DNA, reducing or eliminating the “donuts” seen with commercial glass substrates (Figure 2). To test the impact on sensitivity and reproducibility, we used successive dilutions of an unlabeled gene pool probe, which functioned as a universal positive control binding to RNA from any murine tissue sample [4]. As expected from previous reports [4], no signal was observed on control slides when the DNA was spotted at concentrations below 5 pg/µl. This provides an indication that constraining the deposition surface as well as providing a more hydrophobic region outside the exposed glass areas, produced uniform probe DNA deposition patterns and could dramatically improve the reproducibility of microarray data.

Collectively, these results indicate that arrays spotted on microfabricated substrates are more reproducible than standard arrays. Furthermore, the presence of the polymer during the spotting procedure reduces the background present in inter-spot areas. The combined improvements could allow for faster identification of differentially expressed transcripts in complex tissues. Experiments are under way to evaluate the sensitivity of these microfabricated arrays with real tissue samples.

**References:**


Micropatterned Substrates with Improved Uniformity of Deposition for DNA Microarrays

CNF Project # 762-99
Principal Investigator: Harold G. Craighead
Users: Jose M. Moran-Mirabal, Christine P. Tan, Eric O. Williams, David M. Lin

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• A major challenge for current microarray technology is the uniform deposition of spotted biomaterial.

• We have improved spotted DNA uniformity by developing microfabricated substrates that constrain deposition to micron-sized openings.

• Comparison with commercial glass microarrays showed that our substrates have comparable sensitivity and improved reproducibility between replicates on a single slide and across slides.

Figure 1, left: Fabrication, DNA patterning and hybridization for polymer lift-off microarrays.

Figure 2, below: Fluorescently-labeled oligonucleotides observed with confocal microscopy: a, b) Commercial glass microarrays with most of the signal on the outer rim; c, d) Our microfabricated arrays with uniformly spread signal.
BIOLOGICAL APPLICATIONS

Development of Microfluidic Biosensor Devices Based on Liposome Amplification Strategies

CNF Project # 802-99
Principal Investigator: Antje J. Baeumner

Abstract:

Bioanalytical microsystems for the detection of viable pathogenic organisms are being developed. Pathogenic organisms such as *B. anthracis*, *C. parvum*, *Dengue* virus and *E. coli* are identified via specific mRNA molecules or their genomic RNA (in the case of the virus) with detection limits between 5 and 40 cells/mL. The microsystems consist of modules in which bioanalytical operations are being performed. These modules are, for example, a laser-induced cell lysis system, an RNA purification system, a nucleic acid sequence-base amplification module and a biosensor component for the detection and quantification of the RNA molecules. In addition, research is being performed on the design of mixers, micro-heaters and electrochemical transducers.

In the last year, the main work focused on the development of an efficient passive microfluidic mixer. It takes advantage of a fluid-exchange principle in which the device provides order-changing functions to a microfluid, i.e. allowing sections of fluid separated by a length of the channel to directly interact. The device effectively “folds” the solution to permit streamlines that are normally linearly separated to come into contact.

Summary:

A microfluidic device capable of recirculating nano to microliter volumes in order to efficiently mix solutions was developed. The device was fabricated from a silicon master in poly(dimethylsiloxane) (PDMS) channels with pressure inlet and outlet holes sealed by a glass lid. Recirculation was accomplished by a repeatedly reciprocated flow over an iterated sawtooth structure. The sawtooth structure served to change the fluid velocity of individual streamlines differently dependent on whether the fluid is flowing backward or forward over the structure. Thus, individual streamlines can be accelerated or decelerated relative to the other streamlines to allow sections of the fluid to interact that would normally be linearly separated. Low Reynolds numbers imply that the process is reversible, neglecting diffusion. Computer simulations were carried out using FLUENT®.

Subsequently, fluorescent indicators were employed to experimentally verify these numerical simulations of the recirculation principal. Finally, mixing of a carboxyfluorescein-labeled DMSO plug with an unlabeled DMSO plug across an immiscible hydrocarbon plug was investigated.

At cycling rates of 1 Hz across five sawtooth units, the time was recorded to reach steady state in the channels, i.e. until both DMSO plugs had the same fluorescence intensity. In the case of the sawtooth structures, steady state was reached five times faster than in channels without sawtooth structures, which verified what would be expected based on numerical simulations.

The microfluidic mixer is unique due to its versatility with respect to scaling, its potential to also mix solutions containing small particles such as beads and cells, and its ease of fabrication and use.

References:

Development of Microfluidic Biosensor Devices Based on Liposome Amplification Strategies

CNF Project # 802-99
Principal Investigator: Antje J. Baeumner
Users: Kevin Nichols, Sam Nugen

Affiliation: Dept. of Biological & Environmental Engineering, Cornell University
Primary Funding: Hatch Project NYS123-404
Contact: ajb23@cornell.edu, kevin.paul.nichols@gmail.com, sm6@cornell.edu

Microfluidic mixer as part of a bioanalytical microsystem for pathogen detection:

- Passive microfluidic mixer using a sawtooth structure repeated throughout the channel length.
- Reciprocated flow pattern.
- Recirculation of solution proven experimentally.
- An ideal mixer for enzymatic and biosensor reactions in long channel structures since fluid elements far apart will come into close contact.

Figure 1: FLUENT® modeling of flow pattern in microfluidic mixer for flow left to right (above) and right to left (below).

Figure 2: Photograph and illustration of four separate volumetric elements recirculated next to each other. Four regions of clearly varying concentration are visible in the photograph (a) and illustrated below (b).
Microfluidic Flow Cytometer for Detection and Quantification of Viral Antibodies

CNF Project # 808-99
Principal Investigator: Joel Tabb

Abstract:

Immunoassays performed on solid supports are familiar to most bioscience professionals and the technology is routinely used in a variety of laboratory settings. Such assays are most commonly realized in the form of the 96-well microliter plate ELISA. One major limitation of the format is that it is not amenable to simultaneous analysis of multiple antigens and thus is both labor and sample intensive. A second limitation is that the assay is not enclosed and must be handled with additional care when analyzing potentially pathogenic material, as is often the case in a clinical setting.

Microsphere technology, in conjunction with flow cytometry, has been developed to overcome the first obstacle. Microspheres, conjugated to different ligands and identified with unique fluorescent labels, can be exploited in development of multiplex assays. The net result is faster analysis time and increased data output with minimal sample requirements. Despite these advantages, flow cytometry requires sample aerosolization that can lead to potentially biohazardous conditions when working with pathogenic materials. The utility of multiplex microsphere-based immunoassays in clinical settings is recognized, but the majority of effort has been toward demonstrating applicability for research purposes.

Microfabrication design and microfluidic instrumentation have arisen as an extension of the technology used in the electronics industry for building integrated circuits. Using photolithographic and related techniques, fluid networks and pumping systems can be built to accommodate low microliter volumes with sensitivity comparable to, or better than that associated with conventional analytical systems. One area of interest is the development of microfluidic flow cytometers to enable cell counting of small samples that could not be achieved in traditional units.

Summary:

Molds for the microfluidic channels were fabricated using SU-8 (25) photoresist to achieve the required thicknesses on standard silicon wafers. Poly (Dimethyl Siloxane) (PDMS) was cast onto the patterned silicon wafers to fabricate the fluidic channels to a thickness of 3 mm. Holes were cut into the PDMS channels taken from the molds to accommodate fluid interconnects and subsequently bonded to glass slides. Small bore Teflon® tubing was used for the fluidic connections to the PDMS cytometer. Using electronics developed in-house at Agave BioSystems, the modules were tested using fluorescent microspheres.

Data was taken and presented in standard flow cytometry format.
Microfluidic Flow Cytometer for Detection and Quantification of Viral Antibodies

CNF Project # 808-99
Principal Investigator: Joel Tabb
User: Christopher Chase

Affiliation: Agave BioSystems, Ithaca, NY
Primary Funding: National Cancer Institute
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Web Site: www.agavebio.com

Figure 1, top left: Microfluidic device mold with raised photoresist features (SU-8 25) on a silicon wafer substrate. The mold allows for 8 devices to be cast at once.

Figure 2, bottom left: Microfluidic flow cytometer module complete with fluid connections.

Figure 3, above: Fluorescent output data, presented as a dot plot, for green and red microspheres taken from the microfluidic flow cytometry module. Green and red 7.2 µm microspheres were mixed and flowed through a chip and were detected using a 488 nm blue Ar-Ion laser. PMT1 represents the green channel and PMT2 represents the red channel.
Microfabricated Electrode Arrays to Study Exocytosis

CNF Project # 848-00
Principal Investigator: Manfred Lindau

Abstract:

Neurons release neurotransmitters in a process called exocytosis, wherein membrane bound packets of transmitter molecules are released from the cell upon stimulation. The mechanics of this process are poorly understood, but are known to involve three proteins collectively called the SNARE complex. Chromaffin cells of the adrenal gland share this release mechanism, and thus constitute a model system for neuronal release. Quantal release adrenaline from chromaffin cells can be measured using the electrochemical technique of amperometry, historically employing a carbon fiber electrode.

To gain information about the exocytotic mechanism, and the release and diffusion kinetics of catecholamine during exocytosis, we have developed a method of electrochemical imaging employing microfabricated devices based on amperometry. We have successfully measured electrochemical events using Platinum, Gold, and transparent Indium-Tin-Oxide (ITO) electrodes. By comparing the experimental electrochemical signals with signals generated by Monte Carlo simulations, we can gain information about the release and diffusion kinetics of catecholamine during exocytosis. These devices have been combined with fluorescence imaging, yielding more information about the exocytotic process, and fast scan cyclic voltammetry (FSCV) which permits identification of some neurotransmitters. We are also working towards manipulating cells with the electrode arrays using Dielectrophoresis (DEP).

Summary:

Several important cell types, including neurons and chromaffin cells of the adrenal gland, release transmitter molecules via exocytosis—the release of membrane bound packets or “vesicles” of transmitter molecules. In amperometry, exocytosis is observed by electrochemical detection, usually employing a carbon fiber electrode [1]. Transmitter molecules released during an exocytotic event are oxidized by a nearby electrode, and the oxidation current is recorded.

We have fabricated electrochemical detector (ECD) arrays of four Pt, Au, or ITO electrodes on microscope coverglass to perform simultaneous amperometry by four electrodes at four different positions. The resulting current traces reveal the time course, quantal size, and position of vesicle fusion on the cell membrane. The average quantal size in individual cells on Pt electrodes was 0.5 pC to 3.5 pC, as expected for chromaffin vesicles [2]. The charge measured by the individual electrodes depends on the release position on the cell surface, allowing the release location to be pinpointed.

Each ECD is fabricated using 405 nm photolithography and lift off (Pt, Au) or etching (ITO) techniques. The ECD circuit consists of four wires, insulated by glass or photoresist, where the tips of the wires are exposed at the detector site, located at the corners of an ~ 12 x 12 μm square. The ECD is also coupled to an inverted fluorescence microscope, allowing us to observe the locations of release events both electrically and via fluorescence imaging. The positions of the release events determined from the ECD were in good agreement with the fluorescence locations [2].

Recently, we successfully performed amperometry using ITO ECDs. This gives us the ability to see what is directly above an electrode, via fluorescence if desired, and may be useful in studying transmitter release in neurons. The Au ECDs have also been successfully used for FSCV, where rapid oxidation and reduction of transmitter molecules results in a redox “fingerprint” that can be used to identify certain types of transmitter molecules [1].

A major drawback of the ECDs has been the need to place cells atop the ECDs “by hand” using a micropipette. This is tedious and time consuming. We are testing methods to position cells using Dielectrophoresis (DEP). DEP is the process by which a non-uniform electric field induces a dipole in a particle (a cell in our case), causing it to move.

References:

Microfabricated Electrode Arrays to Study Exocytosis

CNF Project # 848-00
Principal Investigator: Manfred Lindau
Users: Kassandra Kisler Elliott, Khajak Berberian, Brian Kim
Affiliation: School of Applied and Engineering Physics, Cornell University
Primary Funding: Nanobiotechnology Center, NIH
Contact: ml95@cornell.edu, kjk29@cornell.edu, knb6@cornell.edu

- Study of neurotransmitter release mechanism using microfabricated electrode arrays (ECDs) and fluorescence.
- Possible to localize events using electrochemical information.
- Transparent electrodes allow observation of fluorescence through electrodes while recording electrochemical information.
- Fast cyclic voltammetry for identification of transmitter molecules.
- Dielectrophoretic (DEP) force to move and trap cells.

Figure 1, top left: 3- and 4-electrode Platinum ECDs.

Figure 2, bottom left: Transparent electrodes (white outline) with photoresist insulation (black outline).

Figure 3, above: A 9.6 µm polystyrene bead trapped between ECD electrodes using DEP.
On-Chip Bead Array for Bio-Analysis

CNF Project # 854-00
Principal Investigator: Hui Huang, Ph.D.

Abstract:
Prototype devices have been fabricated and optimized for multiplex DNA and protein analysis.

Summary:
BioArray Solutions has developed a proprietary BeadChip™ format that combines semiconductor technology, microparticle chemistry and molecular biology to bring unparalleled flexibility and performance to quantitative DNA and protein analysis.

To produce our functional chips, color-coded microparticles (“beads”) are assembled into a silicon chip and immobilized to form a planar array in a designated area. Our continuous effort at CNF has resulted in significantly improved chip design and fabrication process to ensure high quality chips.

The BeadChip™ products have been successfully used for human leukocyte antigen (HLA) molecular typing via hybridization-mediated multiplexed analysis.

References:
On-Chip Bead Array for Bio-Analysis

CNF Project # 854-00
Principal Investigator: Hui Huang, Ph.D.
Users: Hui Huang, Ph.D.; Yi Zhang, Ph.D.
Affiliation: BioArray Solutions. Ltd., 35 Technology Drive, Warren, NJ 07059
Primary Funding: Private Funding
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Web Site: www.bioarrays.com

Figure 1: Image of green fluorescent dye coded bead array on chip.

Figure 2: Cy3 fluorescent image of the array after hybridization with fluorescent dye tagged DNA targets.
A Microfabricated Polymerase Chain Reaction-Based Biosensor

CNF Project # 884-00
Principal Investigator: Dr. Carl A. Batt

Abstract:
A portable, fully-automated, PCR-based detection system has been developed for the rapid detection of bacterial pathogens and forensic DNA analysis. Microfabricated DNA purification and real-time PCR microchips were fabricated and tested for their ability to purify and detect DNA sequences from a variety of bacteria including *Bacillus anthracis* (Anthrax). Chips were also tested for their ability to purify and detect single nucleotide polymorphisms (SNPs) for forensic DNA identification.

To extend the utility of the system, we are currently developing a multiplex bead-based DNA detection module. This module will allow for the rapid identification of up to 20-30 different SNPs, or an equivalent number of target pathogens. DNA purification and PCR amplification would be performed in the previously developed microchip, followed by hybridization of PCR products to multi-colored fluorescent microbeads. Beads will then be analyzed with a miniaturized, microfluidic fluorescent particle counter that we are developing. This particle counter has been constructed in polydimethyl siloxane (PDMS) using photolithographically patterned SU-8 molds. The counter uses five fluid inputs to focus a central fluid stream using three-dimensional sheath flow. We have successfully focused fluids to a 10 µm by 10 µm fluid stream using this system. Furthermore, we have integrated this flow cell with fiber optics and a molded wave guide to excite a 20 µm region of the fluid. This region is also fiber optically coupled to a fluorescence detection system that is capable of counting particles that pass by the excitation region.

Using this system, we have been able to observe and count multiple colors of 10 µm diameter fluorescent beads, demonstrating the potential for bead-based DNA analysis.

Summary:
We have developed a portable DNA purification and PCR-based detection system for bacterial pathogens and human DNA targets. Current work has focused on the development of a fluorescent bead-based detection system for multiplex detection of PCR-amplified DNA.

References:

A Microfabricated Polymerase Chain Reaction-Based Biosensor

CNF Project # 884-00
Principal Investigator: Dr. Carl A. Batt
Users: Nathaniel C. Cady, Scott J. Stelick, Matthew Kennedy, Clarissa Lui
Affiliation: Dept. of Microbiology, Cornell University
Primary Funding: USDA, FDA, National Institute of Justice
Contact: cab10@cornell.edu, ncc4@cornell.edu
Web Site: http://foodmicro.foodsci.cornell.edu/fmlab/personnel/cady.htm

- A device capable of detecting a fluorescence microsphere in a microfluidic channel.
- Sample flow is confined in the center of the channel by having the sheath flow in the vertical and horizontal direction.
- Includes an integrated waveguide which confines the excitation region to 20 µm.

Figure 1, left: The fluorescent signal from a 10 µm fluorescent microsphere in a 125 µm wide and 125 µm high microfluidic channel.

Figure 2, below: Detection of green and orange fluorescent microbeads in microfluidic channel. Fluorescence intensity (arbitrary units) are shown on the Y axis with time (seconds) shown on the X axis.
A Microfabricated Magnetic-Bead-Based Micromixer

CNF Project # 884-00
Principal Investigator: Dr. Carl A. Batt

Abstract:

For the purposes of rapid detection of bacterial pathogens and forensic DNA analysis, a portable, fully-automated, PCR-based detection system has been developed in our lab. To further broaden the utility of this system, we are developing a sample preparation module, which would remove cells of interest from a given sample by mixing the raw sample with magnetic beads coated with specific antibodies. The captured cells are mixed with lysis buffer to release DNA and other cell contents, and the resulting lysate is fed into the DNA purification microchip, while the magnetic beads are left captured in the mixing chamber. A thin piezoelectric diaphragm, actuating at its resonant frequency of 0.6 ± 0.5 kHz, is used to produce vibrations to agitate 2.8-µm-sized magnetic beads inside the microfluidic chamber.

The microfluidic mixer in our experiments is made up of three separate layers. The top layer of microfluidic channels and chambers used in our mixers were fabricated from poly(dimethylsiloxane) (PDMS) by casting the silicon rubber on a wafer with SU-8 structures. The chambers were patterned using a 150 µm layer of negative photoresist (SU-8). The middle layer is a flat slab of PDMS of controllable height, cast on a petri dish, with a circular cutout of 7 mm in diameter. The bottom layer, which is the layer in contact with the piezoelectric diaphragm, is a thin PDMS spin-coat membrane layer. The three layers are separately plasma-treated before aligning and adhering them together.

The final device, with all the input and output tubing inserted, is placed on the piezoelectric diaphragm with the PDMS membrane layer facing down. The fluid volume of the current microfluidic devices is approximately 100 microliters, but is easily tunable by changing the height of the middle layer. The evaluation of mixing is determined by observing the color distribution of the beads in solution and also by the real-time PCR measurements of DNA concentration from the collected lysate.

Summary:

A portable DNA purification and PCR-based detection system for bacterial pathogens and human DNA targets has already been developed. Current work is focused on adding a magnetic bead-based sample preparation system for cell capture and cell lysis purposes.

References:


A Microfabricated Magnetic-Bead-Based Micromixer

CNF Project # 884-00
Principal Investigator: Dr. Carl A. Batt
Users: Clarissa Lui, Nathaniel C. Cady, Scott J. Stelick, Matthew Kennedy

Affiliation: Dept. of Microbiology, Cornell University
Primary Funding: USDA, FDA, National Institute of Justice
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Web Site: http://foodmicro.foodsci.cornell.edu/fmlab/personnel/Clarissa%20Lui.html

- Portable DNA purification and PCR-based detection system for bacterial pathogens and human DNA targets.
- Current work is focused on the development of a sample preparation system for cell capture and cell lysis purposes.

Figure 1: A schematic of the microfabricated PDMS mixer device.
Bacterial Colonization and Communication in a Microfabricated Fluidic Channel

CNF Project # 935-01
Principal Investigator: Harvey Hoch

Abstract:

The non-flagellated plant pathogenic bacterium, *Xylella fastidiosa*, migrates ‘upstream’ in a microfluidic device through twitching motility, which is mediated by 1.0-5.6 µm long hair-like type IV pili. Another kind of pili (type I pili) are 0.4-1.0 µm in length and are involved predominately in biofilm development. As observed in our microfluidic chambers, both pili types are likely involved in a sudden ‘autoaggregation’ of dispersed bacterial cells into compact masses of bacteria.

Summary:

Previously, using microfluidic chambers it was discovered that the plant pathogenic bacterium, *Xylella fastidiosa*, which lives exclusively in the water conducting xylem vessels of plants, is able to migrate long distances against the flow of the sap stream via type IV pilus-mediated twitching motility [1]. The fact that migration of *X. fastidiosa* is directionally controlled, viz., against rapid flowing currents of growth medium, is particularly interesting and unique. Such directional migration of non-flagellated bacteria helps explain how *X. fastidiosa* might colonize xylem vessels (and stems) upstream from initial sites of infection.

Now, we have further discovered, using our microfluidic chambers, that these bacteria ‘autoaggregate’ from a scattered population of bacteria into compact masses of bacteria. Such aggregation of the dispersed bacteria is potentially very important in explaining how xylem vessels seemingly appear to become ‘plugged’ with cell masses over a short period of time—a period over which disease symptoms also abruptly appear. Neither the aggregation nor the twitching motility phenomena associated with these bacteria would have been observed had we not used microfluidic chambers fabricated to mimic plant xylem vessels.

We now have many different strains and mutants of the bacteria, and wish to compare two or more types at the same time, under the same conditions. We are currently using a chamber that allows both side-by-side chambers (each 80 µm wide, separated by 25 µm of PDMS) to be viewed at the same time using time-lapse imaging.

References:

Bacterial Colonization and Communication in a Microfabricated Fluidic Channel

CNF Project # 935-01
Principal Investigator: Harvey Hoch
Users: Leonardo De La Fuente, Tanya Taylor, Yizhi Meng

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http://www.nysaes.cornell.edu/pp/faculty/hoch/agg/

Figure 1, above: Microscope-microfluidic chamber configuration, construction and chamber design.

Figure 2, middle: Autoaggregation in 9-day old culture, first noted as dispersed cells aggregated into developing cell mass. Cells continued to be attracted to the developing aggregate (arrowheads). Times, h:min. after first recording sequence. http://www.nysaes.cornell.edu/pp/faculty/hoch/agg/

Figure 3, right: Autoaggregation in 11-day old culture. Densely dispersed cells aggregate within minutes. Such aggregates (*) merged with other aggregates (1, 2, 3). h:min.
Equilibrium Dialysis Systems for Selective Molecular Filtration

CNF Project # 937-01
Principal Investigators: James N. Turner, David Martin, Michele Caggana, Michael Spencer

Abstract:

We are developing a family of on-chip membranes to perform a variety of molecular separations [1-4]. Membranes are cast on wafers using conventional spinning methods from solutions of cellulose acetate (CA) with concentrations varying from 10-40%. Substrates are prepared by depositing silicon nitride on silicon wafers and patterning the nitride with multi-micron size holes using reactive ion etching. The silicon nitride is relieved from the back-side producing 1 x 1 mm squares of patterned nitride that act as a frit for the CA membranes.

The membranes are formed from the spun solution by phase inversion in water under a variety of conditions that influence the membrane’s rejection properties. To a first order approximation, the membranes reject molecules based on their molecular weight. However, molecular characteristics such as shape, charge and hydrophobicity also affect molecular transport across the membranes. Positively charged molecules tend to bind throughout the membrane volume. DNA, a high molecular weight but linear molecule, can be electrophoresed through the membrane while heme, a much lower molecular weight but planar molecule, with the same electrophoretic mobility is blocked [3].

We have produced a family of membranes whose rejection properties are dependent on the CA concentration, and the quenching conditions, particularly time and water bath temperature. Membranes have been fabricated with approximate molecular weight cutoffs of 300, 350, 600 and 700 Da. All the membranes have similar structure when observed by SEM after freeze-fracturing to reveal the internal features. Both surfaces have a thin skin but the top one is thicker and more prominent. The internal structure is extremely complex and varies in density across the thickness. The internal matrix also has a large number of small voids. Some preparations have very large voids that are not part of the fluid path through the membranes. Confocal microscopy showed that when the membrane is imaged in a solution of a fluorescent molecule, the large voids are unlabelled indicating that they do not communicate with the fluid path through the membrane. The matrix is strongly fluorescent indicating that the fluid path is very complex and long [4].

It is not known what the actual rejection structure is or whether it is throughout the membrane or if the skin dominates it. Transport is dependent on membrane thickness indicating a complex transport mechanism.

Summary:

A family of on-chip membranes have been developed for the separation of small molecules and for the purification of large linear biomolecules, e.g. DNA, from small molecules especially ones of similar electrophoretic mobility.

References:


Equilibrium Dialysis Systems for Selective Molecular Filtration

CNF Project # 937-01

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- On-chip molecular separation.
- Sharp molecular cutoffs.
- Purification of biomolecules.

Figure 1, top left: SEM of a freeze-fractured cross-section of a 10% CA membrane with a molecular weight cutoff of ~ 600 Da. The membrane is composed of a thin skin on the top surface with a dense matrix that has an increasing number of small voids as a function of distance. Some large voids are present but have been shown not to contribute to the molecular transport.

Figure 2, bottom left: SEM of a freeze-fractured cross-section of a 40% CA membrane with a molecular weight cutoff of ~ 300 Da. The membrane structure is similar to the one in Fig. 1 except there are more large voids that again have been shown not to influence molecular transport.

Figure 3, above: Shows schematically the family of membrane cutoffs that have been developed.
Multidimensional Microscale Separations of Cerebrospinal Fluid Proteins

CNF Project # 942-01
Principal Investigator: Kelvin H. Lee

Abstract:

The long-term objective of this project is to develop microfabricated devices capable of performing automated cerebrospinal fluid (CSF) protein analysis for reliable antemortem diagnosis. Such devices would be useful in the clinical setting and be very useful in the identification of molecular markers for disease.

We have been using separation affinity and isoelectric focusing (IEF) based separations of cerebrospinal fluid proteins. We have integrated both IEF and reverse phase separations with electrospray ionization. The integrated device has been able to separate and analyze CSF proteins.

Summary:

It is important to use separations as a front end to mass spectrometry (MS) in the industrial proteomics community [1,2]. In cerebrospinal fluid, there is an abundance of albumin (60-80% of the total protein content), which is usually not related to disease. Therefore, the possibility of using affinity-based separations for prefracionation (removal of albumin) of cerebrospinal fluid proteins using microfluidic devices has significant potential. We have previously separated CSF proteins using affinity separations and isoelectric focusing [3].

More recently, we have demonstrated the ability to separate proteins using reverse phase separations (also known as solid phase extraction) coupled to electrospray ionization [4,5]. Using these new methods, we have been able to identify proteins in CSF that had not been previously identified using other more traditional (larger length scale) proteomics separations technology including collagen alpha 3 type VI.

References:


**Multidimensional Microscale Separations of Cerebrospinal Fluid Proteins**

**CNF Project # 942-01**
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![Figure 1: Microscale isoelectric focusing with direct electrospray ionization of a mixture of beta lactoglobulin A and myoglobin.](image)

*Figure 1: Microscale isoelectric focusing with direct electrospray ionization of a mixture of beta lactoglobulin A and myoglobin.*
Electrolytic Bubble-Based Microfluidic Pressure Sensor

CNF Project # 990-01
Principal Investigator: Susan Z. Hua

Abstract:
The development of microfluidic systems has advanced the technology for chemical detection, combinatorial synthesis and bioassay. Integration of high density functional fluidic components onto a single chip is the key to success for many applications. Utilizing an electrolytic gas bubble-based actuation mechanism, we have developed a series of microfluidic actuation components including valves, pumps, bi-directional switches, and multiplexers. Recently, we have further extended the bubble-based technology to pressure sensing within microfluidic channels.

A series of electrolytic bubble sensors were integrated into a bubble-based actuation fluid distribution system to measure the pressure difference at various stages of a multiple channel network in real-time. The pressure dependence of the measured resistance was systematically investigated by applying an inlet pressure ranging from 101 kPa to 115 kPa.

Integration of bubble-based sensors with actuators into a complex system demonstrates the capability of forming high-density functional microfluidic system using the same electrolytic principle and with ease of microfabrication.

Summary:
The development of new microfluidic based sensing and actuation schemes are essential towards increasing the functionality and diversity of lab-on-a-chip technologies. In a fluidic system, pressure measurements have been made using piezoresistors, or by measuring the electrostatic capacitance or resistance change caused by the displacement of a diaphragm. These sensing techniques usually involve complicated fabrication processes and are difficult to shrink below a few hundred micrometers whereas the microfluidic channels are typically less than hundred micrometers wide. Utilizing an electrolytic gas bubble based actuation mechanism, we have developed a series of microfluidic actuation components, such as valves, pumps, and multiplexers. Recently, we have further extended the bubble-based technology to pressure sensing in an open microfluidic channel to measure the pressure in a functional fluidic chip.

A prototype bubble sensor is designed and microfabricated on silicon chip. An electrolytic bubble sensor was placed in a parallel channel connected with the main flow channel to provide the measurements of open channel pressures in real-time. Since the gas bubbles are non-conductive, the electrical impedance of a region containing a bubble is a function of its relative volume. The bubble size has been shown to directly reflect the ambient pressures in microfluidic channels according to two sensing modalities, (1) measurement of the resistance corresponding to the initial bubble size, and (2) measurement of the bubble dissolving rate [1].

To measure bubble sensor response to varied pressures in the main flow channel, we applied various inlet pressures to the inlet reservoir and generated steady flow in the main channel with a different flow rate. The bubble sensor response was measured under each inlet pressure ranging from 101 kPa to 115 kPa, while keeping the outlet pressure at atmosphere.

The results show that open flow pressure can be accurately measured using a bubble sensor located in an adjacent sensing channel [2]. The bubble-shrinking rate serves as a measurable parameter for the pressure in main fluid channel. The higher the pressure, the faster the bubble shrinks. In a hydrophobic fluid channel, the sensor usually maintains at high resistance stage during bubble shrinkage until the bubble collapses. The time required for the bubble to collapse is a sensitive measure of channel pressure. We defined this time length as a characteristic time that indicates the local open channel pressure. Our results show that the critical time decays as the inlet pressure increases.

References:
Electrolytic Bubble-Based Microfluidic Pressure Sensor

CNF Project # 990-01
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Figure 1: Left Panel: Schematic and a microfabricated pressure sensor based on electrolytic gas bubbles. Right: An integrated microfluidic chip incorporating pressure sensors and bubble-based fluid actuators.

Figure 2.: Characteristics of bubble sensor response for four different inlet pressures. The duration that bubble stays at high resistance state is a good indicator for the pressure at the point of the interest in main flow channel.

Figure 3: Correspondence of critical time to open channel pressure.
Biosensor Fabrication using Patterned Surfaces

CNF Project # I002-01

Principal Investigators: James N. Turner, David Lawrence, Michael Spencer

Abstract:

We have been developing a three-electrode biosensor to capture and quantify CD4+ cells from human blood. The device is based on electrochemical detection and is being developed to monitor the immune status of AIDS patients [1].

Gold electrodes were patterned on glass wafers using standard lithographic and evaporation methods. The electrode leads were insulated by patterning positive tone polyimide that was opened to expose the electrodes and contact pads. A final cure of the patterned polyimide was performed in a YES 450PB vacuum oven for 8 hrs. The working electrode of our initial device was small, and functionalized using protein-G as a linker to bind the antibodies that captured the cells. The device was useful for quantifying a small number of captured cells and for measuring the electrical properties of individual cells [1]. However, we require improved cell capture and the quantization of a larger numbers of cells (at least 200).

Improved antibody functionalization of the working electrode was achieved by covalently binding the anti-CD4 antibodies to the gold [2]. The latter method of binding resulted in substantially more antibody per unit area. To measure larger numbers of cells requires a larger working electrode which is generally not practical as the associated reference electrode has to be extremely large. Platinization of the reference electrode substantially increased its surface area making it appear electrically as a very large electrode without increasing its footprint. This improved the electrical characteristics of the sensor allowing the use of large (up to 300 x 300 µm) working electrodes.

The biosensor was tested using the J-45 cell culture model system that mimics the behavior of human CD4 blood cells. Approximately 400 J-45 cells were captured on a 300 x 300 µm working electrode and were easily detected by a change in biosensor impedance. Plots of impedance vs. frequency showed that the resistive component was very large and still increasing at very low frequency, arguing that d.c. methods of detection would be difficult to implement. On the other hand, impedances in the frequency range up to ~ 1,000 Hz showed substantial changes associated with easily detected currents. This argues that the capacitance of the system changes more rapidly as cells bind to the biosensor indicating that an a.c. impedance as opposed to an amperimetric detection system would be easier to implement.

Summary:

An on-chip three-electrode bio-impedance sensing device to rapidly detect and quantify several hundred cells was fabricated and is being characterized. Platinization provided an electrically large reference electrode and a few to several hundred cells were detected on a large working electrode.

References:


Biosensor Fabrication using Patterned Surfaces

CNF Project # 1002-01
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- An antibody based biosensor for monitoring CD4⁺ cells.
- Potentially useful for monitoring AIDS patients.
- Cells are detected by changes in the sensor’s electrical impedance.

Figure 1: Three electrode biosensor (left) on a glass wafer composed of a working microelectrode (WE), reference electrode (RE), and a counter electrode (CE). Images of human CD4⁺ cells labeled for CD4 antigen (right-top) and for nuclei (right-bottom) demonstrating that all captured cells are CD4⁺.

Figure 2: Impedance vs frequency plot showing the performance of the platinized reference electrode and the capture of several hundred CD4⁺ J-45 cells.
Silicon Based Ultrasonic Microprobes for Cardiac Signal Recording

CNF Project # 1122-03
Principal Investigator: Amit Lal

Abstract:
Three dimensional mapping of physioelectrical activities in \textit{in vitro} cardiac tissues is highly desirable for the study of the mechanism and development of cardiac arrhythmias. Important information can be obtained for prediction and prevention of arrhythmias, especially ventricular arrhythmia—one of the leading causes of mortality in the United States. In this project, we design and manufacture ultrasonically actuated silicon microprobes for multi-site potential recording from inside the heart, while the ultrasonic actuation reduces insertion force and minimizes tissue damage. Probe tips with multiple recording sites have been successfully inserted into canine heart tissue and cardiac signals were recorded under different conditions. The penetration force—maximum force encountered by the microprobe during insertion into the tissue—on different biological tissues were studied and its dependence on ultrasonic driving and probe dimension was analyzed. These results illustrate not only the encouraging prospect of 3D electrical recording for cardiac arrhythmia studies, but also the novel application of vibrational energy at the micro-scale.

Fabrication:
The device consisted of a silicon ultrasonic horn actuator \cite{1} with a longitudinal $\lambda/2$ resonance at 75 kHz. Two thin beam tips were defined at the small end of the horn to be driven longitudinally. The thickness of the tips (ranging from 70 µm to 140 µm) was defined by DRIE on the front side and the probe was released by wet etching from the backside. The tip length varied from 5 mm to 1 cm for penetration of a heart wall of different thickness. A dummy probe without tip was bonded to the probe for symmetry and to reduce bending mode of the tips. PZT4 plates were bonded to the device for ultrasonic driving. The device is clamped to a customized PC board at resonance node. The PC board also provides metal pads for electrical connection from the Pt/Cr electrodes on the probe through wire bonding. A ground layer was also patterned on top of the conduction paths to reduce cross-talking between channels.

Results:
Cardiac signal recording was conducted on an isolated and perfused canine heart. Recordings were obtained from isolated perfused canine heart during pacing, following the induction of ventricular tachycardia, and during the transition from ventricular tachycardia to ventricular fibrillation. Local conduction velocity of 0.60±0.03 m/s was observed from the multichannel recordings from the canine right ventricular wall under epicardial pacing. Signals from different recording sites were compared and phase/morphology difference can be used for later reconstruction of physioelectrical wave propagation in the heart. Furthermore, signals recorded with/without the presence of ultrasound showed little difference other than some easily filtered high frequency noise, indicating the low voltage ultrasonic driving posed no significant modification on heart cells’ electrical activities.

Penetration and cutting force measurements show that both forces reduced as ultrasonic driving voltage increased. The penetration force—the maximum force encountered by the probe during penetration—is found to reduce with increasing ultrasonic driving voltage, on both excised canine right ventricular muscle and chicken breast muscle. The rate of force decrease varies with tissue type and microprobe dimension. Laser Doppler interferometer measurements showed that flexural vibration modes are excited in the silicon microprobe by longitudinal ultrasonic actuation and may contribute to the force reduction. With ultrasonic actuation, the silicon microprobes are inserted into an isolated perfused canine heart without breakage or significant buckling, under 10 Vpp actuating voltage.

References:
Silicon Based Ultrasonic Microprobes for Cardiac Signal Recording

CNF Project # 1122-03
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Figure 1, top left: Multi-channel recording with silicon microprobes integrated with ultrasonic horn actuator.

Figure 2, bottom left: Flexural vibration mode excited in silicon microprobe by longitudinal ultrasonic actuation.

Figure 3, above: Penetration force on canine cardiac tissue and chicken breast tissue decreases with increasing ultrasonic driving.
Development of Model Neural Prostheses with Integrated Microfluidics for Drug Delivery

CNF Project # 1214-04
Principal Investigators: James N. Turner, William Shain, Christopher Ober

Abstract:

A number of chronic brain diseases are increasingly being treated by the implantation of electrically active probes into specific brain regions. Our goal is to develop more tissue compatible miniaturized probes improving probe function and controlling any adverse effects of probe implantation [1].

Optimization of tissue compatibility requires locally delivered drug therapy that can be altered as a function of time after implantation. We are exploring two approaches: short-term delivery immediately after implantation via time-release from drug-containing polymers, and long-term administration via microfluidics [2]. Advantages of microfluidics are that the drug cocktail can be changed from outside the body, and that the local tissue drug concentration can be maintained at biologically active and controlled levels. Preliminary studies using microfluidic channels with multiple outlets on probes designed for implantation into the cortex of rat brains showed that the diffusion of small molecules from the outlet was faster than the diffusion-driven molecular transport along the channel. This resulted in a pulse of drug release followed by a rapid decrease in injected drug concentration.

Two approaches are being evaluated to maintain drug injection over time. The first is to pressure inject the drug solution using the original dead-ended channels [2]. A second approach is to use lower pressure to circulate flow through microfluidic channels such that diffusion through the outlets is the primary delivery mechanism. The continually circulating flow constantly supplies fresh solution at a constant concentration to the outlets. This is expected to maintain tissue drug concentration using low-pressures with reservoirs external to the tissue being treated. The drug concentration at the outlets can also be maintained at any desired concentration thereby maintaining tissue drug level concentrations.

Model neural probes were fabricated using a DRIE process [2] on both sides of silicon substrate to form the implant. Surface micromachining [3,4] were integrated with bulk subtractive techniques to create free standing, three dimensional silicon structures with integrated microfluidics [5].

Summary:

The successful use of brain implants to treat chronic diseases requires stable two-way electrical communication between neurons, the electrically communicating cells of the brain, and the implant. This level of biocompatibility can likely only be achieved with continuous pharmacologic intervention that is changed as a function of time. Thus, incorporating drug delivery systems into our brain implants is critical. The U-channel design has advantages in terms of changing the drug regime and for delivering controlled high concentrations of drug.

References:


Development of Model Neural Prostheses with Integrated Microfluidics for Drug Delivery

CNF Project # 1214-04
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Figure 1, above: The U-Channel device will allow fluid to be passively cycled through the implanted section of the microfluidic channels. Diluted fluid will evaporate out of the exit port as higher concentration fluid is drawn through the inlet. Material in the implanted region of the channel will remain at a relatively high concentration resulting in a constant diffusion source.

Figure 2, right: Scanning electron micrographs of U-Channel probes fabricated using conventional surface micromachining and Deep Reactive Ion Etching. Alkali Hydroxide etching was used to make channels deeper and facilitate both active and passive cycling of fluid through the devices during implantation.

Figure 3
Arraying of Intact Liposomes on Chemically Functionalized Microwell Surfaces for Application as Membrane Protein Based Biosensors

CNF Project # I235-04
Principal Investigators: Alexander Couzis, Charles Maldarelli, Lane Gilchrist

Abstract:
We have developed a protocol to bind individual, intact phospholipid bilayer liposomes, which are on the order of 1 µm in diameter, in microwells etched in a regular array on a silicon dioxide substrate. The diameter of the wells is on the order of the liposome diameter, so only one liposome is located in each well. The background of the silicon dioxide surface is functionalized with a PEG oligomer using the contact printing of a PEG silane to present a surface that resists the adsorption of proteins, lipid material, and liposomes. The interiors of the wells are functionalized with an aminosilane to facilitate the conjugation of biotin, which is then bound to Neutravidin. The avidin-coated well interiors bind the liposomes whose surfaces contain biotinylated lipids. The specific binding of the liposomes to the surface using the biotin-avidin linkage, together with the resistant nature of the background and the physical confinement of the wells, allows the liposomes to remain intact and to not unravel, rupture, and fuse onto the surface.

Summary:
This article outlines a strategy for arraying intact, micrometer sized unilamellar phospholipid bilayer liposomes on a surface by situating and immobilizing the liposomes individually in microwells that have been etched onto the surface in a regular pattern. The potential application of this liposome array is its use as a platform for sequestering and displaying, in the liposomes, integral and peripheral membrane proteins to form a microarray for multiplexed screening applications. The highly selective binding capabilities of surface membrane proteins make them ideal probe molecules for a protein array, yet their strict requirement for a lipid environment to preserve their conformation, mobility, and binding functionality has made them difficult to display. Reconstituting membrane proteins in a lipid environment of intact liposomes and arraying these liposomes on a surface can prove to be a flexible platform for the development of high-density membrane protein arrays. A further advantage is that the intact liposome format can be geared toward probing membrane protein mediated transport or pore formation in vitro.

In the ongoing research, the surfaces used are microwell arrays with 1.2 µm diameter fabricated by photolithography on a silicon wafer coated with silicon oxide layer. A flat block of polydimethylsiloxane (PDMS) impregnated with polyethylene glycol (PEG) terminated silane is used to put down a PEG-terminal background phase using contact printing. PEG terminated monolayers are resistant to protein/liposome adsorption. The ‘bare’ wells are then backfilled using amine terminal 3-Aminopropyltrimethoxysilane (APS). These amine microwells are biotinylated using NHS-PEO4-Biotin (NPB). Next Neutravidin is attached to the Biotin islands to form patterned Neutravidin arrays capable of binding more Biotin. Low Tg lipid formulations containing 5% biotinylated lipids are used to prepare liposomes of about 1 µm diameter using an extrusion technique. The patterned Neutravidin microwell array is then exposed to the liposome solution, which results in attachment of intact liposomes into wells by ‘Biotin-Neutravidin’ interaction. The intactness of liposomes after attachment is verified by co-localization of fluorescence signal from ‘cargo’ incorporated inside the liposomes, fluorescence signal from the fluorescent tag incorporated in the lipid bilayer of liposomes, and the fluorescence signal from Neutravidin grid on the surface using confocal laser scanning microscopy. Other steps involved in the protocol are confirmed using atomic force microscopy, fluorescence microscopy, particle size analysis, and confocal laser scanning microscopy.

Reference:
**Arraying of Intact Liposomes on Chemically Functionalized Microwell Surfaces for Application as Membrane Protein Based Biosensors**

**CNF Project # 1235-04**

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**Figure 1**

Project concept diagram: selective attachment of intact liposomes (containing biotin lipids in the lipid bilayer) into microwells functionalized using Neutravidin.

**Figure 2**

Microwell surfaces fabricated using photolithography: diameter of wells = 1.2 µm, depth of wells = 0.55 µm, pitch = 3 µm. Substrate: silicon wafer with 1 µm thick oxide layer, Active area = 25 sq.mm.

**Figure 3**

Confocal laser scanning microscopy images of Neutravidin-FITC present selectively inside the microwells: (a) Fluorescence data collected close to the top of the microwells. (b) Fluorescence data collected close to the bottom of the microwells. For details about imaging of intact liposomes arrayed into the Neutravidin microwells, please refer to: Langmuir 2006, 22, 5403-5411.
Microfabrication of Electron-Transparent SiO$_2$ Surfaces for Synthesis of Self-Assembled Bioinspired Nanoarchitectures

CNF Project # 1306-05
Principal Investigator: Dr. Carl A. Batt

Abstract:
S-layer proteins are 2D crystalline arrangements of proteins used as templates for the creation of nanostructures and arrays of small feature sizes (< 50 nm) without the slow throughput of conventional lithographic techniques. However, to realize the potential of S-layers as bio-scaffolds, fine control over the interfacial properties is crucial in order to anchor protein units in a regular array. A new substrate for TEM consisting of electron-transparent SiO$_2$ windows (50 nm-thick) has been fabricated to assess the effects of surface chemistry on protein recrystallization.

Summary:
One of the most important technological challenges in nanotechnology is the development of effective patterning methods to control assembly at the nanoscale. Although various lithographic techniques permit the creation of ordered nanostructured arrays with high resolution (i.e. 100 nm feature sizes and/or spacings), these techniques are not cost-effective and the fabrication processes are relatively slow. An alternative parallel approach that is emerging is the use of biological scaffolds, such as proteins and DNA, for the creation of nanostructured materials. One exceptional class of biotemplates for the creation of nanoarchitectures is bacterial surface-layer (S-layer) proteins.

S-layers are 2D crystalline arrangements of proteins and constitute one of the most common outermost structural components of many bacteria. S-layers display different lattice types and distinct classes of pores (2-8 nm) and morphologies. One interesting characteristic of some S-layers is their ability to recrystallize in vitro into monolayer sheets at air-liquid, liquid-solid interfaces. These protein lattices are particularly suitable as biotemplates for creating metallic/semiconductor nanostructures and arrays [1-3], allowing parallel fabrication of small feature sizes (< 50 nm).

However, to realize the potential of S-layer as scaffolds for synthesizing functional nanostructured materials, it is important to generate large-scale monocrystalline arrays via controlled protein re-assembly on technologically relevant substrates. To achieve this goal, fine control over the interfacial properties is crucial in order to anchor protein units in a regular array. Therefore, we are exploring different surface chemistries to manipulate protein-surface interactions and, in effect, the ability to guide the formation of protein self-assembly nucleation sites on gold and SiO$_2$ substrates.

The best method to characterize S-layer protein re-assembly on solid supports is transmission electron microscopy (TEM). However, the current availability of different types of TEM substrates for chemical functionalization is limited. We have developed a new type of TEM grid substrate consisting of electron-transparent SiO$_2$ windows supported by a Si grid. The process used for grid fabrication involved the growth of a 50 nm-thick thermal oxide layer on both sides of a Si wafer 100 µm-thick. The grid windows were patterned by using the Autostep to expose the photoresist layer. After developing the resist, the SiO$_2$ at the exposed areas was etched with a CH$_3$ plasma (under flowing oxygen) using Oxford 80 RIE tool. A Unaxis deep reactive ion etch (DRIE) process was used to etch the wafer down to the bottom-most 50 nm SiO$_2$ layer. Because the substrates are composed only of Si and its thermal oxide, they can be chemically treated in the same manner as thermal oxides on silicon [4].

The grid substrates fabricated using the processes described above will be functionalized with various organosilane reagents to generate self-assembled monolayers presenting different chain lengths and functionalities for future site-directed S-layer protein re-assembly studies.

References:
Microfabrication of Electron-Transparent SiO₂ Surfaces for Synthesis of Self-Assembled Bioinspired Nanoarchitectures

CNF Project # 1306-05  
Principal Investigator: Dr. Carl A. Batt  
Users: Ebenezer Amposan, Magnus Bergkvist, Parijat Bhatnagar, Hongyu Chen, Sonny S. Mark, Yajaira Sierra, Sofia Sotiropoulou, Aaron Strickland, Leonardo M. Teixeira  
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- A new substrate for TEM, consisting of electron-transparent SiO₂ windows, was fabricated for direct imaging of the effects of surface chemistry in the assembly of nanomaterials at SiO₂ surfaces.

Figure 1, top left: TEM image of S-layer protein from B. sphaericus showing square lattices.  
Figure 2, bottom left: Process employed in the fabrication of TEM SiO₂ grids.  
Figure 3, above: TEM image of polymer-capped gold nano-particles. The 10 nm-thick polymer shell is clearly visible showing the functionality of these grids for good contrast and low background noise imaging.
Effect of Micron Scale Surface Features on Dental Papillae Mesenchyme Cells

CNF Project # 1359-05
Principal Investigator & User: Jian Tan

Abstract:
Dental implants have become a universal treatment for the repair or replacement of natural teeth. The success of dental implants depends on both chemical and physical properties of the implant materials. Because the surface of a material is in direct contact with cells, modification of surface properties can play a crucial role in determining the short and long term effects of the implant [1]. In this study, we focused on the role of micron-scale surface structures in regulating the responses of dental cells that participate in the response to implanted materials. Dental papillae mesenchyme (DPM) cells that were isolated from transgenic mice provide a convenient model system for studying dental cell behaviors on implant materials, as these cells can be induced to differentiate into odontoblasts responsible for dentin formation [2].

Summary:
We produced regular arrays of pillars and parallel ridges on silicon wafers using photolithography and reactive ion etching. The size of the pillars was 4 x 4 x 4 µm, and the space between pillars was varied from 6 to 18 µm. The parallel ridges were 4 µm wide and tall, with various spacing at 6 µm to 18 µm. We found that the presence of micron scale features not only changed cell morphology and cytoskeleton organization but also significantly increased cell adhesion and proliferation rate. The shape and the spatial distribution of the micron scale features had significant impact on cellular behavior. Substantially more mineralized tissue was observed on the patterned surfaces when cells were cultured at non-permissive conditions. Taken together, our study suggests that micron scale surface features are critical parameters in the design of new dental implants.

References:

**Effect of Micron Scale Surface Features on Dental Papillae Mesenchyme Cells**

**CNF Project # 1359-05**
**Principal Investigator & User:** Jian Tan

**Affiliation:** School of Chemical and Biomolecular Engineering, Dept. of Biomedical Engineering, Cornell University  
**Primary Funding:** NIH  
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**Figure 1,** above: Pillars (a) and parallel ridges (b) were produced on silicon wafer.

**Figure 2,** below: DPM cells cultured on micro-patterned surfaces. Actin filaments were stained with AlexFluo 488. Actin orientation was clearly influenced by the patterns. a) Pillars. b) Parallel ridges.
Cellular Responses to Micro- and Nanofabricated Surfaces
CNF Project # 1372-05
Principal Investigators: James N. Turner, William Shain

Abstract:
The novel characteristics of molecular scale devices are an area of growing interest. To successfully integrate these devices into biological systems, they must be engineered to accommodate biological interfaces. The ability to control the spatial localization of molecules at micrometer scales on biologically compatible materials is a key requirement for such technologies.

Hydrogels are cross-linked hydrophilic polymers that have been extensively used in various biomedical applications, including soft contact lenses, wound management and healing, and controlled drug delivery and release. Synthetic hydrogels are ideal for biomedical applications in that they present an optically transparent, flexible, and non-toxic material. Hydrogels were photo-polymerized in the presence of streptavidin-acrylamide, resulting in planar gel surfaces functionalized with the streptavidin protein. This surface was capable of binding biotin-labelled biomolecules. The soft protein lithography technique of microcontact printing offers a way to control the patterning of molecules onto a surface. Microcontact printing uses the relief pattern on the surface of an elastomeric stamp to deposit a molecule into a specific pattern on a surface. The stamp is “inked” with the desired solution, dried and brought into contact with the surface. The molecule of interest is only patterned where the stamp is in focal contact with the surface [1]. Polydimethylsiloxane (PDMS) stamps were used to transfer the biotinylated extracellular matrix proteins, fibronectin and laminin, and the biotinylated laminin peptide, biotin-IKVAV, onto streptavidin-conjugated hydrogel surfaces.

In order to fabricate the elastomeric stamps, a series of silicon masters was generated using four-inch silicon wafers patterned using a technique modified from Kleinfeld et al. [2,3]. Briefly, silicon wafers were coated with Shipley 1818 photoresist and ultraviolet (UV)-irradiated through a lithographic mask containing a variety of patterns. From this silicon master pattern, complementary elastomeric PDMS stamps were patterned. Stamps with relief patterns corresponding to either 10 µm-wide relief structures and 90 µm-wide gap spacings or orthogonal 2 µm-wide lines connecting a 15 µm diameter node, with a repeat spacing of 150 µm were fabricated.

As a biological assay, LRM55 astroglioma cells and primary rat hippocampal neurons were plated onto protein stamped hydrogels. Both cell types were found to selectively adhere to areas stamped with biotin-conjugated proteins only. Brightfield and fluorescence light microscopy were used to quantify cell attachment and cell morphology on modified surfaces. Neurons were observed to exhibit significant neurite extension after 72 hours in vitro, and remained viable on protein stamped areas in excess of 4 weeks.

Summary:
We have developed a novel acrylamide-based hydrogel that can be patterned with multiple biologically relevant molecules at micrometer scale resolution to direct neural cell growth and attachment. Results from this study suggest that hydrogel surfaces can be patterned with multiple proteins to direct neural cell growth and attachment. The ability to specifically pattern hydrogel surfaces with a variety of molecules will have major implications for the future design of patterned biomolecular devices.

References:
Cellular Responses to Micro- and Nanofabricated Surfaces

CNF Project # 1372-05
Principal Investigators: James N. Turner, William Shain
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• Biomimetic hydrogel surfaces allow for the precise patterning of multiple biomolecules at the micrometer scale.
• Polycrylamide-based hydrogels can be used as surfaces to control and direct neural cell attachment and growth.

Figure 1, top left: Silicon wafer pattern consisting of 2 µm-wide orthogonal lines with 15 µm-diameter circles. Repeat spacing is 150 µm. Silicon masters are used as templates for elastomeric stamps for use in microcontact printing.

Figure 2, bottom left: Polyacrylamide-based hydrogel microcontact printed with 100 microgram/mL laminin protein. Rat LRM55 astroglia cells adhere to protein-conjugated regions only.

Figure 3, above: Random culture of primary rat hippocampal neurons on glass coverslips coated with poly-L-lysine (left image). Neurons on a hydrogel surface microcontact printed with 1000 pmol/mL laminin peptide, IKVAV (right image).
Nano-Scale Topography and its Effect on Cellular Behavior

CNF Project # 1375-05
Principal Investigator: Kam W. Leong

Abstract:

Silicon wafers with nanometer scale topographical features were designed for use in soft lithography to fabricate replica patterns on PDMS for the study of nano-scale topography effects on cellular behavior in vitro.

Summary:

Cells interact with their native extra-cellular matrix (ECM), with features at the nanometer scale. Our lab has shown that nanoscale patterns significantly change smooth muscle cell morphology, proliferation and cell migration [1].

To study the cellular response to nanometer-sized topographical features, the fabrication of patterns with well defined features that may be reproduced with high fidelity, in large quantities and at low cost, is critical. In the past year, we fabricated silicon wafers with the desired nanopatterns to be used as a mold in soft lithography to reproduce the patterns on poly(dimethylsiloxane) (PDMS).

One pattern consists of rectangular pits of 300 nm x 500 nm. Each rectangular pit is 150 nm apart from the next. Other patterns comprise line gratings with widths ranging from 50 to 100 nm. Efforts are being made to vary the height dimension of the replica PDMS patterns (relief modification), which may be used to determine the extent of cell sensing of topographical relief.

One cellular study involves evaluating the biological response of monocyte and macrophage cultured on these PDMS replica patterns, which could define the role of nanotopography in dictating foreign body reaction to implants. Another study involves the trans-differentiation of adult human stem cells cultured on these nanopatterned PDMS [2].

References:


Nano-Scale Topography and its Effect on Cellular Behavior

CNF Project # 1375-05
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Figure 1, above: Scanning electron micrograph of the rectangular pit pattern produced by JEOL at magnification of 75000X.

Figure 2, below: AFM 3D surface plot and cross-section images of line gratings on silicone wafer with width (w): trough (t):height (h) dimensions of (a, b) 50nm:150nm:100nm; and (c, d) 100nm:200nm:100nm.

Figure 3: Fluorescent (a) and phase contrast (b) images of macrophage cells elongating along 500 nm PLA gratings at the boundary of patterned glass.
Planer Patch-Clamp Arrays: Neuronal-Electronic Interfaces for Examining Network Development in Neuronal Ensembles

CNF Project # I377-05
Principal Investigator: Professor Hongkun Park

Abstract:
Recent advances in the experimental sciences and coupled data analyses have paved the way for investigating systems of increasing complexity. One system of significant interest is neuronal networks: large composites of neurons interconnected via synaptic junctions. These networks not only form the physiological basis of many biological organisms’ central and peripheral nervous systems, but, functionally, are responsible for manifesting higher order cognitive properties, including memory and logic [1]. Nevertheless, a thorough understanding of the manner through which these properties emerge as an undifferentiated collection of neurons operating as a biological circuit has not yet been realized. And while the materialization of these functions implicates both the networks in question and the inputs they receive, understanding the spatiotemporal development of a model network’s connectivity in response to a series of applied inputs is an important first step toward decoding this emergence. To this end, we have fabricated planer arrays of patch-clamp electrodes in order that we might probe the afore-mentioned development on a one-electrode-to-one-neuron basis [2-5].

Summary:
During the past year, we have completed the construction of a ten by ten planer array of patch-clamp electrodes at 50 µm pitch as a proof-of-principle. This test neuronal-electronic interface, meant to reproduce the geometry of many patch-clamp micropipettes protruding from a cell-culture glass coverslip, is based upon the previous work performed by Pantoja et al. [4] and Lehnert et al. [5]. Essentially, one hundred apertures or micronozzles, respectively, were generated in a silicon wafer using a series of gas etches.

Briefly, a reactive ion etch was used to define a silicon dioxide etch mask for a subsequent front side deep reactive ion etch. After these steps, the silicon wafer possessed a ten by ten array of 2 µm in diameter, 50 µm deep trenches. Using a backside-aligned photolithographic step, another reactive ion etch, and a second deep reactive ion etch, a ten by ten array of 25 µm diameter trenches were etched to meet the front-side set, yielding one hundred apertures within the wafer.

At this point, the wafer was either considered finished or micronozzles were made using a final front-side reactive ion etch and deep reactive ion etch. After electrically and electrochemically passivating the wafer with a series of MOS furnaces, it was ultimately diced for final packaging.

In the packaged state, the above-described interface is coupled to SU-8 microfluidics layer which is spun atop an electrically and electrochemically passivated silver-chloride multi-electrode array. Assembled, each planer pipette in our array has its own solution exchange lines and recording electrode.

Thus far, initial experiments have shown the viability of the implemented multi-electrode array, our microfluidic design, and the chip’s interface as a site for culturing cells. At present, we are in the process of testing our chip for full functionality.

References:
Planer Patch-Clamp Arrays: Neuronal-Electronic Interfaces for Examining Network Development in Neuronal Ensembles

CNF Project # 1377-05
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Web Site: http://www.people.fas.harvard.edu/~hpark/index.html

4-Layered Structure

1. Recording Chamber: Solution Support.
3. SU-8 Microfluidics: Pressure Gradients, Perforated Patching.
4. Multi-Electrode Array: \( V_m \).

Figure 1: Top portion of image taken from www.warnerinstruments.com

Microfluidics Atop MEA

Planar Pippettes - Products

Planar Pippettes - Cell Culture
Optical Biosensor Using Metallic Nano-Structure

CNF Project # 1379-05
Principal Investigator: Michael L. Shuler

Abstract:
We propose a new approach to localized surface plasmon resonance (LSPR) sensors that would be easily implemented into a portable system with high sensitivity. Nano-grating surface plasmon (NGSPR) sensors use vertical illumination to excite localized surface plasmon polarization (LSPP). The use of nano-grating can enhance the refractive index sensitivity of LSPR sensors by large resonance wavelength shifts with the surrounding materials and sharp reflection resonance peaks. The influence of structural factors of nano-gratings on the sensitivity of NGSPR sensors was investigated using the rigorous coupled waveguide analysis (RCWA) method.

NGSPR sensors with line widths less than 50 nm show narrow reflection peaks from the excitation of LSPP. We predict that a refractive index sensitivity of more than 400 nm/RIU can be obtained using an optimized structure and materials. Sharp reflection resonance peaks with FWHM of 0.03 eV will further enhance the sensitivity of the sensors.

The simple optical configuration of normal incidence and high refractive index sensitivity make it possible for NGSPR sensors to be used as portable biosensors for high-throughput screening applications. The proposed sensor structure is suitable for mass fabrication using soft lithography or surface plasmon interference nanolithography (SPIN) method.

Summary:
Surface Plasmon Resonance (SPR) sensors have been widely used as a tool for analyzing various biochemicals with high sensitivity without labeling. In a conventional SPR sensor, one interactant pair is immobilized on a thin metal film layer (usually silver or gold), and the other interactant in a liquid solution is flowed on this surface. The optical reflectivity of the thin metal film layer responds to the surface condition at certain incident angle and wavelength, the so-called surface plasmon resonance condition. One major advantage of the SPR sensor is that it can measure the change of reflectivity with very high sensitivity without any fluorescent or other labeling of the interactants. Furthermore, SPR imaging method allows for making quantitative analysis of biochemical reactions of multichannel samples in real time. Also nano-scale structures or particles can be used to further enhance the sensitivity of the SPR sensor [1].

Till now most of experiments were done in colloidal status or using substrates made by the self-assembled nanosphere lithography (NSL) method. The metallic nano particle in colloidal solution can’t be made into a small size array type chip. NSL is not suitable for mass production and large size array devices because the method is limited by the size and uniformity.

We propose nano-grating surface plasmon resonance (NGSPR) sensors [2]. NGSPR sensors with a minimum grating feature size less than 50 nm and a period of 500 nm produce relatively sharp resonance reflection peaks. The resonance wavelength of the sample peak varies with the refractive index of the sample materials. NGSPR sensors have a simple structure for mass fabrication and are less sensitive to the grating geometry. The predicted refractive index sensitivity of NGSPR sensors is larger than 400 nm/RIU and the FOM value is expected to be more than 60 due to the narrow FWHM, which is much better than alternative methods.

NGSPR sensors can be made into robust equipment using visible wavelength spectroscopy in reflection geometry. This simple vertical illumination reflection spectroscopic configuration can be easily implemented in small size systems without any moving parts and refractive index matching mechanisms. The size of unit cells in NGSPR sensors can be made smaller compared to conventional SPR imaging sensors in which the unit cell size is limited by the surface plasmon propagation length. NGSPR sensors can be used as an economic tools for high-throughput screening (HTS) application.

References:
Optical Biosensor Using Metallic Nano-Structure

CNF Project # I379-05
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Figure 1, above: Schematic diagram of a nano-grating based reflection type SPR sensor.

Figure 2, top left: Calculated reflectance spectra of a NGSPR sensor in various refractive index conditions ($\Lambda = 400$ nm, $\mu = 50$ nm, $d = 40$ nm, $t = 40$ nm).

Figure 3, bottom left: Resonance wavelength vs. refractive index of the sample (upper inset & square: gold, lower inset & circle: silver) $\Lambda = 500$ nm, $\mu = 50$ nm, $d = t = 50$ nm.
Electroactive Nanowell Sensors for Parallel Particle Trapping and Detection

CNF Project # I387-05
Principal Investigator: David Erickson

Abstract:
In this report we present our initial work towards the development of electroactive nanowell arrays. Here we demonstrate electrokinetic particle trapping. However we also lay the framework for developing individually addressable nanowell using a cross electrode architecture. Future work involves the extension to control over the chemical and thermal conditions in the “nano-environment”.

Summary:
Recently, a series of techniques have been demonstrated for single cell trapping in microwell arrays. While these techniques enable discrete optical observation of the biological agents, their passive nature limits the degree of inherent functionality. Here we present an electro-active nanowell platform for precise parallel manipulation of individual nanoparticles and biotargets to discrete nanowell sites. In the platform, parallel manipulation of coupled electrophoretic and dielectrophoretic effects are used to guide individual nanoparticles to the desired well site. In addition, localization of the electric field using this approach enables high sensitivity electrical impedance spectroscopy (EIS) along with simultaneous thermal and chemical control over the nano-environment through manipulation of Joule heating and electrolysis reactions. Immediate applications include high throughput single cell trapping and accurate rapid cell screening without fluorescence.

Device Fabrication and Results:
Nanowell arrays are made by depositing a 2 µm parylene dielectric layer on an indium tin oxide (ITO) coated glass substrate. A thick PMMA resist layer is spin-coated on the dielectric layer and patterned using e-beam lithography. The resist is then developed, followed by an oxygen plasma etch, leaving the ITO exposed on the well sites. In the initial experiment (Figure 1), we applied a 5V DC field to drive 500 nm polystyrene beads into wells, and after reversing the polarity, we observed particle rejection from the wells. In order to provide single well addressability and reduce dielectric charging, 50 µm ITO electrodes were etched from the ITO substrate (Figure 2). Figure 2a is a schematic of our current procedure, in which wells aligned on top of interdigitated ITO electrodes are etched on a polyimide layer. A top ITO electrode layer is then placed orthogonal to the bottom layer, and they are separated by two 70 µm spacers.

From our electrokinetic model, reducing the electrode area should enhance dielectrophoretic effects, which we are currently characterizing. Furthermore, since particles have a characteristic length on the same order as that of our wells, well occupancy should greatly affect the field lines and impedance spectra; rendering EIS as an adequate mechanism for testing well occupancy, biotarget viability and particle size.
Electroactive Nanowell Sensors for Parallel Particle Trapping and Detection

CNF Project # 1387-05
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Figure 1: Initial device prototype and experimental results. (a) Single well experiment attracting particles from solution into electroactive microwells. (b) SEM image of electroactive well after fabrication. (c) SEM image showing particles which were electrostatically attracted into the well. (d) Illustration of electrokinetic body forces applied to the nanoparticles near the well. Scale bar in (b) 2 µm.

Figure 2: Current device setup layout. (a) Cross electrodes layout enabling single well trapping and rejection in nanowell array. (b) 3 µm polystyrene beads in proximity of nanowells patterned on polyimide on top of ITO electrode.
Fabrication of Fused Silica Nanofluidic Channels

CNF Project # [393-05]
Principal Investigator: Dr. Jun Kameoka

Abstract:

Miniaturization to the nanometer scale opens up the possibility to manipulate biomolecules and study biological processes at single molecule resolution. A nanofluidic channel is one such device that enables us to investigate individual molecules of interest by physically confining the molecule while it passes through the detection volume [1]. Because the dimension of a nanochannel is comparable with the size of single molecule, single molecule resolution can be achieved.

A variety of approaches to fabricate nanofluidic channel devices have been reported. In this project we used the state-of-the-art JBX-9300FS electron beam lithography system, which has a sub-20 nm resolution. UV-grade fused silica was chosen as the substrate material due to its superior optical properties and processibility. A 4-inch fused silica wafer was first spin-coated with a layer of electron beam lithography resist (PMMA) and then a layer of water-soluble conductive polymer (Spacer 300Z, Showa Denko) to eliminate charging during exposure. After electron beam exposure and develop of PMMA, a 50 nm-wide trench pattern was transferred onto PMMA. The fused silica wafer was then etched by CF$_4$ plasma to a depth of 40 nm.

To facilitate the injection of analyte solutions, a microchannel was aligned and patterned on top of the nanochannel using photolithography, connecting the nanochannel in the center from both ends. The microchannel was then dry etched to a depth of 1 µm so that the channel would not collapse upon bonding. The inlets were blasted through at two ends of the microchannel using an alumina powder blaster. Finally, the fused silica wafer was bonded with another cover fused silica wafer and made to cling to each other using de-ionized water.

Summary:

A 50 nm-wide and 40 nm-deep fused silica nanochannel was fabricated by electron beam lithography. To facilitate liquid injection, two parts of microchannels connecting the nanochannel in between were also fabricated by photolithography. The nanofluidic channel device was achieved by bonding the fused silica wafer with another fused silica wafer using de-ionized water.

This device enables us to study biomolecules at single molecule resolution by spatially confining the molecule of interest in the nanochannel while it moves through the detection volume.

References:

Fabrication of Fused Silica Nanofluidic Channels

CNF Project #: 1393-05
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Figure 1: Scanning electron microscope image of cross section of a 50 nm-wide and 40 nm-deep trench.

Figure 2: Scanning electron microscope image of top view of a fused silica nanochannel connected by a microchannel.
Examining and Influencing Order in the Flow of Worm-Like Micelles Through Porous Media

CNF Project # I405-05
Principal Investigator: Robert K. Prud’homme

Abstract:
Surfactants are widely used in industry as rheological modifiers. In particular, surfactants which form worm-like micelles have found use in the energy industry as fracturing fluids [1]. It has been demonstrated that these solutions can align in shear, as well as in flow through porous media—a fact that complicates their usage [2]. In order to better study the behavior of these worm-like micelles, we have fabricated a simple microfluidic flow device which mimics the flow in natural rock formations.

Summary:
We have created a series of devices in silicon, which consist of a narrow flow channel with circular wells at either end for fluid entry and exit. In the center of each flow channel, there is an array of identical offset octagons, which are spaced one diameter apart. The octagons range in size from 30 µm to 1 µm in diameter. Each device is then etched to a depth equal to this diameter, thus ensuring the fluid sees a tortuous flow channel inside this array. The silicon wafer can then be anodically welded to glass. Fluidic connections to the device are established by drilling through the silicon prior to welding, and then using Upchurch Nanoport fittings.

References:
Examining and Influencing Order in the Flow of Worm-Like Micelles Through Porous Media

CNF Project # 1405-05
Principal Investigator: Robert K. Prud’homme
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Figure 1: Sample wafer before etching to illustrate the general design of the device.

Figure 2: This micrograph shows a close-up of the offset octagonal array present in the center of each flow channel. This array can be seen in Figure 1.
Development of Non-Chemically Amplified Molecular Glass Photoresists for Next Generation Lithography

CNF Project # 386-90
Principal Investigator: Christopher K. Ober

Abstract:
In order to meet the growing demand of the electronics industry for smaller, higher resolution features, much recent attention has focused on next generation lithographic techniques, such as extreme ultraviolet (EUV) or e-beam lithography. Complementary to this field of research is the design of novel photoresists to produce sub 50 nm feature sizes. Chemically amplified molecular glass resists are among the most promising alternatives to traditional polymeric materials. These materials are monodisperse, amorphous organic molecules which lead to high resolution patterns with low line edge roughness owing to their small size and lack of chain entanglement. In this report, we describe our work in the development of molecular glass resists. The materials are designed with rigid cores, to ensure high glass-transition temperature, and with bulky side groups to inhibit crystallization. We show that these materials are capable of producing high resolution feature sizes and show great promise in meeting the demands of emerging next-generation lithographic techniques.

Summary:
As the semiconductor industry moves to ever smaller feature sizes, extreme ultraviolet (EUV) lithography is emerging as one of the most promising next-generation lithographic techniques for the production of sub 50 nm size features [1]. Resists designed for this wavelength must be sensitive in order to compensate for the typically low power of EUV sources, and capable of producing high resolution features. In addition, they must fulfill low values of line edge roughness (LER). A recent advance in the design of resists has been the introduction of molecular glass (MG) resists [2]. Unlike conventional polymeric resists, which are large, polydisperse systems, MG resists are materials made up of small, discrete molecules rendered amorphous by functionalizing asymmetric core structures with bulky side groups [3].

In this report, we describe the lithographic evaluation of a non-chemically amplified calix[4]resorcinarene resist system. p-tert-Butyl calix[4]resorcinarene was prepared using conventional wet-chemistry techniques and further protected with 1,2-naphthoquinone-2-diazido-4-sulfonyl chloride (DNQ-Cl) to produce solubility switch [4]. Lithographic evaluation of the DNQ-protected calix[4]resorcinarene was performed using the equipment in the Cornell NanoScale Science and Technology Facility (CNF). The DNQ-calix[4]resorcinarene was dissolved in propylene glycol mono-methyl ether acetate (PGMEA) containing triphenylsulfonium nonaflate as a photoacid generator (PAG). The resulting resist formulation was filtered and then spin-coated onto a hexamethyldisilazane (HMDS) primed wafer. After exposure, the wafer was baked at an elevated temperature and developed in a tetramethylammonium hydroxide (TMAH) solution.

The resist produced positive tone images after exposure to 405 nm radiation and development in 0.26N TMAH, but surprisingly, no images were immediately apparent after exposure to e-beam radiation. However, negative tone images were obtained after the wafer was flood-exposed with 365 nm UV light after e-beam exposure [5], then developed in 0.26 N TMAH. In summary, e-beam radiation caused a crosslinking of the resist, rendering it insoluble in aqueous base.

Flooding exposure to UV light of the surrounding wafer enabled the facile removal of the unexposed resist, leaving the negative tone patterns behind. This particular resist resulted in the formation of 150 nm images, though we are confident that higher resolution images can be obtained after optimization of the chemical structure of the resist molecule and lithographic process parameters.

We have investigated the single component molecular glass resist bearing photoactive DNQ side groups, resulting in a non-chemically amplified resist for advanced lithography. Preliminary results demonstrate that this resist functions as a positive tone resist when exposed in the presence of water, but undergoes a cross-linking reaction when exposed in a vacuum, resulting in a negative tone system. We believe that the incorporation of the DNQ-moiety into the molecular glass backbone can lead to a new horizon in the next-generation photolithographic technology platform.

References:
Development of Non-Chemically Amplified Molecular Glass Photoresists for Next Generation Lithography

CNF Project # 386-90
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Figure 1: DNQ-modified p-tert-Butyl calix[4]resorcinarene.

Figure 2: (A) Contrast curve of DNQ-modified p-tert-Butyl calix[4]resorcinarene exposed at 405 nm, E_o 63 mJ/cm², contrast 1.13. (B) 150 nm negative tone features obtained after e-beam exposure (dose 600 µC/cm²).
Non-PFOS Photoacid Generators for Chemically Amplified Resists

CNF Project # 386-90
Principal Investigator: Christopher K. Ober

Abstract:
Chemically amplified resists (CAR) based on acid catalyzed reaction have become the primary process in all advanced semiconductor manufacturing. Further improvements could enable use of the CAR process in all next generation lithography (NGL) including extreme ultra violet lithography (EUVL). The photoacid generator (PAG) present in the CAR formulation influences the performance of a CAR in dramatic ways and becomes one of the indispensable components in a CAR. Any PAG that is expected to be viable in NGL should be perfluorocyclosulfonate (PFOS) free because of the environmental and technical concerns associated with PFOS.

To address the PFOS concern, new ionic PAGs comprising a triphenylsulfonium unit as the photoactive cation and aryloxyperfluorooctanesulfonate as the anion were synthesized. The new PAG produces perfluoroalkylsulfonic acid containing only two perfluorinated carbons—much less than what is found in PFOS. E-beam and EUV lithography experiments performed in a model resist containing new non-PFOS PAGs and TPS PFBS reference PAG exhibited comparable resist sensitivity, resolution and line edge roughness (LER).

Summary:
Non-PFOS photoacid generator [1,2] was synthesized by a multi-step reaction using phenoxide and 1,2-dibromotetrafluoroethane as the precursors. The detailed synthetic procedure used to obtain PAG 1 (Figure 1) is described elsewhere [3-5]. Lithographically relevant properties of the PAGs such as absorption, thermal, solubility in resist formulation, and formulation stability, were quantified and compared with commercially accepted TPS PFBS PAG (Figure 1). The generation of photoacid from the new PAGs and their function as a strong catalyst in a model positive tone chemically amplified resist was investigated at different wavelengths including deep ultraviolet (DUV or 254 nm), e-beam and extreme ultraviolet (EUV or 13.4 nm) radiation [5].

Lithographic evaluation was performed using equipment in the Cornell NanoScale Science and Technology Facility (CNF). The resist solution of polymer and PAG in propylene glycol monomethyl ether acetate (PGMEA) was prepared. To get a film with ~125 nm thickness, 6% of polymer [poly(α-gamma-butyrolactone methacrylate-co-methylcadamantyl methacrylate)] solution in PGMEA was used. PAG 1 and TPS PFBS (5 weight % to polymer) were used as PAG. The solution was spun coated onto an HMDS primed silicon wafer, and baked at 115°C for 60 seconds. Preliminary lithographic evaluations were performed at DUV (254 nm) wavelength using Hybrid Technology Group’s (HTG) III-HR contact/proximity mask aligner. E-beam exposure was conducted using a Leica VB-6 e-beam tool operating at 100 KeV. A job file containing a pattern ranging from 1 µm to 30 nm was projected onto the resist film. Areas on each wafer were exposed at a dose ranging from 12 to 40 µC/cm² with increment of 4 µC/cm² in each step. After exposure each wafer was baked at 120°C for 60 s. The wafers were then immersed in a 0.26 N TMAH solution for 30 seconds and rinsed with water. The areas of imaging layers exposed to the e-beam radiation were washed away by the developer solution and the areas unexposed to e-beam radiation were left on the wafer. In order to understand the effect of PAG on resist sensitivity and the other lithographic performances the same molar concentration of PAGs was maintained in both resist formulation. The dose to size (Eₙ) 100 nm equally spaced lines observed for different PAG is shown in Figure 2. The resist sensitivity for both formulations are comparable as expected from their physico-chemical properties (acid strength and size). In order to understand the effect of PAG structures on resolution and line edge roughness (LER), the printed line and spaces were examined with high resolution Zeiss scanning electron microscope (SEM). Both formulations resolved sub 100 nm lines and spaces.

We have successfully developed new environmentally friendly PAGs containing reduced amount of fluorine for chemically amplified resists. The PAGs have good solubility in common solvents and comparable thermal properties to standard TPS PFBS PAG. The PAGs also show high sensitivity upon exposure to e-beam radiation and are capable of resolving sub-100 nm lines and spaces. In addition to their superior lithographic performance, the PAGs contain a reduced amount of fluorine along with added functional groups so these new PAGs are expected to be friendlier to the environment.

References:
Non-PFOS Photoacid Generators for Chemically Amplified Resists

CNF Project # 386-90
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- Physico-chemical properties of PAG 1 and TPS PFBS are comparable.
- Tested in a model positive tone resist at EUV and e-beam radiation.
- Resist sensitivity, resolution and LER of PAG 1 and TPS PFBS are comparable.

Figure 1: Photoacid Generators (PAGs). (A) Non-PFOS PAG (PAG 1). (B) Standard PAG (TPS PFBS).

Figure 2: SEM Micrographs obtained with resist formulation containing PAG 1 (A) and TPS PFBS (B) exposed to e-beam radiation.
Molecular Glass Resists for Next Generation Lithography

CNF Project # 386-90

Principal Investigator: Christopher K. Ober

Abstract:

The semiconductor industry requires new photoresist materials in order to operate in the sub 50 nm regime. In addition to meeting resolution and line edge roughness requirements, these photoresists must be transparent in the extreme ultraviolet (EUV) regime and have excellent etch resistance characteristics. This report highlights several small molecule molecular glasses, which are low molecular weight organic materials that demonstrate a glass transition temperature as well as a low tendency towards crystallization, with new architectures designed for EUV lithography. Transparency at the EUV wavelength of 13.4 nm may be enhanced by incorporation of low absorbing atoms such as C, H and Si. Rigid, asymmetric structures have been included in order to reduce crystallization and increase $T_g$. Studying the effects of these design characteristics across a continuum of architectures enables greater insight into the factors affecting photoresist performance.

Summary:

This work highlights the challenges of designing a molecular glass photoresist for EUV lithography. When choosing a robust glass-forming core, molecular shape is a very important parameter. This defines molecular flexibility as well as the packing ability of various molecular systems. With respect to their topology, glass forming molecules can be twin, spiro, star, branched or tetrahedral in nature. Our research focuses on a molecular system with a silicon core atom with its $sp^3$ orbitals enforcing the tetrahedral shape through molecular geometry. A persistent tetrahedral structure offers possibilities towards constructing a diamond-like 3-D architecture with promising glass forming properties without the need to incorporate $T_g$-suppressing aliphatic chains to prevent crystallinity.

Since masking the phenolic groups with a protecting group such as t-butoxy carbonate made these tetrahedral structures too flexible and produced too low $T_g$, these molecular glass cores were used as negative tone photoresists with the addition of a commercially available crosslinker. Upon exposure to UV or electron beam radiation, crosslinking between the multifunctional crosslinker and the hydroxyl groups of the photoresist, catalyzed by the photoacid generator, resulted in a robust cross linked network that does not dissolve in aqueous base.

Contrast curves were conducted using the HTG contact aligner with a wavelength of 405 nm to evaluate the lithographic performance of the resist systems. Better test tools are being sought in the NNIN network since these molecular glass photoresists are capable of extremely high resolution.

Both systems demonstrated good sensitivities in the 1-1.2 mJ/cm² and can be developed in a range of aqueous base concentrations. High sensitivity could be due to the efficient o-alkylating crosslinking reaction between the phenolic resist moieties and the crosslinking agent.

The silane based molecular glasses are being further evaluated through e-beam lithography. The SEM in Figure 3 shows a 200 nm Line/Space pattern of the tetrakis (3-hydroxyphenyl) silane with Powderlink® crosslinker system. More lithographic pattern testing is still underway.

References:

Molecular Glass Resists for Next Generation Lithography

CNF Project # 386-90
Principal Investigator: Christopher K. Ober
Users: Anuja De Silva, Drew Forman, Nelson Felix

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Figure 1: Tetrakis (n-hydroxy phenyl) silane derivative.

Figure 2: Contrast curve for Tetrakis (3-hydroxyphenyl) silane with Powderlink® crosslinker.

Figure 3: Tetrakis (3-hydroxyphenyl) silane with Powderlink® crosslinker.
High-Resolution, Supercritical CO$_2$-Developable Molecular Glass Photoresists

CNF Project #386-90
Principal Investigator: Christopher K. Ober

Abstract:

The use of supercritical CO$_2$ (scCO$_2$) in semiconductor manufacturing has many potential benefits from both an environmental and technical point of view. Along with being non-toxic and non-flammable, scCO$_2$ is a zero surface tension, high diffusivity fluid capable of processing features on the nanometer scale. However, finding photoresists and strategies suitable for CO$_2$ processing has proved to be a challenge. Until recently, patterning and development of sub 100 nm photoresist features has not been possible, and photoresists required fluorination to impart solubility. With the growing adoption of small molecule resists, also known as molecular glasses, the possibility of CO$_2$ solubility and high solubility contrast drastically increases. With scCO$_2$ development, we have been able to show positive- and negative-working resists patterned down to 50 nm features, all without the need for fluorine. This performance with an environmentally friendly solvent is comparable to current photoresist technologies.

Summary:

Molecular glasses are an innovative type of photoresist based on small, discrete molecules rather than polymers [1-3]. Because of this, they possess potential advantages over standard polymeric resists. The small, uniform size of molecular glass resists has the potential to alleviate the line-edge roughness problem facing next-generation lithography. Also, due to their small size, molecular glasses have the potential for supercritical CO$_2$ solubility without the need for fluorination. This greatly expands the range of materials that can be developed in scCO$_2$.

A high contrast molecular glass resist, hexa(hydroxyphenyl)benzene (HHPB) was used as a high contrast photoresist. Details of the synthesis are described elsewhere [4]. This molecule with six hydroxyl functionalities was protected with tert-butoxycarbonyl (tBOC), which is acid labile. Upon exposure, the resist is deprotected, rendering the molecule more polar and less soluble in scCO$_2$.

A 10 wt% solution of tBOC-protected HHPB was made in propylene glycol methyl ether acetate (PGMEA). To this solution, a 5 wt% loading of a photoacid generator, triphenylsulfonium perfluoro-1-butanesulfonate, was added. This was spin-coated to a 200 nm thickness, soft baked at 115°C for 60 s and patterned using CNF’s Leica VB6 electron beam system operating at 100 kV. Post-exposure bake was performed at 90°C for 30 s and then developed in scCO$_2$. Supercritical CO$_2$ exists above 31°C and 1070 psi, and processing conditions were explored from 35°C to 65°C and 2000 psi to 10,000 psi. Using contrast curves, the optimal condition for development was found to be 35°C and 5000 psi.

After development at 35°C and 5000 psi for 3 minutes, features as small as 50 nm lines and spaces were resolved. This shows the high development contrast of the molecular glass resist in scCO$_2$ as well as the potential of scCO$_2$ processing. These features were at an aspect ratio of 3:1 without collapse, showing the ability of supercritical developers to alleviate pattern collapse.

This is the first reported instance of sub 100 nm resist features developed in scCO$_2$ and potentially brings supercritical processing to the same level as conventional resist development.

References:

High-Resolution, Supercritical CO$_2$-Developable Molecular Glass Photoresists

CNF Project # 386-90
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Figure 1: Reaction scheme of tBOC-protected hexa(hydroxyl phenyl)benzene upon exposure and acid generation. Exposure renders the photoresist more polar and therefore less soluble in supercritical CO$_2$.

Figure 2: After development in supercritical CO$_2$ at 35°C and 5000 psi for 3 min, 50 nm lines and spaces can be resolved in the resist. Aspect ratios of resist features are as high as 3:1, demonstrating the advantages of supercritical development.
NanoLC Fraction Analysis by Chip-Based Nanoelectrospray

CNF Project # 740-98
Principal Investigator: Dr. Thomas N. Corso

Abstract:
In previous reports we have shown our progress in the development and commercialization of a monolithic microfabricated microfluidic device for the purpose of conducting nano-electrospray ionization (nESI) when coupled to a mass spectrometer. We have recently developed further fluidic handling capabilities for users of nano liquid chromatography columns (nanoLC) and created the first ultra-low volume fraction collection technique for nanoLC with subsequent nanospray infusion analysis for extend MS analysis time targeted at glycopeptide analysis.

Summary:
NanoLC with 75 µm id columns and flow rates of 200 nL/min is gaining in popularity due to improved resolution, lower sample injection requirements, and better ionization efficiency leading to improved sensitivity. NanoLC peaks typically elute within 20 sec, providing most modern mass spectrometers sufficient time to perform MS/MS for simple protein ID experiments. However, for complex samples, such as glycopeptides, where MS³ or MS⁴ experiments may be needed, nanoLC does not provide adequate analysis time.

By collecting fractions from the nanoLC, analysis times can be extended via nanospray infusion analysis. Fractions from a nanoLC were collected into pipette tips, these 200 nL fractions were collected every 60 sec from a column flowing at 200 nL/min with a 30 min gradient. Fractions were collected from the peak elution window of interest in an automated fashion using the NanoMate. The nanoLC fractions in the pipette tips dried within several minutes. Following fraction collection the residue in each tip was reconstituted. The sample was analyzed directly from the tip with chip-based nanoelectrospray using our ultra low flow chip. This chip has 3 µM id nozzles, producing flow rates of < 40 nL/min and providing 8 min analysis per fraction. Fraction collection, reconstitution and analysis steps were fully automated.

To demonstrate the system, Ribonuclease B (RNaseB) tryptic digest, which is known to have a single N-linked glycosylation site on the asparagine 34 residue, was used. 5 pmol of RNaseB tryptic digest was injected onto a 75 µm x 360 µm x 15 cm operated at a flow rate of 200 nL/min. 60 sec nanoLC fractions (200 nL) were collected into pipette tips by robotic capillary insertion. During the collection time a solvent droplet on the end of the capillary grew, and at the end of the collection time, the capillary was withdrawn from the pipette tip, capturing the droplet inside the end of the pipette tip. For the RNaseB analysis 24 fractions were collected. The 200 nL fractions in the tips dried in minutes and a reconstitution step was performed with a desired solvent composition. 75% methanol in water with 0.1% acetic acid was added to wells of a 96-well plate.

The NanoMate robotically aspirated 500 nL into a pipette tip containing a fraction, and then waited 75 sec before engaging the tip to the chip and performing the infusion analysis. The purpose of this delay period was to allow for both solvation of the LC fraction and evaporation of the reconstitution solvent to reduce volume and concentrate the sample. It was estimated that the final volume of solvent was approximately 300 nL and the composition was 50% methanol. The analyses were performed on an LCQ Deca XP ion trap equipped with a NanoMate. MS³ and MS⁴ experiments were performed on the nanoLC fractions containing isoforms of the glycopeptide. The extended analysis time of each fraction allowed for collision energy optimization, data averaging, and multiple tandem MS experiments. The site of glycosylation was determined, the PTM was characterized as having 9 mannose and 2 N-acetylglucosamine groups, and the sequence of the peptide in which the glycosylation occurred was determined. This approach will be demonstrated as a useful means of extending analysis times for nanoLC.
NanoLC Fraction Analysis by Chip-Based Nanoelectrospray

CNF Project # 740-98
Principal Investigator: Dr. Thomas N. Corso
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Figure 1, opposite left: (A) NanoLC fractions are collected into pipette tips when the fused silica column tail is inserted through the end of a pipette tip by the NanoMate. A droplet of column effluent grows until the pre-determined collection time has been reached. (B) Then the fused silica is quickly retracted and the effluent is captured at the tip of the pipette.

Figure 2, below left: (A) NanoLC chromatogram of 100 fmol of RNase B tryptic digest. Four 90-second nanoLC fractions were collected in the ranges indicated. Glycopeptides were found in tips #2 and #3, but there was no evidence of them in tips #1 or #4, showing that the chromatography is preserved in the nanoLC fractions. (B) The full scan MS spectrum from tip #2 (shown in Figure 9A) shows the presence of five glycopeptides in the nanoLC fraction, varying from five to nine mannose groups.

Figure 3, above: Cross-section cartoon depicting of the fluid interface for the nanoelectrospray chip. A micropipette tip supplies the sample to the back of the chip’s inlet. Inset: SEM image of the 2.5 µm id nanoelectrospray nozzle.
Integration of Microfluidics to Electrospray Ionization Mass Spectrometry Using a Chip-Embedded SU-8 Electrospray Tip

CNF Project # 762-99
Principal Investigator: Harold G. Craighead

Abstract:

We present two methods for creating polymeric microfluidic devices integrated with electrospray tips for use with mass spectrometry. SU-8 was used to create microfluidic channels and electrospray tips. The planar electrospray tips were located at the end of microfluidic channels and were formed using standard photolithography. The encapsulation of the microfluidic channels was accomplished by using thermal and press bonding between two SU-8 layers, or by employing a sacrificial layer removal technique. We successfully coupled these microfluidic chips to a mass spectrometer. In particular, the microfluidic device fabricated with the sacrificial layer removal method shows satisfactory electrospray stability.

Summary:

Progress towards the combination of microfluidics and mass spectrometry has been promising. In particular, polymeric microfluidic devices have been studied because of their compatibility with mass spectrometry. Recently, polymers including cyclic olefin copolymer and parylene were employed to create microfluidic devices for pre-concentration [1], analyte separation [2] and electrospray ionization [3]. In this work, we present two methods to create an SU-8 microfluidic device with an electrospray tip.

The integration of a polymer-based planar electrospray tip with a polymeric microfluidic device has been achieved without a transfer capillary or liquid junction. By using SU-8, both the channels and the electrospray tip can be patterned accurately. Due to its solvent-resistant property, SU-8 is an excellent material for microfluidic devices. Figure 1 shows optical micrographs and SEM images of the devices. In the first process, pressure and thermal bonding of two SU-8 layers has been used for channel encapsulation. Three SU-8 layers with 14 µm thickness were formed by photolithography. After the deposition of the first tip layer, a gold electrode was patterned using e-beam deposition and photolithography. The microfluidic channels (60 µm x 14 µm cross-section) were deposited on the tip (bottom) layer and encapsulated by a lid layer using thermal and press bonding. Secondly, a sacrificial template of AZP 4620 was employed for channel formation. After the SU-8 channel formation on the tip layer, these channels were filled with AZP 4620 and the sacrificial template pattern was defined by standard photolithography. After deposition and exposure of another top SU-8 layer, the entire device was developed in SU-8 developer. High-pressure use of the device is possible due to the tight sealing between the two SU-8 layers. It is possible to demonstrate the capability of an integrated on-chip polymer electrospray tip. We have monitored the total ion current (TIC) of a calibration sample solution containing caffeine (m/z = 195), L-methionyl-arginyl-phenylalanyl-alanineacetate.H_2O (MRFA) (m/z = 524) and ultramark 1621 using both devices. We also present the mass spectra of the calibration sample and the stability of the electrospray through an integrated tip (Figure 2). The TIC from the device using the sacrificial template method yields better electrospray stability (Relative standard deviation (RSD) = 9%) than that from the device using the bonding method (RSD = 23%).

References:

Integration of Microfluidics to Electrospray Ionization Mass Spectrometry Using a Chip-Embedded SU-8 Electrospray Tip

CNF Project # 762-99
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User: Seung-Min Park

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- Polymeric microfluidic device coupled to mass spectrometry.
- Polymer electrospray ionization tip.
- Encapsulation: Direct bonding, sacrificial layer removal.

Figure 1: (A) SEM image of the electrospray tip. (B) SEM image of the channel cross-section. (C) Optical images of the channel filled with liquid and the electrospray tip. (D) Device picture.

Figure 2: (A) TIC (RSD = 23%) from the device fabricated with a direct bonding method. (B) TIC (RSD = 9%) from the device fabricated with a sacrificial method. (C) Mass spectrum of the calibration sample.
Micromachined Test Patterns for Rapid Screening of Etchants

CNF Project # 1130-03
Principal Investigator: Melissa A. Hines

Abstract:
A new technique for the production of large (~1 µm x 50 µm), regularly spaced atomically flat regions on vicinal silicon surfaces has been developed. This technique makes use of patterned etch barriers to control the flow of atomic steps during aqueous etching.

Summary:
Aqueous processing is crucial to today’s microelectronics industry, accounting for almost 20% of worldwide semiconductor chemicals sales. Aqueous etchants are the basis of current wafer cleaning and surface planarization processes, and they find specialized applications in bulk micromachining, the production of ink-jet nozzles, the texturing of high-performance solar cells, and the fabrication of nanoscale transistors. As an inherently low cost, low temperature technology that can, under optimum conditions, produce surfaces of atomically controlled chemistry and morphology, aqueous chemistries have tremendous potential in nanoscale processing, particularly for the control of surface morphology and shape. In spite of this potential, aqueous processes are underutilized in nanoscale R&D, as new protocols require tedious trial-and-error development, primarily because these chemistries are poorly understood—even the concentration dependence of the etch rate has defied explanation!

Using a combination of aqueous etching and microfabricated etch barriers, we have demonstrated the production of continuous trains (2 mm long) of atomically flat regions (~1 µm x 50 µm) on vicinal silicon wafers. The atomically flat regions have a surprisingly regular spacing, as shown by an analysis of the terrace width distribution, suggesting a kinetically driven, long-range self-organization whose origin is under investigation. The width of the terraces is controlled by the etch time.

This discovery was enabled by our development of a pseudo-combinatorial technique which uses an array of etch barriers of differing orientation. Interestingly, the orientation of the etch barriers has a profound effect on surface morphology. For example, the orientation of the macrosteps that separate each atomically flat region is controlled by the orientation of the etch barrier, not by the miscut orientation; an unmasked surface etches to form an entirely different surface morphology. The regularity of the trains is also a function of barrier orientation. These findings illustrate the subtlety of these effects and the need for predictive indicators of etch morphology. The development of a computational model of this process is underway.

References:
**Micromachined Test Patterns for Rapid Screening of Etchants**

CNF Project # 1130-03  
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- Large, atomically flat regions on a vicinal silicon surface produced by chemical etching.  
- Width of regions controlled by etch time.  
- Rapid and economical.

**Figure 1:** AFM image of a train of atomically flat, chemically produced terraces on a vicinal silicon surface.

**Figure 2:** A statistical analysis of the chemically etched surface in Figure 1 shows remarkable regularity in the widths of the etched terraces.
**Thermal Integration Issues in Micro Fuel Processor: Development of an Integrated Silicon Microreactor Based Methanol Steam Reformer as a Model**

**CNF Project # 1184-04**
**Principal Investigator: Prof. Ronald S. Besser**

**Abstract:**

Miniature fuel cells offer promise for the conversion of chemical energy into electrical energy in a highly efficient way, achieving the goals of light weight and low volume. However, the success of fuel cell technology for portable power depends heavily on development of an efficient way of delivering fuel to the cell.

The processing of high density liquid hydrocarbon fuels appears to be the most promising method of supplying a hydrogen stream for feeding portable fuel cells. Because of their size and portability, microreactor technology has shown promising results in the field of fuel processing to convert hydrocarbon fuels to hydrogen, for the production of electricity.

Thermal management in miniature systems is perhaps the most crucial challenge for microscale fuel processors [1]. The fuel processor needs effective thermal coupling to allow transfer of energy from the heat producing combustor to the endothermic steam reformer. Coupling endothermic and exothermic components of the fuel processor and minimizing losses can achieve a high thermal efficiency. However, such coupling must be accomplished in a manner that permits the maintenance of specific temperatures in the various components and maintains the surface of the package near room temperature. Microreactors generally offer high heat transfer rates mainly because of high surface-to-volume ratio and short conduction paths. This characteristic results in efficient heat extraction but at the same time results in higher heat losses to the ambient. Therefore, thermal management offers a dual challenge of opposing the heat losses from the system that arise from high surface-to-volume ratio in conjunction with maintaining temperature gradients within the system to allow desired conditions in the unit reaction steps.

**Fabrication:**

In this study, a silicon microreactor-based catalytic methanol steam reforming reactor was designed and fabricated in the context of complete thermal integration to directly address the heat management issue. The design is made where vacuum packaging chips, thin film heater, and temperature sensors are directly embedded with the microreactor to simulate an integrated steam reformer in an overall fuel processing scheme.

Silicon microfabrication technology was used to fabricate reforming microreactors, vacuum packaging chips, thin-film heaters and temperature sensors on silicon and glass substrates. Four microreactors were fabricated on a single silicon wafer by silicon bulk micromachining techniques using photolithography followed by DRIE, where these structures were etched to a depth of 400 µm. Once the front side was processed, a second mask was used for backside patterning of inlet and outlet vias. These vias were then opened from the backside by wafer through-etching with DRIE. The thin film Pt resistive temperature device type sensors were fabricated on backside of this microreactor wafer. The microreactor wafer was then sealed with a Pyrex® cover by anodic bonding. The bonded wafer was then diced into individual microreactors of size 3.2 cm x 2.0 cm. For the surrounding insulation, a square cavity was patterned on the silicon wafer and etched to the required depth using DRIE. A thin-film Pt heater and temperature sensors were fabricated on Pyrex® glass wafers. The Pyrex® glass wafers patterned above with sensors and heater were bonded to enclose a vacuum cavity in Si. The bonded device was then diced into an individual insulation chip of 1.1 cm x 1.5 cm. Figure 1 shows the fabricated components of an integrated device. The catalyst particles were then incorporated into the silicon microreactor to create a packed bed reaction zone. Figure 2 shows the microreactor packed with catalyst particles of mean diameter 60-80 µm. Next, the insulation chips are bonded on the front and backsides of the catalyst loaded microreactor using high temperature epoxy. Figure 3 shows the microreactor bonded with the insulation chips.

Detailed experiments are carried out to quantify heat losses through various pathways from the planar microreactor structure. The result provides fundamental insight in understanding of critical thermal transfer issues of an integrated microreactor system such as transfer of heat between reactor components, control of temperature, insulation, and heat losses. Based on this understanding, suggestions are made for scale up of reactor components and a packaging scheme for reduction of convective and radiative losses.

**Summary:**

The management of heat in a compact format is perhaps the most crucial challenge for microscale fuel processors for portable hydrogen production. A Si microreactor-based catalytic steam reforming reactor was designed and fabricated in the context of complete thermal integration to directly address the heat management issue.

**References:**

Thermal Integration Issues in Micro Fuel Processor: Development of an Integrated Silicon Microreactor Based Methanol Steam Reformer as a Model

CNF Project # 1184-04
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- Silicon MEMS technology was used to fabricate an integrated thermochemical device to understand heat management issues in small scale fuel processing.

- Thermal characterization of integrated reformer was carried out for the acquisition of critical thermal parameters of the overall coefficients for the transfer of heat from system fluids to one another and to the outside environment by different means of heat loss.

- Based on thermal measurements, an experimental correlation was derived to predict natural convection heat transfer coefficient from surfaces of millimeter to submillimeter scale devices.

Figure 1, above: Fabrication of an integrated steam reformer using silicon microfabrication techniques.

Figure 2, top right: Incorporation of catalyst into microreactor.

Figure 3, bottom right: Silicon microreactor bonded with insulation chips.

Packed bed of catalyst

Figure 2

Figure 3
Fabrication of Inorganic-Organic Interfaces

CNF Project # 1239-04
Principal Investigator: James R. Engstrom

Introduction:

Interfaces have always governed the performance characteristics of solid state devices. Right from a simple diode to complex interconnects, inorganic-inorganic interfaces have defined the semiconductor technology to date. However, as the devices shrink and their dimensions approach the molecular regime, attempts are being made to use organic molecules as active components of electronic circuitry [1]. Organic molecules are attractive owing to their property of self-assembly; but to use them as conductors, insulators and ultimately in transistors, the challenge is to integrate them with inorganics-fabricating reliable inorganic-organic interfaces and making macroscopic contacts to the microscopic world.

Summary:

Whereas, “self-assembly” (chemically specific adsorption) has been used to successfully make a contact between organic molecules and inorganic substrates, often more crude means are adopted to make the second (top) contact. In the case of metals, evaporation or sputter deposition of elemental metal is the preferred route but it has been shown to penetrate the organic film and hence is not a very effective technique. We are addressing these bottom and top contact problems individually.

For the bottom contact on metals, we have carried out the self-assembly of tailor-made conjugated, long chain thiophene molecules on Au. In particular, a thiophene headgroup has been chosen for its affinity for Au surfaces [2]; isopropylamine or hydroxyl terminal functional groups can serve as potential nucleation sites for subsequent surface modification, and a phenylene ethynylene backbone can provide electrical conduction. Thiophenes offer potential advantages over the related (more commonly studied) thiols which may be reduced to thiolates [3] or oxidized to disulfides [4]. These organics-on-metal interfaces have applications in molecular electronics.

In a parallel study, to build organic-on-inorganic architectures, we have grown covalently attached molecular interfacial layers on SiO$_2$ and other low dielectric constant films (e.g. - methyl silsesquioxane) using hyperbranched polymeric growth. These films have a branched covalent backbone and a high terminal group density.

In order to obtain -NH$_2$ functionalized branched SAMs, firstly a thin layer of SiO$_2$ was grown on freshly cleaved Si wafers. The hydrophilic Si wafers thus obtained were used to grow a monolayer of (3-aminopropyl)ethoxydimethylsilane (by forming Si-O-Si bonds). This -NH$_2$ terminated monolayer is termed as Generation 0. The wafers were then treated with an acrylate to convert amine termination to a branched ester termination (Generation 0.5). Following this, the samples were immersed in a diamine solution to re-obtain -NH$_2$ terminal functionalities (in twice the density as Generation 0). This organic film is termed as a Generation 1 SAM. Such organic-on-dielectric architectures can find applications in the pore sealing of low-κ dielectrics.

Our approach towards the reverse interface formation, inorganic-on-organic, is to make use of transition metal coordination complexes to initiate top contact formation with these organic self assembled monolayers (SAM) both on metals and dielectric films. We have been examining the reactions of transition metal coordination complexes, such as Ti[N(CH$_3$)$_2$]$_4$ and Ta[N(CH$_3$)$_2$]$_5$, with SAMs possessing different head group chemistries [e.g., RSiCl$_3$ on SiO$_2$, R-(thiophene) on Au] and endgroup functionalities (e.g., -CH$_3$, -NH$_2$, (-NH$_2$)$_2$ and -OH) in order to develop an understanding of interface and contact formation [5]. We have used x-ray photoelectron spectroscopy (XPS) to probe the nature of SAMs that are formed, quantify the kinetics of adsorption of the Ti and Ta complexes on these layers, and determine the specificity and/or spatial extent of reaction between the complex and the SAMs. Our results indicate that transition metal complexes can bind very specifically and non-invasively to the endgroups on self-assembled monolayers. The seamless metal-organic-metal architectures have applications in molecular electronics while metal-organic-inorganic may be used for pore sealing of low-κ dielectrics.

References:

Fabrication of Inorganic-Organic Interfaces

CNF Project # I239-04
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- Specifically designed long chain conjugated thiophene molecules with isopropylamine and hydroxyl terminations were self-assembled on Au. The thiophene with a longer backbone (2 benzene rings) was found to form denser monolayers.

- Covalently bound, hyperbranched, functionalized, polymeric organic films were grown on SiO$_2$. A quantitative analysis of the XPS spectra for the films was carried out after each reaction step to analyze the increase in terminal group densities with an increase in film thickness.

- The organic films, both on metal and inorganic substrates were exposed to the organometallics of Ta and Ti to carry out a gas-surface reaction in a UHV environment. The reaction was found to be self-limiting, site-specific and directly dependent on the terminal group density of the SAM leading to the chemisorption of Ta/Ti at the SAM-vacuum interface.

Figure 1: (a) Schematic for the synthesis of -NH$_2$ terminated branched self assembled monolayers on SiO$_2$. (b) XPS spectra for N(1s) peaks for Generations 0 and 1. Following a Shirley background subtraction, the spectra have been fitted to respective Gaussian-Lorentzian product functions shown by smooth curves. The G-L fits satisfactorily define single N(1s) peaks.

Figure 2: (a) A Coverage-exposure relationship, deduced from XPS, for the adsorption of Ti[N(CH$_3$)$_2$]$_4$ on the 2P SAM, both for $T_s$ = 50 and 30°C. The fits to the data, shown as smooth curves, are for first-order Langmuirian kinetics. (b) Integrated peak areas for the Ti(2p) region as a function of take-off angle for a saturation exposure of the 1P (filled symbols) and 2P SAMs (open symbols) to Ti[N(CH$_3$)$_2$]$_4$ at $T_s$ = 30°C. (c) XPS spectra for Ta(4f) feature for gen 0 and gen 1 SAMs after exposing the corresponding surface grafted monolayers to Ta[N(CH$_3$)$_2$]$_5$ at 25°C. (d) ARXPS data for Ta(4f) feature for gens 0 and 1. The data points are the integrated peak areas of Ta(4f) spectrum as a function of take-off angle. The smooth curves are the fits assuming a uniform deposition of Ta at a depth d from the surface with $\lambda$ being the inelastic mean free path for Ta(4f) electrons.
Microfluidic Mixing

CNF Project # 1278-04
Principal Investigator: Abraham D. Stroock

Abstract:

The ability to control chemical processes in microliters of fluid would benefit a broad range of chemical technologies, such as portable chemical analysis with clinical and security applications, portable power supplies in the form of fuel cells, and miniature reactors for combinatorial chemistry. A major challenge in developing chemical operations on this scale comes from the fact that flows of simple fluids through small structures (> 1 mm) are dominated by viscous damping and are thus laminar (non-turbulent). In this regime, mixing does not occur spontaneously and the rate of chemical processes is likely to be limited by the rate of molecular diffusion between distinct regions of the fluid. Our work focuses on the development of passive micro-structures that, when present inside a microfluidic channel induce efficient mixing even under laminar conditions [1].

Summary:

Here we report on the application of this concept to a micro fuel cell [2]; the fabrication challenge is to build the appropriate micro-structure directly onto the electrodes of the fuel cell. This project is in collaboration with Prof. Hector Abruna of the Chemistry Department at Cornell University.

Under optimal conditions, fuel cells are operated such that the current is limited by the rate of diffusion of fuel and/or oxidant to the electrodes. In this situation, the creation of three dimensional flows in the vicinity of the electrodes increases the rate of this mass transfer process and thus improves the current and power density achieved at a given efficiency of fuel usage. We have recently developed a theoretical understanding of this process [3].

Over the past year, we have developed a fabrication process with which to create the appropriate topography on electrically insulating substrates (glass) on which we can deposit metals and electrocatalysts to form the anode and cathode of our fuel cell. The mixing structures are created by etching herringbone shaped grooves into quartz wafers (4", 750 µm thick, Mark Optics) as follows: A hard mask (~ 400 nm) of amorphous silicon (a-Si) was deposited by plasma enhanced chemical vapor deposition (PECVD) onto the quartz in a load locked GSI PECVD system. The EVG 620 contact aligner was used to define the windows in the a-Si mask through which the grooves are to be etched. An SF$_6$ / O$_2$ reactive ion etch was used to etch through the a-Si in the pattern left by the developed photoresist. After stripping the photoresist, the underlying glass is etched isotropically to the desired depth (~ 50 µm) in hydrofluoric acid (HF) or buffered oxide etch (BOE, 6:1 NH$_4$F:HF). The resulting grooves are smooth and essentially cylindirical or spherical due to the isotropic undercut of the etch. The metallization of the electrode surface and contacts was achieved by electron beam evaporation.

We have demonstrated that these electrodes are appropriate for generating chaotic, three dimensional flows. We are in the process of characterizing their function within the fuel cell.

References:


Microfluidic Mixing

CNF Project # 1278-04
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Goals:

1) Investigate fundamental questions related to mass transfer in microfluidics and,

2) Optimize mass transfer to reactive boundaries for applications in fuel cells and biosensors.

Figure 1: Chaotic mixing to increase current in a Planar Membraneless Micro Fuel Cell.
(A) Schematic illustration of cross-section of a fuel cell with topographically patterned electrodes that generate chaotic flows within the streams of oxidant and fuel.
(B) Computed concentration distribution of fuel species as it progresses through fuel cell.

Figure 2: Mixing structures formed by isotropic etching of glass. (A) Optical micrograph of grooves in glass. The image shows four cycles of the mixing structure with four units of the Staggered Herringbone Mixer running side-by-side. Scale bar is 1 mm. (B) Fluorescence micrograph showing evolution of a narrow stream of fluorescent solution in the 3-D flow generated over the region indicated with a dotted outline in (A). The patterned glass was the floor of a 120 µm-deep microchannel in PDMS. Scale bar is 250 µm.
Shearing Confined Colloidal Suspensions Using Microfabricated Templates

CNF Project # 1361-05
Principal Investigator: Itai Cohen

Abstract:

Microfabricated PMMA templates prepared using photolithography and reactive ion etching techniques are used to structure colloidal suspensions of micron-sized particles. Two parallel patterned plates confine the suspension while the structure and dynamics of the constituent particles are monitored in realtime using confocal microscopy. By applying an oscillatory force to one plate while holding the other fixed, the effects of shearing on the confined suspension are probed.

Summary:

Recently, microfabricated templates have been successfully used [1] to structure sedimented colloidal particles. This technique allows for accelerated colloidal crystallization, which has been exploited to study the effects of various boundary conditions, including stretched templates [2] and surfaces with embedded grain boundaries [3].

In our current research project, we fabricate PMMA templates on both silicon wafers and silica glass substrates. A suspension of 1 µm diameter PMMA colloidal particles is then sandwiched in a 1 to 50 µm gap between the glass and silicon templates. By using index-matched PMMA on glass, we enable confocal microscopy of the colloidal suspension through one of the patterned plates. This technique allows direct 3D imaging of the particle positions and dynamics of the confined suspension.

The fabrication process involves both photolithography and reactive ion etching. Photoresist layered on PMMA is exposed using a custom-designed mask and is then developed. The remaining photoresist is then used as a mask for the PMMA, which is etched away using oxygen plasma reactive ion etching. The photoresist is finally flood exposed and developed away, leaving the desired pattern in the PMMA film.

Two kinds of templates are used to probe different behavior in dense colloidal suspensions. A square lattice of micron-sized holes is used to grow an FCC crystal along the <100> axis; the colloidal crystal is then slowly sheared between the patterned plates and shear-induced structural rearrangements are imaged. Alternatively, we pattern templates of randomly positioned micron-sized holes, which are used to prevent crystallization between the plates. This enables the investigation of glassy and supercooled confined colloidal suspensions. Again, the effects of shearing on glassy suspensions is monitored in realtime on the particle scale using confocal microscopy.

Our technique allows us to address some of the most compelling questions in glassy physics. One defining characteristic of glassy systems is the observation of non-exponential correlation functions using relaxation techniques. There are two dominant models for this behavior. In the first, the system is spatially heterogeneous with respect to relaxation times, but does not evolve in time. The second model describes a system in which one region is first characterized by a particular relaxation time, then by another at a later time. These models have been explored in polymeric glasses using such techniques as multidimensional NMR [4], but the issue remains unresolved. Our direct visualization experiments on confined colloidal glasses allow us to address this active question in glassy physics. We watch a single region of the system and monitor its characteristic relaxation time as the glass ages. The patterned templates we fabricate at CNF are essential to this technique, since they grant us the unique ability to confine our colloidal suspensions while simultaneously jamming them and preventing crystallization.

Our work on glassy systems is supported by NSF grant DMR0606040.

References:

Shearing Confined Colloidal Suspensions Using Microfabricated Templates

CNF Project # I36I-05
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- Microfabricated templates structure dense colloidal suspensions.
- Square lattice of holes accelerates crystallization.
- Randomly distributed holes aide glass formation.
- Confocal microscopy enables direct visualization.
- Effects of shearing are probed using piezoelectric stage.

Figure A, top left: A square lattice of 1 µm diameter circular holes etched into PMMA.

Figure B, bottom left: One layer of an FCC colloidal crystal of 1 µm diameter particles.

Figure C, above: A random distribution of 1 µm diameter circular holes etched into PMMA.
1. Solid Memory: Structural Preferences in Group 2 Dihalide Monomers, Dimers and Solids. Kelling J. Donald and Roald Hoffmann (paper submitted to Journal of the American Chemical Society.)

In this study, the link between structural preferences in the monomers, dimers and extended solid state structures of the group 2 dihalides (MX₂: M = Be, Mg, Ca, Sr, Ba and X = F, Cl, Br, I) is examined theoretically. The question posed is how well are geometric properties of the gas phase MX₂ monomers and lower order oligomers “remembered” in the corresponding MX₂ solids? Significant links between the bending in the MX₂ monomers, the D_{2h}/C_{3v} M₂X₄ dimer structures are identified. At the B3LYP computational level, the monomers that are bent prefer the C_{3v} triply-bridged geometry, while the rigid linear molecules prefer a D_{2h} doubly-bridged structure. Quasilinear or floppy monomers show, in general, only a weak preference for either the D_{2h} or the C_{3v} dimer structure. A frontier orbital perspective, looking at the interaction of monomer units as led by a donor-acceptor interaction, proves to be a useful way to think about the monomer-oligomer relationships. There is also a relationship between the structural trends in these two (MX₂ and M₂X₄) series of molecular structures and the prevalent structure types in the group 2 dihalide solids. The most bent monomers condense to form the high co-ordination number fluorite and PbCl₂ structure types. The rigidly linear monomers condense to form extended solids with low coordination numbers, 4 or 6. The reasons for these correlations are explored.

This work made use of CNF computation facilities.


A way to circumvent the high pressures needed to metallize hydrogen is to “precompress” it in hydrogen-rich molecules, a strategy probed theoretically for silane. We examined a total of thirteen possible structures for silane at high pressure computationally using density functional theoretic approach. We show that phases with tetrahedral SiH₄ molecules should undergo phase transitions with 6- and 8-coordinate Si appearing above 25 GPa. The most stable structure (O3 shown) found can be metallized at under a megabar, and at a compression close to the prediction of Goldhammer-Herzfeld criterion, which is based on very simple physical picture and has been surprisingly useful. According to a BCS-like estimate, metallic silane should be a high temperature superconductor. More crucially, we found that the T_c increases rapidly with pressure.

A part of this project made use of the CNF computing facilities.
The Electronic Structure of Molecules and Extended Systems

CNF Project # I371-05
Principal Investigator: Roald Hoffmann
Users: Ji Feng, Kelling Donald

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Structural Preferences in the Group 2 Dihalide Gas Phase Monomers and Dimers Systems and Extended Solids at Ambient Conditions.
GaN Ballistic Electron Acceleration Negative Differential-Conductivity Diodes for THz Applications

CNF Project # 370-89
Principal Investigator: Dr. Lester F. Eastman

Abstract:
Electronic generation of THz signals using GaN is predicted to exhibit high efficiency. Theory shows that electrons can be ballistically accelerated to the Brillouin zone energy limit of 2.7 V [1,2], and above 1.0 V, they will pass beyond the inflection point and have negative effective mass. Experiments by M. Wraback [3] indicate the presence of negative differential resistance (NDR). Finite energy injection at 0.4 to 0.6 eV launches electrons closer to the inflection point to increase the ratio of negative to positive mass electrons.

Summary:
First generation devices were n⁺-i-n⁺ type structures (2 x 10¹⁹/cm³ n⁺ doping) with high contact and spreading resistances that prevented establishment of the necessary amount of voltage drop across the intrinsic layer (i-layer) to push electrons beyond the inflection point. The domination of the parasitic resistances in the device behavior is seen for the I-V comparison of an actual device with a planar no-mesa diagnostic version. Almost all of the bias voltage drops across the parasitics and only a small portion of it falls across the i-layer. Moreover, bias voltages above approximately 3 volts result in heating at the contact to a high enough temperature to vaporize the connection at the air bridge.

The high contact resistances to n⁺ regions were unexpected because the same ohmic contact technology used for AlGaN/GaN FET’s was employed. A series of TLM experiments with various metals were performed and as-deposited Ti/Al/Mo/Au proved to be the best with a specific contact resistance of 4.92 x 10⁻⁸ ohm·cm² for 1.26 x 10²⁰/cm³ n⁺ doping. This showed a hundred to one reduction in the contact resistance. Second generation devices included an AlGaN launcher to increase injection energy. The design utilized non-alloyed Ti/Al/Mo/Au ohmic contacts on the AlGaN/GaN heterostructures. Long periods of operation at high bias resulted in change in parasitic resistance. While bottom ohmic contacts exhibited low resistance, the hotter top contact was found to be the source of the unstable high resistance. It is not conclusive how much role the unstable, high resistance anode ohmic contact plays in the observation of the negative differential resistance.

We usually process 15 x 15 mm square samples cut out from a 2 inch GaN wafer. GaN is grown on sapphire substrates using our own MBE facility in Phillips Hall. The epilayer consists of doped and undoped GaN and AlGaN layers. Thus far we’ve tried both n⁺-i-n⁺ structures and AlGaN/GaN heterostructures, both having an i-layer thickness ranging from 600Å to 300Å. Fabricated devices have a circular active region, defined by a dry etch process (chlorine based ICP). The diameters of these devices are 3, 5, 10, 15 and 25 µm. Then, two different interconnect types were fabricated after the deposition of the ohmic contacts. First type required the deposition of a 2 µm thick silicon dioxide layer to provide the dielectric medium for the fabrication of on-chip microwave resonators. These were attached to some of the devices to force them to oscillate at certain frequencies in case NDR is present. Sloped holes in the dielectric were then drilled to make electrical contact to the devices through appropriate size cascade probe pads. The other type employed a standard airbridge process connecting the two terminals of the devices directly to these pads.

References:
GaN Ballistic Electron Acceleration Negative
Differential-Conductivity Diodes for THz Applications

CNF Project # 370-89
Principal Investigator: Dr. Lester F. Eastman
User: Barbaros Aslan

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- GaN ballistic electron acceleration device.
- Heating at the contacts is a problem.
- Current decreases with increasing voltage at around 1.8-1.9 volts. It may be genuine NDR predicted by the theory or some other current limiting mechanism due to extremely high temperatures reached at the contacts.

Figure 1, top left: SEM image of a device.
Figure 2, bottom left: SEM image of a blown contact.
Figure 3, above: CW I-V characteristics showing five successive voltage sweeps.

Figure 1

Figure 2

Figure 3
Fabrication of Poly-Si TFT on Flexible Thin Glass Substrates

CNF Project # 564-95
Principal Investigator: Dr. Dieter G. Ast

Abstract:

Moderately flexible liquid crystal displays (LCD’s) are thinner, lighter and require less protective packaging than today’s rigid LCD displays. All flexible displays developed to date use either a plastic substrate covered with an inorganic layer (SiO$_x$, SiN$_x$) or a metal foil substrate. Amorphous Si:H can be deposited at temperatures below 300°C and therefore a-Si:H based active matrix LCDs, as well as solar cells, have been fabricated on plastic substrates. Plastic substrates are not a suitable material for low cost poly silicon (poly-Si) thin film transistors (TFTs) that require processing at several hundred degrees. The work-around to date has been to cover the polymer substrate first with a 1 µm inorganic layer on which a much thinner a-Si layer is subsequently deposited at low temperatures. The a-Si is then crystallized into a poly Si layer by pulsed laser annealing. The underlying inorganic film, which is 10 times thicker, reduces the peak temperature at the surface of the polymer, to first order by the thickness ratio; ~ 200 to 300°C.

This approach poses several problems, the most difficult one being that the coefficient of thermal expansion (CTE) of plastic substrates far exceeds those of the inorganic semiconductors and insulators. Another problem is coating the polymer with a a well adhering, crack free, 1 µm thick inorganic layer. The deposition of a high quality gate dielectric at temperatures compatible with the polymer is difficult as well.

Summary:

The alternative approach pursued here is to fabricate the thin film electronics on a flexible thin glass substrate that can be bent to a radius of curvature smaller than 3 inches. After fabrication of the circuit, the glass substrate is bonded to a polymer substrate. The approach avoids heating the polymer and all the associated problems that come with it.

Problems to be solved in this approach are: i) suppression of the out-migration of unwanted glass constituents from the glass into the semiconductor during high temperature processing, ii) the accommodation of the smaller but still existing CTE difference between the various materials, and, iii) the handling of 60 to 100 µm thick glass substrates during the TFT fabrication process, such as MOS cleaning, lithography and film deposition.

To prevent impurities from the glass entering the active poly-Si layer we coat the glass on both sides first with 100 nm of PECVD SiN$_x$ and then with 100 nm of low temperature (400°C) SiO$_x$, deposited by LPCVD from diethylenesilane. The purpose of the SiO$_x$ is to provide a low recombination velocity at the poly-Si/SiO$_2$ interface (low back channel leakage) and to getter any Na that might out-migrate from the glass substrate (Corning 00 Microsheet). These dual barrier layers were found to work well up to 580°C. At higher temperatures, the rapid increase in the CTE of 00 Microsheet as it approaches the glass transition stresses the barrier layer to the point where it cracks with a concomitant penetration of boron into the active layer (see its SIMS profile in Figure 1). These cracks cannot be seen once the sample is removed from the reactor as the barrier layer goes into compression during cool down and the cracks close. A work-around is to deposit the barrier layers at higher temperatures, but given the available equipment, this approach can not be implemented at Cornell.

To process the thin glass sheets in conventional semiconductor equipment we cut the thin glass into rectangles, which were then inserted into a retaining “pocket” fabricated on a standard Si wafer. The “pocket” consists of Si “rails” that are electrostatically bonded with a Pyrex® spacer to the silicon wafer (Figure 2). This approach, developed after a series of experiments with other methods (e.g. Ga-In bonding, which floats the glass on a liquid layer during processing) works well and permits a problem-free wet process of the glass as needed.

The TFT process is using a triple path:

1. A base line process using oxidized Si wafers. Its major purpose, besides equipment check, is to serve as test bed for improving the LT gate oxide and the hydrogenation treatment of the channel poly.

2. A two step process in which we first deposited Si from silane at 580°C by LPCVD. At this temperature, the deposit contains widely spaced, crystalline nuclei dispersed in an amorphous matrix. The material is then crystallized into a relatively large grain size (for CVD) poly-Si with a 24 hr, 620°C anneal.

3. An LPCVD process in which a-Si is deposited from silane at 500°C, and then laser recrystallized. Because of the much lower hydrogen content, LPCVD deposited a-Si is very much easier to laser anneal than the PECVD a-Si: H which one is forced to use with polymer substrates. The crystallinity of these three materials, as revealed by x-ray diffraction, is shown in Figure 3.

Figure 4 shows the electrical characteristics of a base line TFT made with the 580/620 process. Figure 5 shows those for a TFT made on 00 Microsheet using 580°C poly-Si but omitting the 620 anneal (which, as discussed above, introduces barrier layer failure). Data on excimer laser recrystallized devices will be available very shortly and can be obtained by e-mailing the authors. Because failure of dual barrier layers at T > 580°C our future work will concentrate on laser recrystallized TFTs.

Acknowledgments:

This research was made possible by a gift from Corning Inc. and made possible by the facilities available at the CNF, an NSF supported NNIN facility.
Fabrication of Poly-Si TFT on Flexible Thin Glass Substrates

CNF Project # 564-95
Principal Investigator: Dr. Dieter G. Ast
User: Yoochul Jung

Affiliation: Department of Material Science and Engineering, Cornell University
Primary Funding: Corning Inc.
Contact: dast@ccmr.cornell.edu, yj29@cornell.edu

Figure 1, top: SIMS profile of triple layer of poly-Si / SiO₂ / SiNx deposited on Microsheet glass after a 24 hr 620°C anneal.

Figure 2, middle: Fixture to process MS glass substrates in 6" semiconductor equipment. Fabricated by electrostatic bonding.

Figure 3, bottom: X-ray diffraction data for LPCVD silicon: (a) deposited at 580°C, (b) annealed at 620°C; (c) LPCVD silicon deposited at 500°C and (d) excimer laser annealed.

Figure 4, top: Electrical characteristics of a TFT fabricated with the base line process.

Figure 5, bottom: Electrical characteristics of a TFT fabricated in partially crystallized silicon (LPCVD, 580°C).
3-D Electrostatic Modeling in Metal Nanocrystal Memory

CNF Project # 715-98
Principal Investigator: Edwin C. Kan

Abstract:
Although theoretical models of nanocrystal (NC) memories have been investigated by several groups [1,2], only 1-D electrostatic models were employed, despite the very nature of the 3-D spherical NCs and their 2-D arrayed distribution. In this work, we establish a physical model based on the 3-D electrostatics. We demonstrate, by replacing SiO$_2$ with HfO$_2$ as the control oxide, that continuous control oxide scaling is possible with improved program/erase (P/E) efficiency and retention time owing to the unique 3-D electrostatic effects. The results confirm 3-D electrostatics instead of 1-D should be considered in NC memory modeling.

Summary:
Numerical solution of the 3-D electrostatics in the NC memories is developed to calculate the electrostatic potential, the single-electron charging energy $E_c$, and the 3-D channel-control factor $R_{3D}$ [2], which can not be quantitatively addressed in the previous 1-D models. $R_{3D}$, less than 1 in general, is a correction factor to the classic flatband voltage shift ($\Delta V_{FB}$) model in the continuous floating-gate devices, because the charges in NCs can only perturb the channel potential in a smaller effective coverage area less than the NC unit cell area but significantly larger than the NC cross-section area due to 3-D fringing field. Meanwhile, the tunneling calculation at the least-action path is carried out by modified 1-D Wentzel-Kramers-Brillouin (WKB) approximation. The P/E and retention characteristics are determined from time-dependent, self-consistent tunneling current because the potential has to be updated due to the Coulomb blockade effect whenever electrons are in or out of the NCs.

Good agreements have been shown between the simulated and experimental programming transients of an Au metal NC memory. The device fabrication, similar to one reported previously [3], was done in the Cornell Nanoscale Science and Technology Facility (CNF). The simulation parameters are taken or close to those from various types of physical characterization in the given sample, which validates the accuracy of our formalism.

The scaling of the control oxide effective oxide thickness (EOT) is necessary to reduce the memory cell size. In the NC memories, high coupling ratio by scaling $T_{conf}$ may allow more charges stored in NCs at self saturation, but does not guarantee larger $\Delta V_{FB}$, which is influenced by the combined effect of charge density, $T_{conf}$, and $R_{3D}$. Even more importantly, if $E_c$ does not scale much with $T_{conf}$, more charges stored in NCs may adversely affect retention characteristics.

High-$\kappa$ dielectrics as the control oxide can provide an effective solution through the unique 3-D electrostatic nature. Lower $E_c$ and larger $R_{3D}$ with HfO$_2$ are found in our simulation. It can be understood by the much stronger fringing field through HfO$_2$ to the Si channel due to the higher dielectric constant of HfO$_2$. This substantially increases the substrate-NC coupling capacitance and the effective channel coverage area under the influence of the charges stored in NCs. With the same number of charges stored in NCs, large $R_{3D}$ benefits the programming efficiency by allowing larger $\Delta V_{FB}$, while lower $E_c$ improves the retention characteristics by maintaining larger band offset between the NC fermi-level and the Si conduction band.

References:
3-D Electrostatic Modeling in Metal Nanocystal Memory

CNF Project # 715-98
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User: Tuo-Hung Hou
Affiliation: School of Electrical and Computer Engineering, Cornell University
Primary Funding: NSF
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Figure 1, top left: Good agreement between modeled and experimental programming transients in a metal NC memory with a single set of simulation parameters.

Figure 2, bottom left: Improved programming efficiency with HfO$_2$ as the control oxide.

Figure 3, above: Improved room-temperature retention with HfO$_2$ as the control oxide.
Electrolyte Pulse Current Measurements by CvMOS with Microsecond and Thermal Voltage Resolution

CNF Project # 752-98
Principal Investigator: Edwin C. Kan

Abstract:

Non-invasive, charge-based sensing in chemoreceptive neuron MOS (CvMOS) transistors with extended floating-gate structure has brought forth features that are beneficial to the system integration of biological sensing. This paper presents the results of fast electrolytic signal detection on silicon dioxide, which advances possible technologies for rapid DNA discrimination or external monitoring of cell action potentials.

Summary:

Non-invasive, integrated electronic detection of ion transport in biological systems has many promising applications including electrophoretic molecule recognition, entropic channels, fast DNA sequencing, and recording of cell action potentials [1-5]. The basic difficulties of electrical sensing lie in the signal-to-noise ratio, memory effects, and contamination isolation in the fluidic system. In contrast to sensing through a direct DC or AC current [6] such as the stripping potential method, CvMOS uses capacitive coupling between the sensing gate and the extended floating gate for transducing, which can potentially reduce invasiveness and help fluidic isolation. For the demonstration of the operational principles and achievable sensitivity, SiO$_2$ is used on top of the polysilicon sensing gate with the understanding that the material selection can be further optimized.

The CvMOS structure reduces the invasiveness of ion transport detection while maintaining the advantages of high transconductance available from FET-based sensors (see Figure 1). The inclusion of several sensing gates coupling with a single floating gate creates a neuron-like effect, analogous to several weighted inputs connecting to a single node. The CvMOS device was fabricated through a foundry service with the AMIS 1.5 µm process.

The microfluidic delivery chamber (Figure 2) involves a four-mask process. A silicon-rich nitride membrane separates two silicon wafers bonded by PDMS. The nitride membrane houses the inlet and outlet pores, which deliver the fluid to the analyte chamber.

The extended floating gate structure of CvMOS allows for fast electrolytic current sensing. In a step towards integrated rapid DNA discrimination, we have demonstrated that a non-optimized CvMOS design can detect 10s of nA current at 1 microsecond and 25 mV, at which point our present instrumentation limit was reached. System requirements for rapid DNA sequencing include 0.1-1.0 microsecond detection for 100s of pA at 100s of mV. With optimization and further characterization, a fully CvMOS-compatible DNA sequencer can be likely realized.

References:

Electrolyte Pulse Current Measurements by $\text{C}_n\text{MOS}$ with Microsecond and Thermal Voltage Resolution

CNF Project # 752-98
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Figure 1: Schematic of a $\text{C}_n\text{MOS}$ transistor. The MOS area has similar fabrication to commercial flash memory cells. Fluids are delivered to the sensing gates through superimposed microfluidics.

Figure 2: Sensor response to a 1 microsecond electrolytic signal at 25 mV, which corresponds to an electrolytic current on the order of tens of nA. Sensor demonstrates adequate sensitivity for BLM-based DNA sequencing.

Figure 3: Physical design of a microfluidic delivery chamber. The design uses LPCVD, PDMS bonding, and wet/dry etching.
Organic Electronic Devices and Circuits

CNF Project # 775-99
Principal Investigator: George G. Malliaras

Abstract:

Transistors based on organic small molecules and polymers have been studied extensively for more than a decade. In order to realize low-cost circuits for applications such as RFID tags and flexible display backplanes, individual device performance must be optimized by studying material properties and interfaces in the devices, as well as processing techniques used in fabrication. Once these performance parameters are maximized, test circuits must be made to see how they translate into circuit speed and robustness.

Summary:

Organic thin-film transistors can be fabricated using a variety of techniques including inkjet printing, stamping and thermal deposition through a shadow mask. Photolithographic patterning has not received much attention in the organic electronics community partly because the chemicals involved are not compatible with the organic films. Pentacene, the highest performing organic small-molecule, is particularly sensitive to solvents as well as the roughness and chemistry of the surface onto which it is deposited. Photolithography has many advantages over alternative techniques, including high resolution, good registration for multiple level patterning and parallel throughput, which can be scaled to high production capacity, not to mention the 40+ years of industry experience in the technique.

We have demonstrated that the gap can be bridged in many cases using parylene liftoff, a technique invented at Cornell University for patterning cells [1] and adapted for use with organic electronic materials by DeFranco et al [2]. The technique involves depositing and etching a chemically robust polymer (parylene-C) onto the substrate, patterning it with standard photolithography and oxygen plasma etching, and then transferring that pattern to the deposited organic semiconducting material by mechanically peeling the parylene film. Patterns as small as 1 µm have been made with this process. Subtractive patterning can also be accomplished using parylene as an etch mask with similar success.

Using the liftoff technique, transistor electrodes can be made using the conductive polymer PEDOT:PSS with a channel width of 2 µm. Pentacene was used as the semiconducting material in these devices, though it remained unpatterned. A two step process which patterns both the PEDOT and pentacene layers can be found in the second reference.

The next step beyond making individual devices is making simple circuits to test the materials under AC conditions. Though the electrodes in this case are made of metals instead of conducting polymers, the active semiconducting layer (pentacene) is patterned using the same technique used to make the PEDOT:PSS layer in the individual circuits. The pentacene patterning step serves to isolate different circuit elements from one another. Leakage current between the transistors in the ring oscillator would destroy the operation of the circuit by creating shorts.

Optimizing the smoothness of the gate dielectric is vital to the performance of a pentacene transistor, which makes large grains only on smooth surfaces. Larger grains translate to higher mobility because the carriers have fewer grain boundaries to cross. ATEOS (Tetraethyl Orthosilicate) process on the GSI PECVD tool was used to deposit SiO₂ onto the platinum gate electrodes. This produced films with RMS roughness ~ 2 nm, similar to that of the metal electrode.

In conclusion, both individual organic transistors and simple circuits have been fabricated using modified photolithographic techniques, using parylene as a pattern transfer layer.

References:


Organic Electronic Devices and Circuits

CNF Project # 775-99
Principal Investigator: George G. Malliaras
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Figure 1. PEDOT:PSS electrodes patterned using parylene liftoff technique. Pentacene deposited to form transistor.

Figure 2. Performance of pentacene transistor with PEDOT:PSS electrodes.

Figure 3. A 5-stage ring oscillator. Pt gates and gold electrodes patterned using photoresist liftoff technique on GCA AutoStep. TEOS oxide deposited on GSI PECVD.
Direct 120 Volt, 60 Hertz Operation of an Organic Light Emitting Device

CNF Project # 775-99
Principal Investigator: George G. Malliaras

Abstract:
Organic electroluminescent devices are being developed for solid-state lighting applications due to their ease of fabrication, compatibility with glass and plastic substrates, color purity, and potential as highly efficient emitters. This work [1] involves the fabrication and operation of “plug and play” organic light emitting devices that can be sourced directly from a standard U.S. outlet; namely, at 110-120 V rms and 60 Hz. We demonstrate a technique for developing large-area solid-state lighting panels that are cost-effective and fault tolerant, further support for the implementation of OLEDs for general lighting purposes.

Summary:
Typically, sandwich-structure OLEDs are made by depositing an organic semiconductor between two metal electrodes. One is a transparent metal which injects positively charged holes, often indium-tin oxide (ITO), and the other is a highly reflective electrode which injects electrons. The latter is usually made from a metal which is prone to donating electrons, but this fact causes them to oxidize in air and subsequently degrade. Within the class of organic electroluminescent materials, ionic transition metal complexes (iTMC) enable the use of air-stable metals due to the presence of mobile ions in the solid-state film. The drawback of iTMCs is that the ionic mobility is typically low, so the turn-on time suffers. Furthermore, typical sandwich structure devices operate near 3V and degrade rapidly at higher voltages. Thus, it was necessary to overcome two distinct challenges in order to fabricate a “plug and play” device: 1) engineering the material to respond at a 60 Hz frequency; and 2) developing a device architecture to withstand the 110-120 V rms amplitude signal supplied by a standard U.S. outlet.

In order to achieve operation at 60 Hz, the turn-on time of the device needed to be improved dramatically. Recently, an ionic liquid was shown to improve the turn on time of Ir electroluminescent devices by increasing the ionic mobility of the organic material [2]. In the current work, we demonstrated 60 Hz operation of a single ITO/iTMC complex : ionic liquid/Au device, thus achieving the necessary response time.

As standard U.S. outlet voltage is 110-120 V rms, it was necessary to implement a device architecture that would withstand high voltages. Recently, Bernards et al. demonstrated dc operation of a cascaded device architecture [3]. In this configuration, devices were connected in series so that the total voltage required was the sum of the individual device voltages. A cascaded design that allowed us to address between 4 and 96 devices in series was fabricated for outlet operation.

The ITO layer was patterned using photolithography along with wet etching to remove unwanted material. The iTMC complex : ionic liquid layer was spun onto the patterned ITO layer. The top contacts were deposited through a Si wafer shadow mask defining 96 devices per substrate, each with a 1 mm$^2$ active area. This mask was patterned by photolithography and etched with a Unaxis 770 plasma etcher employing a Bosch etch technique.

Stable 120 V, 60 Hz operation was observed for 16, 24 and 36 connected devices. In general, the panel brightness increased and half-life decreased with decreasing number of connected devices. For 16-device operation, the maximum rms external quantum efficiency was 5% at a luminance of 200 cd/m$^2$.

References:
Direct 120 Volt, 60 Hertz Operation of an Organic Light Emitting Device

CNF Project # 775-99
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Figure 1, top left: Layout for a 96-device cascaded panel showing the indium-tin oxide (ITO) and Au electrodes. A device is completed by spin coating the organic layer on top of the ITO electrodes and depositing the Au electrodes on top.

Figure 2, above: Si wafer shadow mask for deposition of the Au electrodes, patterned by photolithography and etched with a Unaxis 770 plasma etcher employing a Bosch etch technique.

Figure 3, bottom left: Operation of 16 devices of a 96-device cascaded panel.
Accumulated Body MOSFET: A Side-Gated Approach for Extreme Electrostatic Threshold Voltage Tuning

CNF Project # 804-99
Principal Investigator: Prof. Sandip Tiwari

Abstract:
CMOS device dimension reduction at the nanometer scale is limited by problems arising from variability in threshold voltage ($V_t$) control, variance due to random dopant effects, and poor short channel effects. Scaling of the device width below 20 nm allows electrostatic control of the active area of a MOSFET from the two sides. A device where the substrate does not float, $V_t$ is tunable, and the other effects are minimized, is very appealing. We show such a device using holes for halos to suppress short-channel effects. This novel approach also allows threshold voltage tuning in our structure.

Description:

In the accumulated body MOSFET, additional gate electrodes on the two sides are integrated as a part of the shallow trench isolation to an otherwise planar nFET. These side-gates are isolated from the active area by 18 nm of $Si_3N_4$ and are connected together surrounding the active area [1]. Ultra-narrow channel devices are fabricated along with wider devices. Larger current drives than bulk Si are achieved at short dimensions using optical lithography.

The effective width of the device in the figures is estimated to be approximately 7 nm. With the application of a large negative side-gate bias, the side interfaces are accumulated with holes. This is manifested as accumulation of the whole body up to $10^{19}$ cm$^{-3}$ in volume concentration for the given $W_{eff}$. This results in significant change in the depletion depth under the gate of the FET.

The change in $V_t$ follows a square root dependence (proportional to $\sqrt{-I_{id}}$) suggesting that the effect of the accumulated charges under the gate is similar to body doping.

The $V_t$ sensitivity achieved by accumulated body operation is more than 1.6 V/V in a range exceeding 3 V. These values far exceed $V_t$ tuning achieved in double/back-gate transistors where the two parallel gates straddle the Si body [2].

Summary:
The use of accumulated body approach allows electrostatic $V_t$ control, mitigates the limitations due to random dopant effects by eliminating channel doping, and provides improved short channel behavior using mobile charge halos. The structure may be amenable to mixed-signal applications requiring signal mixing.

References:
Accumulated Body MOSFET: A Side-Gated Approach for Extreme Electrostatic Threshold Voltage Tuning

CNF Project # 804-99
Principal Investigator: Prof. Sandip Tiwari
User: Ali Gokirmak
Affiliation: Electrical & Computer Engineering, Cornell University
Primary Funding: NSF
Contact: agg6@cornell.edu, st222@cornell.edu

- Side-gated ultra-narrow channel devices using Si$_3$N$_4$ STI.
- Negative side-gate bias accumulates ultra-narrow body with holes with concentration up to $10^{19}$ cm$^{-3}$.
- No visible short channel effects down to $L_{\text{eff}} = 150$ nm (no halo implants).
- Threshold voltage tuning in a range exceeding 3 V.
- Threshold voltage sensitivity to $V_{\text{side}}$ exceeding -1.6 V/V.
- Allows further device scaling without suffering from stochastic effects.

Figure 1, above: SEM image of a side-gated FET and cross-section schematics (inset). $t_{ox} = 4$ nm.

Figure 2, top right: In an ultra-narrow channel device depletion depth is reduced to $W_d$ due to accumulation of the body with holes when a large negative $V_{\text{side}}$ is applied. $W_{d0}$ is the depletion depth for a wide device.

Figure 3, bottom left: Transfer characteristics for $V_{\text{side}} = 0$ to -2 V, $V_{\text{sub}} = 0$ V, $V_d = 1$ V

Figure 4, bottom right: Threshold voltage response to $V_{\text{side}}$ and a square root trend line.
3-Dimensional Electronic Integration

CNF Project # 804-99
Principal Investigator: Prof. Sandip Tiwari

Abstract:
Hierarchical organization of massively integrated electronics benefits significantly from the shorter vertical interconnection distances that are achievable via three-dimensional integration. We have developed and analyzed an approach where direct oxide to oxide thermal bonding or polymer assisted bonding makes the integration possible. The largest impediment in such approaches is the increasing difficulty of managing power due to poorer thermal conductivity of insulators and due to increasing number of power dissipating planes of devices. Thermal characteristics of the approach and the desirable attributes in mixed-signal and digital environment have been explored.

Description:
Our low temperature 3D integration technique employs temperatures below 350°C and uses a process in which simultaneous bonding of the transferred structure onto a host substrate occurs together with simultaneous delamination from the holding glass wafer. Figure 1 shows the approach. Up to 4 layers of such devices have been fabricated, and the temperature consequences of the poorer thermal conductivity analyzed through the junction transfer characteristics which has a temperature dependence because of its energy activated basis. Figure 2 shows an example of multiple layers of transistors, and Figure 3 shows a top-view of the structure with underlying devices and temperature dependence. The technology of this effort is summarized in [1-3].

Summary:
A low temperature three-dimensional integration approach has been developed, and up to four transistor planes integrated. We have evaluated the thermal constraints of such designs by experimental temperature dependent measurements. The short interconnectivity (and hence low latency, lower related communication power, and shorter signal times) and poorer thermal properties are the two dominant attributes that provide a number of compelling designs in functional integration of digital and mixed-signal designs. Together with the back-gated transistor approach’s adaptive power control, these techniques address needs of massive integration.

References:
3-Dimensional Electronic Integration

CNF Project # 804-99
Principal Investigator: Prof. Sandip Tiwari
User: Sang Kevin Kim

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Figure 1, above: Essentials of the fabrication approach.

Figure 2, top right: Four-transistor planes assembled using the low temperature fabrication.

Figure 3, bottom left: Top view and temperature dependence of junction when no effort is made to mitigate heat issues resulting in large increase in temperatures in the top-most device plane.
Development of a Nano-Scale Charge Trapping Memory Array

CNF Project # 804-99
Principal Investigator: Prof. Sandip Tiwari

Abstract:
We have been exploring the use of single/few electrons in distributed storage for use in non-volatile, low power, and fast memories. Providing statistical reproducibility at the nanoscale is a key challenge since we are working with a limited number of storage sites. We have used defects at interfaces of dielectrics (oxide/oxide and oxide/nitride) to evaluate this reproducibility and evaluate the performance of memories. Memory arrays with short channel widths and lengths (30 nm and above) have been fabricated on SOI substrates using electron-beam lithography and the devices and arrays show fast program and erase time. However drain-induced barrier lowering (DIBL) characteristics are degraded compared to large scale devices due to short channel effects (SCE).

Fabrication:
Using a common alignment and mixed-lithography (stepper and e-beam lithography system), 30 nm scale NAND and NOR memory structures and arrays were fabricated. The shortest dimensions—channel width and length—are defined in resist by electron beam lithography combined with pattern transfer through dry plasma processes. Following the active region definition, an SiO$_2$ spacer is deposited to prevent discontinuity in the very thin and narrow poly-silicon gate which is patterned by e-beam lithography. The electron storage node films, ONO (2.8nm/7.5nm/5nm), are grown and deposited. The nitride and blocking oxide are deposited by LPCVD after bottom tunnel oxide is grown by thermal oxidation. The poly-silicon is deposited immediately following the insulator stack, and this is followed by deposition of oxide that is employed as a hard mask for gate definition using e-beam lithography. A sidewall process with lightly-doped drain and deep implants is employed to obtain suitably electrically scaled structures, and aluminum is employed for interconnects.

Summary:
30 nm dimension 64 bit SONOS with long and narrow gates are fabricated by adding a spacer step after the active processes.

In this experiment, the process for the bulk biasing, which helps prevent floating body effects, has been skipped. Sub-threshold swing of fabricated structures is ~ 250 mV/dec, and the DIBL is a 300 mV/V. These can be improved by reducing ONO layers and increasing channel doping in the next fabrication. Devices where no nitride is employed, defects are grown, and a deposited oxide interfaces are utilized, point to the potential of this approach to scaling the gate stack thickness. In the ONO structures, applying 11 V for 1 µsec causes a 2 V threshold voltage shift from a fully erased condition.

To achieve -2 V threshold voltage shift from a programmed state, -18 V pulse is applied to the gate for 100 µsec. Emission process has a higher activation energy than a capture process due to the use of a cold mechanism. Back tunneling of electrons from the gate has also been observed to cause a slow erasure process. Changing fields, such as through the use of high-κ materials instead of ONO layers, can potentially improve the program/erase speed.

References:
Development of a Nano-Scale Charge Trapping Memory Array

CNF Project # 804-99
Principal Investigator: Prof. Sandip Tiwari
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Primary Funding: National Program for Tera level Nano Devices through MOST, Samsung Advanced Institute of Technology
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Figure 1, above: Nanoscale SONOS memory arrays of (a) NOR (b) NAND and (c) unit cell.

Figure 2, below left: Characteristics of (a) Programming time and (b) Erasing time.

Figure 3, below: DIBL characteristics.
Strained Si Channel Super-Self-Aligned Back-Gate/Double-Gate Planar Transistors

CNF Project # 804-99
Principal Investigator: Prof. Sandip Tiwari

Abstract:
A robust framework in electronics that emphasizes power, adaptation, and configurability while achieving speed, noise margin, reproducibility and reliability is highly desired in CMOS technology [1] to tackle issues of complexity and massive scale. A back-gated thin silicon channel transistor is an approach that can lead to the smallest scale while responding to these needs. We have developed necessary techniques and demonstrate self-aligned planar back gate structures. These structures provide the short channel electrostatic control necessary for low power application [2-3] and threshold voltage tuning necessary for power control. The approach also allows introduction of strain for improving the transport of carriers.

Description:
Our novel approach of fabrication [4] starts with an oversized back-gate structure underneath isolated single crystal silicon channels obtained using exfoliation through hydrogen implantation and bonding. Following a series of insulator caps and sidewall spacers formed by deposition and selective dry etching processes, we achieve a super self aligned back-gate transistor structure with thin silicon channel, a back-gate defined by the top gate, and thick source and drain regions. This is a near ideal small transistor geometry and has a separately controlled back-gate with buried interconnect structure. Through oxidation of back-gate during isolation, compressive strain and tensile strain can be introduced at the front channel and back channel of the device respectively. The consequent bending can be seen in an over-oxidized back-gate structure (Figure 1). Devices with variable gate lengths as well as different back-gate oxidation have been characterized. While double gate operation can provide higher current drive (Figure 2), the separation of back-gate provides adaptive power control through threshold voltage adjustment (Figure 3). Our unique approach also allows the formation of buried interconnects, and the structure is amenable to novel circuits and configurable switches.

Summary:
A super-self-aligned back-gate/double-gate transistor has been demonstrated. The structure has thin silicon channels, sidewalls to reduce parasitic capacitances, and thick source and drains to improve parasitic resistance. Strain can also be introduced into the structure. Device measurements show adaptive power control.

References:
Strained Si Channel Super-Self-Aligned Back-Gate/Double-Gate Planar Transistors

CNF Project # 804-99
Principal Investigator: Prof. Sandip Tiwari
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Primary Funding: CNS/NSF
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Figure 1: SEM image of the strained device structure.

Figure 2: Transfer characteristics of a 300 nm pFET in front-gate mode (FG), back-gate mode (BG) and double-gate (DG) mode.

Figure 3: Modulation of the front-channel sub-threshold characteristics at different back-gate bias.
A Compact Single-Walled Carbon Nanotube Transistor Integrated with Silicon MOSFET Using a Single Common Gate

CNF Project # 804-99
Principal Investigator: Prof. Sandip Tiwari

Abstract:
Integration of carbon nanotubes with silicon devices can potentially leverage compactness together with the nanotube’s superior transport properties. However, integration of carbon nanotubes, control of placement, good contacts, and suppression of instabilities and drift are a challenge. In this work, we have implemented a compact integrated structure with a silicon MOSFET on the bottom and a carbon nanotube MOSFET on the top, sharing a common gate (Figure 1). Such a structure, an inverter, is highly compact, unique in its sharing of a common buried gate, and demonstrates the potential for integration in nanotube-based sensing.

Description:
Difficulties in making local back gate device comes from the need for planarization of the back gate to the surrounding insulating material-usually silicon dioxide. In our novel approach of fabricating a single-walled carbon nanotube local back-gate, devices with good planarization and thin gate dielectric film are achieved. MOS devices were first fabricated on the bulk silicon wafer following silicon CMOS technology. A low-κ dielectric material, a spin-on-glass, was then deposited, and followed by a chemical mechanical polishing (CMP) process to obtain a planarized buried gate structure for the integration of carbon nanotube devices on top. N+ polysilicon was employed as gate for both silicon transistor and carbon nanotube transistor. A grown oxide with a thickness of 12 nm from polysilicon was employed as a gate dielectric for the CNFET. The growth of the nanotube takes place after the fabrication of the silicon transistor.

Summary:
We have demonstrated a working concept of the hybrid integration of a silicon and nanotube transistor by making an inverter with a p-type CNTFET placed directly on top of an n-type MOSFET. The two transistors share a common polysilicon gate. A cross section schematic of the integrated structure is shown in Figure 1. Its top view can be seen from the SEM image in Figure 2. Separate device transfer characteristics are shown in Figure 3. Thin oxide results in low instability in the CNFET. The performance of these structures is limited by the formation of good metal contacts to the carbon nanotube. The approach potentially provides high density and high local gain for ultra-sensitive detection using CMOS circuits underneath.
A Compact Single-Walled Carbon Nanotube Transistor Integrated with Silicon MOSFET Using a Single Common Gate

CNF Project # 804-99
Principal Investigator: Prof. Sandip Tiwari²
User: Hao Lin¹

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2. School of Electrical & Computer Engineering, Cornell University
Primary Funding: Center for Nanoscale Systems, Cornell University
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Figure 1, top left: Cross section schematic of the inverter structure.

Figure 2, bottom left: Top view SEM image of the inverter (two dashed lines indicate underneath nMOSFET active area).

Figure 3, above: Independent transfer characteristics of the CNFET and the nMOSFET.
In-Plane Alignment of Carbon Nanotubes using Oblique-Oxide-Deposition

CNF Project # 804-99
Principal Investigator: Prof. Sandip Tiwari

Abstract:
Preferentially-aligned growth of single-walled carbon nanotubes using oblique oxide deposition is reported. By tilting the surface normal relative to the incident oxide vapor stream, a structured surface results that is conducive to preferentially-aligned chemical vapor deposition growth of carbon nanotubes. Preliminary experimental observations are reported.

Summary:
Carbon nanotubes (CNTs) have many unique physical properties [1] that spur a lot of current interest. However, before they are of practical use, controlled growth and patterning must be achieved, whereby the size (diameter and length), location (anchor point), alignment (growth direction) and chirality are reproducible for the specific application.

Whereas the diameter [2] and anchor point [3] of CNTs have seen progress in process control, the direction of growth/alignment remains entirely unsatisfactory. Directionality/alignment is a key necessity of any application requiring in-plane nanotube structures.

In this work, we report on a novel method to achieve preferentially-aligned growth of in-plane, single-walled carbon nanotubes using oblique oxide deposition (OOD). With OOD, we are able to structure the surface topography on a nm-scale, prior to conventional chemical vapor deposition (CVD) growth, thereby causing the CNTs to grow in a preferred direction relative to the topography. This method is a powerful one in that multiple and arbitrary directions for CNT growth can be achieved in a single CVD growth run, thereby facilitating the processing of large, complex, networks of organized CNTs.

Obliquely-evaporated films of silicon dioxide have long been known to cause “sympathetic” alignment of liquid crystal molecules [4]. Therefore, might it be possible to utilize OOD to preferentially align CNTs during CVD growth? This was the rational by the first user for conceiving this work.

Following resist-processing, a ~ 0.3 nm-thick iron (Fe) catalyst layer was evaporated (at normal incidence) across the wafer and subsequently lifted off during a 30 min soak in AZ1165.

Following lift-off, the wafers are scribed, cleaved into smaller chips, and readied for CVD growth. During a typical growth run, a quartz tube furnace is raised from room temperature to 700°C at 70°/min while flowing 800 cm³/min of argon. After reaching 700°C, argon continues to flow for a period of 10 min to purge the furnace tube of any remaining oxygen. Following that, for an additional period of 10 min, 800 cm³/min of argon and 150 cm³/min of hydrogen are passed through the furnace tube at 700°C for pre-conditioning. Carbon nanotube growth then ensues for a period of 6 min, during which time 5.5 cm³/min of ethylene (C₂H₄) is added to the Ar and H₂ pre-growth gases. Cool-down at 12°/min with 800 cm³/min of argon completes the process.

Work remains to be done that differentiates between gas-flow alignment and nano-structured alignment using OOD.

We thank Prof. Paul McEuen and his group for the use of his CNT growth furnace and numerous interactions.

References:
In-Plane Alignment of Carbon Nanotubes using Oblique-Oxide-Deposition

CNF Project # 804-99
Principal Investigator: Prof. Sandip Tiwari
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- Oblique-oxide deposition causes nano-structuring of the surface.
- Carbon nanotubes respond to this surface nano-structure during CVD growth.
- The alignment of carbon nanotubes can be controlled via feedgas flow direction, electric fields, chemical-functionalization and surface nano-structure.
- Work remains to differentiate between these various alignment mechanisms.

Figure 1: AFM image of a 100 nm thick obliquely-deposited oxide film. (The size of the image is 1 x 1 µm²).

Figure 2: SEM of carbon nanotubes grown on the obliquely-deposited oxide layer.
Ballistic Deflection Transistor Development
CNF Project # 1301-04
Principal Investigator: Martin Margala

Abstract:
The ballistic deflection transistor utilizes a novel non-linearity first discovered by Song et. al [1,2]. This non-linearity is based upon a shift in momentum created by an artificial scattering mechanism rather than a PN junction. We are currently developing a room temperature transistor based upon this non-linearity in combination with electric gates that direct electrons to the appropriate portion of the artificial scatter. The materials used are InGaAs-InAlAs forming a heterostructure that creates a two dimensional electron gas (2DEG). The general structure of the device is a cross shaped structure with a triangular section removed from the intersection of the cross, with two lateral gates on the longer portion of the cross. The removed triangular section acts as the artificial scattering mechanism, and in combination with the gates enables direction of electrons to either lateral channel. A bias current is applied across the long section of the device much in the same way as an electron gun in a CRT, with the gates acting as the steering field.

Summary:
This structure has several advantages over conventional transistors. The lateral gates only direct current, and are not used to stop the current through the device (though they can deprive a channel of current). This improves the transit time through the device. The lateral gates also integrate the gate noise reducing the noise of the device. The general structure of the device is inherently a differential pair rather than a single on/off switch, increasing compactness. Also there is no intrinsic threshold as such; current is always flowing but is controlled between two ports. The switching voltage is a ratio between the bias voltage and gate size, and therefore, a very low voltage operation is possible (below 100 mV) with noise being the limiting factor.

The fabrication of the device begins with the growth of the heterostructure using molecular beam epitaxy. The InGaAs-InAlAs heterostructure is grown on an InP substrate with the optimization being mean free path at room temperature. Gold alignment marks suitable for electron beam lithography are created to enable multiple alignment steps. Hard mask formation is achieved by the evaporation of carbon followed by patterning using XR-1541 resist. Unexposed areas are subjected to an oxygen plasma that removes the carbon, creating the transfer mask. The etch is achieved using an ion mill. Lateral gates can be formed one of two ways, as a semiconductor or as a metal. For semiconductor gates, they will be patterned at the same time as the rest of the structure. For either type of gates, a layer of XR-1541 is exposed and used as an oxide layer. A contact layer is then formed using Ni-Ge-Au contacts and annealed at 350°C for 30 seconds. If metal gates are required, an additional lift-off process is performed to create the metal gates. Currently, gate formation has been the most challenging portion of this project with only limited success observed.

The recent measured results have shown successful fabrication of the microchannels required for non-linear behavior. This diode type non-linearity is the enhancement by the deflective structure. While triode behavior has been observed at this level (by altering the bias voltage and measuring the output port voltages), we have not achieved gain in this fashion. The gates will enhance this non-linearity to provide gain for this device. The non-linear behavior of the channels is currently being modeled based upon a kinetic inductance and quantum resistance. Initial results indicate an inherent high frequency oscillator transport property that is proportional to electric field strength for narrow 2DEG channels as a possible source for the non-linearity.

References:
**Ballistic Deflection Transistor Development**

CNF Project # 1301-04  
Principal Investigator: Martin Margala  
User: Quentin Diduck  

Affiliation: Electrical and Computer Engineering Department, University of Rochester  
Primary Funding: Office of Naval Research Grant N000140510052  
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Figure 1: Fabricated ballistic deflection transistor. The top port is referenced as $V_b$, with the left and right ports considered $V_c$ for the measurements shown. The gates are currently non-functional.

Figure 2: IV curves taken from ground to $V_c$, with $V_b$ voltages from top to bottom of -1, -0.5, 0, 0.5, and 1 Volts respectively. Solid lines are measured results, dashed are the mathematical model.

Figure 3: Equivalent circuit used in our mathematical model which assumes a natural oscillation frequency that is a function of electric field.
Fabrication of SOI-Based Nanowires

CNF Project # I353-05
Principal Investigator: Mark Reed

Abstract:

The goal of this project is to realize Si nanowires from the active Si layer of silicon-on-insulator (SOI) wafers. The potential use of nanowires for chemical and biological sensing applications is a subject of intense recent interest and study. Nanowires synthesized by chemical vapor deposition and other methods have shown much promise as sensors but must be transplanted from their growth substrate onto planar wafers for device fabrication, which is an inherently random process (no alignment is possible). Nanowires fabricated from SOI have the advantage of being fabricated with traditional top-down processing techniques, thus alignment is straightforward. Furthermore, such devices have the added advantage that they can be fabricated into Hall bar structures to determine Hall mobility for the first time in a Si nanowire.

Summary:

A critical shortcoming of nanowire-based sensors is the bottom-up assembly required for device fabrication: the nanowires must be deposited on a planar substrate (typically a silicon wafer with a thin oxide that serves as the gate dielectric in a backgate geometry) because they are generally synthesized on a growth substrate incompatible with processing. Additionally, there is a great variation in the device characteristics of nanowires. In order for any statistically relevant study to be performed, a multitude of similar devices must be fabricated, thus a bottom-up approach is impractical.

Using the tools available at the Cornell NanoScale Facility (reactive ion etching, e-beam, oxidation furnaces), we have developed a fabrication process to produce silicon nanowires from an ultra-thin SOI wafer with traditional top-down lithography.

We have successfully realized Si nanowires with widths of ~ 50 nm—the height of the active layer is 80 nm—using chlorine reactive ion etching (RIE). Large (3 µm wide) control structures produced by a fluorine RIE process have been fabricated and their electrical characteristics have been measured. We are currently investigating the electrical characteristics of Hall bar nanowire samples and the use of these nanowires as chemical and biological sensors.

References:


Fabrication of SOI-Based Nanowires

CNF Project #: 1353-05
Principal Investigator: Mark Reed

Users: Eric Stern, David Routenberg

Affiliations: 1. Depts. of Electrical Engineering and Applied Physics; 2. Dept. of Biomedical Engineering;
3. Dept. of Electrical Engineering; Yale University

Primary Funding: DARPA through AFOSR (FA9550-05-1-0395) and ONR (N66001-04-1-8902) and NSF and Department of Homeland Security graduate fellowships

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Figure 1: Device fabrication. A. Optical micrograph showing the die layout: four devices in the center fanning out to pads. The darker pads are contacts through the BOX to the handle Si; B. Optical image of a 3-µm-wide control structure defined by a fluorine reactive ion etch. The device is light on the dark BOX background; C. SEM of a control (3 µm) Hall-bar structure. The light lines within the leads are regions of degenerate doping; D. Optical micrograph of a thin ~ 50 nm nanowire. This structure is defined with e-beam lithography and realized with a chlorine reactive ion etch after the initial optical lithographic steps are complete. The original fluorine RIE etches the BOX whereas the chlorine RIE does not, thus the BOX is multi-colored.

Figure 2: Control structure transport properties. A. I_{SD} vs. V_{SD} with varying V_{GD} plot for the device shown in Fig. 1B. The Si handle wafer serves as the backgate; B. Plot of I_{SD} vs. V_{GD} for V_{SD} = -1V. There is a clear hysteretic effect in the sweeps of this control structure.
Changes in the Morphology of Si <111> Surfaces Under High-Temperature, Ultra-High-Vacuum Annealing Applications

CNF Project # 317-87
Principal Investigator: Jack M. Blakely

Abstract:
This research project involves analyzing the processes that take place on the Si <111> surface under high-temperature, ultra-high-vacuum (UHV) annealing. Atomically flat surfaces can be obtained by high-temperature annealing in UHV of specially patterned silicon samples according to a procedure developed previously [1]. Normally, many hours of high temperature anneal and slow cooling is required in order to obtain large atomically flat areas. For this project, we investigated the effect of short time annealing (~ 5 minutes) on the silicon surface evolution. Different annealing times and temperatures result in various atomic step distributions. The surface morphology was analyzed utilizing AFM. As an application, we used the annealed silicon samples as substrates for pentacene deposition. Due to the polycrystalline structure of the pentacene film, there is significant scattering of charge carriers at the grain boundaries. Using atomic steps as preferential sites for nucleation, we are working to find the proper growth conditions for azimuthal alignment of the pentacene crystals so that only small angle boundaries are present. In this way, the average mobility of the charge carriers in the pentacene film should be greatly enhanced.

Summary:
Using standard photolithography techniques, various geometric patterns, with dimensions ranging from 10 to 120 µm, were transferred to the wafers in form of ~ 1 µm deep craters. A temperature gradient was intentionally induced by deliberately clamping the sample tighter on the lower side so that we could investigate the influence of a range of temperatures on the same sample. The difference in temperature between the hottest and the coldest areas on the sample was about 100°C. Several other samples were uniformly heated using various annealing temperatures and short annealing times (1-15 mins).

We measured the root mean square (rms) roughness of the surface in unpatterned regions along a vertical line in the middle of the active part of the sample [2], where the temperature gradient was approximately constant. The value of the roughness was obtained from 5 x 5 µm AFM scans, each scan being 0.4 mm apart. Before annealing, the rms roughness of the surface was 0.88 nm. Towards the lower edge of the sample, where the temperature was high enough, the surface succeeds to reach a thermodynamically stable structure of steps and flat terraces even after such a short annealing time. On the upper side of the sample, where the temperature was lower, the terraces do not develop completely. The measured roughness becomes about four times bigger than the starting surface roughness and then drops. Increased roughness of the surface can be explained by the mechanism known in thin film growth as Ostwald ripening. The atoms from the surface of smaller and sharper irregularities have a higher chemical potential than those of larger clusters. As a result of surface transport, smaller clusters will shrink and disappear while larger clusters coarsen. In this process, the measured roughness reaches a maximum under the conditions where enough larger clusters exist. At higher temperature, even the largest clusters start to shrink and flat terraces become easily visible. At lower temperatures, many small islands exist on the surface instead of fewer bigger ones and, as a result, the measured roughness decreases, but it is still higher than the starting roughness.

Another phenomenon analyzed was the process of ridge formation at the edge of mesas and trench formation at the bottom edge of craters. Previously reported by former group members [3], we observed that these structures form even before the silicon surface reaches the thermodynamic equilibrium. As a consequence, surfaces with low step density can be engineered on top of mesas structures.

Finally, some of the samples were used as substrates for pentacene deposition. Pentacene deposition was performed using the deposition facility in Professor Malliaras’ group by the undergraduate student Jin-Chih Michael Hsu. Different substrate temperatures, in general in the 100-130°C range were investigated. The preliminary results show that atomic steps of height 2-3 times the <111> interplanar separation and at least 2-3 µm apart constitute the best silicon substrate for the pentacene crystals to nucleate primarily along the steps. Substrates other than Si, such as sapphire (0001) are being considered.

References:
Changes in the Morphology of Si <111> Surfaces Under High-Temperature, Ultra-High-Vacuum Annealing Applications

CNF Project # 317-87
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- Atomically flat surface regions are obtained by heating patterned samples in ultra-high vacuum chamber.
- Surface roughness was measured on non-uniform annealed samples.
- A non-monotonic variation in the surface roughness was observed in the first stages of UHV annealing.
- Trench formation at crater borders and ridge development at the edge of mesas was observed even in these first stages of UHV annealing.
- Under proper conditions, pentacene crystals grow along atomic steps.

Figure 1, top left: AFM scan over a large area (60 x 60 µm), showing a crater and the surrounding, stepped region; the surface became atomically flat only in the upper right corner. Some islands formed as a result of surface ripening can still be observed in the middle of the crater and in the left side of the image.

Figure 2, above: Surface roughness changes from the lower (high temperature) to upper side of the sample (lower temperature) going through a maximum.

Figure 3, below left: Monolayer high islands of pentacene crystals aligned along the silicon steps.
Thin Film Nucleation and Growth using Energetic Neutral Species

CNF Project # 459-92
Principal Investigator: James R. Engstrom

Introduction:
The deposition and growth of organic materials for applications in electronics and photonics differs fundamentally from that of more conventional inorganic materials such as metals, semiconductors and oxides. A key difference involves the presence of strong covalent and ionic bonding in the latter class of materials, whereas organic materials are often bound by rather weak dispersion (van der Waals) forces. In this project, we have used supersonic molecular beams as sources for film deposition, in particular, for the deposition of thin films of pentacene, an organic semiconductor.

Summary:
Pentacene is a promising candidate for applications in organic thin-film electronics owing to the ability to form highly ordered thin films near room temperature with excellent electrical transport properties [1,2]. Pentacene, which possesses a very low vapor pressure, presents a number of experimental challenges that must be overcome in order to generate energy tunable beams. First, we designed an in-vacuum evaporator heatable to 200-300°C to provide enough flux for supersonic molecular beams. Next, using time-of-flight quadrupole mass spectrometry, we characterized supersonic molecular beams of pentacene generated using carrier gases of N₂, He and H₂. With these carrier gases, we were able to obtain kinetic energies over the range of E_i = 1.4-10.6 eV whereas incident molecular fluxes were on the order of 10^{15} molecules·cm^{-2}·s^{-1} [3]. We find that beam energy affects a number of phenomena, namely nucleation in the monolayer regime, and both the kinetics of thin film deposition and the microstructure in the multilayer regime. Closer examination of the data indicates that the deposition rate in the monolayer regime is determined by the trapping probability of pentacene [3], which decreases with increasing energy. However, in this same regime the trapping probability is found to decrease with more glancing angles of incidence, a result inconsistent with so-called normal energy scaling. In the multilayer regime, trapping probability also decreases with increasing incident energy, but does so at a rate less than that observed in the submonolayer regime. In addition, grain size of multilayer pentacene films is found to increase with increasing incident energy at normal incidence. These results demonstrate that the morphological evolution of organic thin films can be modified substantially at high incident kinetic energies. The nucleation and growth of pentacene on silicon dioxide surfaces modified with hexamethyldisilazane, HN[Si(CH₃)₃]₂, has also been examined [4]. Similar to growth on clean SiO₂ surfaces, the rate of deposition at a fixed incident flux decreases with increasing kinetic energy of the incident pentacene, indicative of trapping mediated adsorption. Unlike clean, unmodified SiO₂ surfaces, however, growth on the modified surface exhibits the characteristics of heterogeneous nucleation, where the maximum island density is independent of the deposition rate. Deposition in the sub-monolayer regime involves island growth, except that on the modified surface the islands are two molecules high, unlike the one molecule high islands observed on clean SiO₂.

We have also fabricated organic thin film transistors (OTFTs) with these pentacene films with gold top contacts on bare thermal SiO₂ and hexamethyldisilizane (HMDS) coated SiO₂ substrates to understand the relationship between performance characteristics of organic thin film transistors (OTFTs), film microstructure and film-dielectric interface properties. We find that with increasing incident energy of pentacene molecules, the grains get larger and hence the field effect mobility obtained for the corresponding OTFTs increases. In addition, the devices with HMDS primed SiO₂ showed improved performance characteristics with respect to bare thermal SiO₂, owing to reduced trapping at the semiconductor-dielectric interface.

References:
Thin Film Nucleation and Growth using Energetic Neutral Species

CNF Project # 459-92

Principal Investigator: James R. Engstrom

Users: Aravind Killampalli, Jared Mack, Sugandha Bhargava

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Primary Funding: CCMR

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- Pentacene thin films have been deposited using energetic supersonic molecular beams.
- Incident energy of pentacene molecules strongly influences nucleation, film deposition rate and thin film microstructure.
- Organic thin film transistors fabricated from these pentacene films show promising performance characteristics.

Figure 1: AFMs of pentacene on SiO$_2$ surfaces modified with HMDS as a function of exposure to the supersonic beam ($E_i = 6.7$ eV, normal incidence). In each case the field of view is $5 \times 5 \, \mu$m$^2$. Also shown in the bottom right corner is a micrograph of pentacene on clean SiO$_2$ for otherwise identical deposition conditions and an exposure of 75 s.

Figure 2: Occupancy of the first and second monolayers of pentacene as a function of total amount of material deposited on clean SiO$_2$ (lower panel) and SiO$_2$ modified with HMDS (upper panel). Also shown are examples of two histograms that were used to generate the data that is displayed in the figure.
Low Heat Capacity Substrates for Calorimetry Measurements

CNF Project # 522-94
Principal Investigator: Leslie H. Allen

Abstract:
Our group focuses on developing thermal analysis characterization techniques and instruments for thin film material, nanometer scale particles of metals, polymers and nanoliter volumes of proteins [1-9]. The development of the sensing devices for such a characterization technique-NanoDSC-makes use of MEMS fabrication [4,5] techniques using Si-N membranes. A variety of sample configurations can be used including vacuum-deposited, spin-cast, or growth via liquid solution. A variety of materials phenomenon which occurs at the nanometer length scale have been investigated, such as the size-dependence melting point depression and magic number sizes in metal nanoparticles [1,2,8], order-disorder transitions of self-assembled monolayers of alkanethiols (SAMS) [6], and thickness dependence of glass transition in polymers [3,7]. In this summary report, we discuss work on two topics: (1) heat capacity measurements of thin film polymers [7], and (2) the unusual melting characteristics of bismuth nanoparticles [8].

Summary:
One particular size-dependent materials phenomenon which occurs at the nanometer length scale is the thermodynamic properties of ultra-thin polymer films. Shifts in the glass transition temperature $T_g$ (from bulk values) have been reported for nanometer films. Currently, there is a lack of consensus among various experimental techniques concerning the nature of this phenomenon, which is due in part to the limited number of measurement techniques. Our results using the NanoDSC shows promise as a tool for developing a more general understanding to this problem. Analysis of the glass transition obtained by NanoDSC of polystyrene, poly(2-vinyl pyridine) P2VP and poly(methyl methacrylate) PMMA shows (Figure 1) only modest thickness dependence. This particular NanoDSC experiments was done using a annealing/scanning technique described in Macromolecule [7]. The NanoDSC was also used to investigate the melting of Bi nanoparticles. The particles were formed by evaporating Bi onto a silicon nitride substrate. Upon deposition, bismuth self-assembles into truncated spherical particles. As the total amount of bismuth increases, the average size of the particles also increases. By systematically changing the “integrated” amount of Bi deposited (0.3, 0.6, and 2.0 nm) as shown in Figure 2, the size of the bismuth particle changes as can be seen in the TEM micrographs in Figure 2. The same samples as used for the NanoDSC measurements are also used for TEM via the specially designed sensor and fixture [9]. The companion heat capacity measurements of the nanoparticles, shown in Figure 2, indicate the subsequent decrease of the melting point for smaller size particles [8]. The nature of the size-dependence melting point decrease for bismuth is much different than for tin or indium as measured in previous experiments.

References:
Low Heat Capacity Substrates for Calorimetry Measurements

CNF Project # 522-94
Principal Investigator: Leslie H. Allen
Users: Mike Efremov, Eric Olson, Ming Zhang, Zishu (Sue) Zhang, Liang Hu

Affiliation: Material Science Department, University of Illinois at Urbana-Champaign
Primary Funding: NSF Materials Research Dr. L. Hess (0108694), American Chemical Society-Petroleum Research Fund (37027-AC7), NSF Dr. William Nes (0304149)
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Figure 1: (A) Cross-sectional and planar views of a typical NanoDSC sensor with a polymer sample on it (not to scale). (B) Glass transition temperature of (circle) polystyrene, (diamond) high molecular weight polystyrene (square) poly-2-vinyl pyridine), and (triangle) poly-methyl methacrylate assigned using first-pulse curves of the annealing/scanning technique [Macromolecule, 37, 4607 (2004)].

Figure 2: (a-c) TEMs of various amounts of bismuth deposited on the Si-N side of the NanoDSC. Nominal equivalent thickness values are shown. As the total amount of Bi is increases the average size of the particles also increases. The bottom graph shows the corresponding heat capacity $C_p$ data from these films and shows the decrease in melting point for smaller particles—size-dependent melting point depression [J. of Appl. Phys. 97, 34304 (2005)].
Ultrathin Silicon Nitride Membranes for use as a STEM Substrate

CNF Project # 877-00
Principal Investigator: Professor John Silcox

Abstract:
An ultrathin silicon nitride membrane with estimated thickness of 3 nm was produced for use as a sample substrate in a scanning transmission electron microscope (STEM).

Summary:
Silicon nitride membranes are routinely produced using standard procedures, and are available commercially for TEM use with membrane thicknesses reaching 20 nm. However, for the purposes of high-resolution imaging and electron energy loss spectroscopy (EELS) on the tiniest of nanoparticles using STEM, even such thin membranes can swamp the signal from the nanoparticle of interest. The goal of our project was, therefore, to produce silicon nitride membranes with thicknesses of 10 nm or less for use in Cornell’s STEM.

Silicon nitride was chosen as a substrate because, unlike ultrathin carbon substrates, silicon nitride has a bandgap of about 5 eV and can therefore hopefully provide a low-signal area in the low-loss region of the EELS spectra.

Using standard procedures of low-pressure chemical vapor deposition of silicon-rich silicon nitride on <100> silicon wafers, photolithographic techniques, and anisotropic KOH etching, we successfully produced ultrathin silicon nitride membranes. Using the FilMetrics film measuring system located in CNF, the silicon nitride thin film was optically measured to be approximately 5 nm thick on the silicon wafer.

Using STEM imaging, we examined the membranes at high resolution and observed pinholes throughout the film. Despite the extreme thinness of the sample, the silicon nitride membranes show remarkable strength, suspending large particles 4 µm in diameter!

The thickness of the silicon nitride membrane was measured in the STEM using EELS zero-loss and low-loss spectra. Initial estimates using this technique show the membrane to be 3 nm, however at these extreme limits it is not clear that this technique is valid.

Further work is to be done on these membranes by varying the ratio of silicon and nitrogen in an attempt to lower the overall Z-value of the membrane.

Silicon nitride membranes of 5-3 nm were produced for use as a scanning transmission electron microscope (STEM) substrate.
Ultrathin Silicon Nitride Membranes for use as a STEM Substrate

CNF Project # 877-00
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Figure 1, top left: Optical microscope image of silicon nitride membrane. Bright spots are most likely specks of silicon created by the etching process. The buckling of the membrane seen here disappears as the windows decrease in diameter.

Figure 2, bottom left: STEM annular dark field (ADF) image of the membrane. Dark areas are possible pinholes.

Figure 3, above: STEM ADF image of the membrane as a substrate for CdSe quantum dots.
Nanocalorimeter Fabrication for Implantation Defect Investigations

CNF Project # I048-02
Principal Investigator & User: François Schiettekatte

Abstract:

Nanocalorimetry is used to measure the heat release during the annealing of low-energy ion implantation damage in silicon. Low-stress silicon nitride is deposited by LPCVD at CNF in order to fabricate thin-film nanocalorimeters. These consist of a membrane that supports a metal strip, which serves as heater and thermometer during calorimetry experiments. Amorphous and polycrystalline silicon (a-Si and poly-Si) is deposited on the other side of the membrane and implanted with low-energy ions (4-30 keV) at fluences from $10^{11}$ to $10^{15}$ ion/cm$^2$.

In both types of silicon, we observe a striking similarity in shape of the heat release over the wide range of fluences, suggesting that each impacting ion produces a damage zone similar to the damage generated by high fluence implantation, releasing heat in a same way during annealing. Experiments were carried out at heating rates between 33 and 144 kK/s, showing that the heat release is characteristic of a series of processes continuously distributed in terms of activation energy.

We also observe that implants at a lower beam flux translate into smaller heat release for the same fluence. Shorter implantation times at higher fluxes leaves less time for dynamic annealing and higher impact rate may stabilize disordered zones that would have enough time to undergo dynamic annealing at lower fluxes.

Summary:

Nanocalorimetry is a technique developed by the group of Leslie H. Allen at the University of Illinois in collaboration with the CNF, and used successfully to measure the heat involved in a variety of phase change and chemical reactions occurring at the surface of materials [1]. It operates on the same principles as conventional differential scanning calorimetry (DSC) but the thin-film structure of the device provides a small addendum and allows fast heating rates (up to $10^6$ K/s), making possible the observation of thermal processes involving energy exchanges of the order of the nJ/K. Our devices consist of a low-stress Si$_3$N$_4$ membrane (180 nm, deposited at the CNF) that supports a 0.5 mm wide, 7 mm long Pt heating strip. The current flowing through and the voltage drop along the strip are measured and provide power and resistance measurements, the latter being used to measure the temperature of the strip as a function of time. For our investigations, a 140 nm a-Si film is deposited in correspondence with the Pt strip, and can be recrystallized at 900°C for 100 s in order to form poly-Si [2].

In this research project, we use nanocalorimetry to investigate, from the thermal point of view, the kinetics of damage annealing following low-energy ion implantation in a-Si and poly-Si. We found that the heat released has the same profile over the fluence range examined ($10^{11}$ to $10^{15}$ ion/cm$^2$), featuring no peaks but rather a smooth, continuous shape. This result suggests that each impacting ion produces a high-damage zone similar to the damage generated by high-fluence implantation. Such heat release is thus interpreted as the result of highly damaged zones that undergo annealing by small recrystallisation and relaxation steps, each step featuring a wide range of activation energies [3]. As it depends of the details of the damage structure of each ion, the annealing kinetics in silicon is thus independent of ion fluence and implantation energy (4-30 keV).

Experiments were carried out at heating rates between 33 and 144 kK/s. The shape of the heat release and its behaviour with increasing heating rate is characteristic of a series of processes continuously distributed in terms of activation energy [4]. Ion beam flux was also investigated and it was shown that damage annealing releases more heat for implantations carried out at higher flux, suggesting that damage undergoes more dynamics annealing at comparatively lower flux, simply because the implantation time is longer. Another interpretation is that more frequent impacts stabilize structure by producing displacements around a previously damaged region before it has time to dynamically anneal.

References:


Nanocalorimeter Fabrication for Implantation Defect Investigations

CNF Project # 1048-02
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Figure 1, above: Thin film calorimeters fabricated using LPCVD low-stress nitride deposited at CNF.

Figure 2, top right: a) Heat release after 30 keV Si implantation in poly-Si for a wide range of fluences.
   b) The normalized signal shows that the heat release is the same at all fluence, indicating that the underlying processes are similar.

Figure 3, bottom right: Heat released by poly-Si implanted with 30 keV Si at 0.1 Si/nm² for different heating rates. The heat release is characteristic of a wide distribution of activation energies.
Improving Mechanical Reliability of Si Nanobeams with Self Assembled Monolayers

CNF Project # 1154-03
Principal Investigator: Alan T. Zehnder

Abstract:
The fracture durability of silicon nano-beams in air was improved considerably by methyl surface termination. 210-nm thick, doubly clamped beams with functionalized surfaces were tested after exposure to air over a period of 23 days. Experiments showed that the native oxide growth reduced the strength of the beams with H terminated surfaces from 16.2 GPa to 11.2 GPa. Coating the sample surfaces with methyl monolayers prevented oxidation and maintained the strength throughout the test period under same environmental conditions.

Summary:
Due to their small sizes and use of materials with very low defect densities, materials used in the nanoscale have the potential to sustain very high stresses. For microelectromechanical system (MEMS) applications, as important as the high initial strength, is the prevention of aging of the structure throughout its service life. Si devices operating in air are expected to have very high durability under mechanical loading as Si is not susceptible to stress corrosion cracking. However, the native oxide layer (amorphous SiO$_2$) may enhance fracture initiation and cause catastrophic failure [1]. How does air exposure influence the fracture reliability of single crystal Si under static loading? And how can the service life of Si structures in air be improved? Our study shows that effective protection of the surfaces is essential for the maintenance of the mechanical strength of nanostructures.

To characterize the effects of air exposure on the fracture reliability of nanostructures, 210-nm thick, 500-nm-wide, 12-µm-long doubly clamped suspended beams with H and CH$_3$ terminated surfaces were fabricated from Si (111) wafers using previously described procedures [2,3]. After their fabrication, samples with different surface terminations were exposed to office air and tested at the 3rd, 13th and 23rd days with an AFM [4].

The Weibull strength, $\sigma_0$, of the H-terminated beams decreased steadily from 16.2 GPa to 11.2 GPa with increasing air exposure time, whereas the strength of the CH$_3$ terminated samples remained at 18 GPa over the test period of 23 days.

It is known that H-terminated surfaces show only a temporary protection against oxidation. An approximate 7-day air exposure results in an oxide thickness of 1.5 nm for H-terminated Si (111) [5]. The native SiO$_2$ has a lower fracture resistance and is prone to undergo moisture assisted cracking under applied stress. With increasing oxide thickness, the load that is necessary to initiate the crack, and hence the observed fracture strength, decreases. It should also be noted that the native oxide, unlike thermally grown oxide, does not necessarily have uniform properties. Its thickness and quality change randomly on the surface. We hypothesize that the inhomogeneous oxidation of the surface results in randomly distributed, local residual stresses, which, together with the increase in surface roughness may enhance fracture initiation, hence reducing the strength of the tested beams.

Our results show that methyl surface coating effectively protected the surfaces for up to 23 days by preventing oxidation and allowed fracture strengths to be maintained.

References:
Improving Mechanical Reliability of Si Nanobeams with Self Assembled Monolayers

CNF Project # 1154-03
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Users: Tuncay Alan, Debodhonyaa Sengupta

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- Surface protection plays a crucial role in maintaining the mechanical reliability of Si nanobeams.

Figure 1, top left: Weibull fracture probability plot of beams with H terminated surfaces. Inset: a) A test beam before and b) after the experiment.

Figure 2, bottom left: Weibull fracture probability plot of beams with CH$_3$ terminated surfaces.

Figure 3, above: Change in Weibull fracture strength of beams with H and CH$_3$ terminated surfaces with increasing air exposure.
Nanoporous Gold Thin Films

CNF Project # 1163-03
Principal Investigator: Christopher Umbach

Abstract:
Nanoporous gold (NPG) can be fabricated as a thin film by the sputtering of Ag-Au onto suitable substrates followed by a wet chemical dealloying treatment. During dealloying, removal of the silver and redistribution of gold results in a nanoporous network with openings on the order of 10 nm. Metallic buffer layers allow the fabrication of crack-free films that remain adhered after dealloying. The thin-film NPG shows the same morphology after dealloying as that of a NPG produced from a 200 µm thick membrane sheet of the alloy. The pore structure of both the thin film and the bulk membrane can be coarsened through annealing.

Experimental Details:
Nanoporous gold has potential for filtration of nano-objects and use in high surface area detectors and ultra-capacitors. Thin-film implementation of NPG allows the use of this material in devices at lower cost. We have fabricated thin film NPG on conventional substrates.

Both glass and oxidized silicon were used as substrates. Glass substrates were first cleaned with distilled water and then methanol in an ultrasonic bath for several minutes before being plasma cleaned for 10 minutes. In CNF, Si wafers were MOS cleaned and then wet oxidized to a thickness of 100 nm. Chromium was evaporated as an adhesion layer, on top of which gold was evaporated as a buffer layer. The uniform metallic buffer layers were deposited in NBTC using a CHA Mark50 evaporator. Ag-Au (70%-30%) alloy was used as a target in a Baltec sputter coater to deposit films with thicknesses ranging from 100 nm to 500 nm. Diluted nitric acid was used to dissolve the silver from the Ag-Au alloy. Samples were annealed in either an oxygen atmosphere or in a vacuum. The annealing took place at 200°C and lasted four hours. Samples were imaged with both atomic force microscopy in NBTC and with high resolution Zeiss Ultra SEM in CNF.

Results:
The nanoporous thin films exhibit a morphology similar to that of the bulk membrane both in pore size (generally between 20 and 30 nm) and degree of interconnectedness. The films deadhered upon dealloying when the Au/Ag alloy was deposited on only a chromium adhesion layer. Best adhesion was observed with the gold buffer layer. The samples dealloyed for 12 hours appear to be less pore-dense than when dealloyed for several minutes. The pore openings can be increased to several hundreds nanometer by annealing.

Summary:
NPG films have been fabricated on glass and Si substrates. The pore size and pore density of the films is similar to that observed for NPG fabricated from bulk alloy material.

References
Nanoporous Gold Thin Films

CNF Project # 1163-03
Principal Investigator: Christopher Umbach
Users: Totka Ouzounova\textsuperscript{1}, Andrew Thiel\textsuperscript{2}

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- Films deposited by sputtering: 70/30 at % Ag/Au alloy target.
- Dealloying in diluted nitric acid produces nanoporous gold.
- Initial pore size ~ 30 nm.
- Similar to dealloyed bulk membrane sheet.
- Pore size increases with annealing.

Figure 1: (a) 200 µm bulk membrane sheet dealloyed 12 hours. (b) 200 nm thin film dealloyed 10 minutes.

Figure 2: Average pore size with increasing time of annealing at 200°C for bulk membrane and for thin film.
Self-Assembly of Non-Spherical Colloids

CNF Project # 1177-03
Principal Investigator: Abraham D. Stroock

Abstract:
There exists an outstanding need for methods with which to form complex, three-dimensional structure on the colloidal scale (10 nm-10 µm). Such methods would be valuable for the development of photonics, high density memory and electronics, and micromechanical devices. An attractive approach to this challenge is to exploit the concepts of chemical thermodynamics to form structure from colloidal building blocks. This approach, based on self assembly, requires the development of a diversity of building blocks (elements), selective and directional interactions (bonds), and methods of processing (reactions). In this project, we are developing a self assembling colloidal system based on direct lithographic fabrication of particles and tailored electrostatic, van der Waals, and depletion interactions.

Summary:
We have developed a procedure by which to form right-cylindrical particles directly from a negative photoresist, SU-8 2002 (Microchem). The key aspects of the process are: 1) spin casting a sacrificial layer (Omnicoat) on a 4-in. silicon wafer; this layer is dissolved in order to release the particles. 2) Spin casting a layer of SU-8; this step defines the height, $h$ [µm] of the particles that will be formed. 3) Exposure of layer of SU-8 through a photomask (GCA Autostep 200 DSW i-line Wafer Stepper) and development; these steps define the diameter, $d$ [µm], of the particles. 4) Release via dissolution of the sacrificial layer, washing via repeated centrifugation, and stabilization in aqueous solution with a non-ionic surfactant (Tergitol, NP-40). This route to the formation of particles generates $\sim 10^9$ colloidal particles on a 4-in. wafer. We have formed particles with thicknesses in the range, $0.2 < h < 3$ µm, and diameters in the range, $0.9 < d < 8$ µm. The nominal resolution of the Autostep 200 is 0.5 µm. We have also generated non-circular, right parallelepipeds in SU-8.

Analysis of scanning electron micrographs (SEM) of lithographically-defined particles in SU-8 indicates that the distributions of size and shape are narrow within a single wafer (Figure S1): $\Delta d = \pm 5\%$ and $\Delta h = \pm 4\%$ for 1:1 and $\Delta d = \pm 5\%$ and $\Delta h = \pm 4\%$ for 1:3. SU-8 is a complex epoxy with a density after curing of $\rho = 1.2$ g/cm$^3$. We note the roughness of the surface of the particles: the sides are significantly rougher than the top and bottom. This contrast in the roughness is likely to play a role in defining the interactions between particles.

Here, we present results for right cylindrical particles of aspect ratio 1:1 ($h = 1.2$ µm, $d = 1.2$ µm) and 1:3 ($h = 0.4$ µm, $d = 1.2$ µm). These right cylindrical particles allow us to explore the role of particle shape in dictating colloidal interactions. The three principal types of interactions that cylinders can undergo: flat-to-flat, flat-to-curved, or curved-to-curved. We expect that the attractive interactions, due to both van der Waals and depletion interactions, will be stronger between surfaces that can form closer contact with one another. Thus, the flat-to-flat conformation should be most thermodynamically favorable, followed by flat-to-curved, and finally, by curved-to-curved. State diagrams of the structures formed in dispersions of these particles illustrate this shape-selectivity: as the electrostatic repulsion between the particles is diminished with rising ionic strength and the depletion interaction is increased with rising concentration of a non-adsorbing polymer, the particles pass through a columnar state in which aggregation occurs with strong selectivity for end-to-end assembly. These results demonstrate the rational design of a colloidal system that exhibits self-assembly of ordered, highly anisotropic structures.
Self-Assembly of Non-Spherical Colloids

CNF Project # 1177-03
Principal Investigator: Abraham D. Stroock
Users: Stephane Badaire, Cecile Cottin-Bizonne, Joseph Woody
Affiliation: Department of Chemical and Biomolecular Engineering, Cornell University
Primary Funding: Cornell Center for Materials Research (CCMR), NSF, Cornell University
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Goals:

1) Investigate the role of shape in defining the interaction between colloidal particles and

2) develop a set of tools with which to self assemble structures on the 100 nm to 100 µm scale.

Figure 1: Fabrication of colloids. (A) Schematic diagram of formation of cylindrical particles in a photocurable epoxy (SU-8) via projection photolithography (Autostep 200 - 10x stepper). (B) Scanning electron micrograph of particles formed by the process in (A). Scale bar = 1 µm.

Figure 2: Selective interactions tuned with shape, electrostatics, and depletion. (A) Orientations of interaction of cylindrical particles. (B) Diagrams indicating structures observed in dispersions of cylindrical particles of aspect ratio 1:1 (h = 1.2 µm; d = 1.2 µm in A) at various ionic strengths, I [mM] and volume fractions of dextrane, φ_d [v/v %]. (C) Optical micrographs of columnar state for 1:1 particle with I = 15 mM and φ_d = 0.5%. Scale bar indicates 10 µm.
Mimicry of Biological Adhesion Through Fabrication of Fibrillar Surfaces

CNF Project # 1225-04
Principal Investigator: Anand Jagota

Abstract:
Geckos and other lizards as well as many species of insects utilize fibrillar contact surfaces to enhance their ability to adhere to a wide variety of natural surfaces. This work seeks both to understand and mimic the distinctive biological geometry and its superior non-covalent adhesion performance.

Summary:
We have shown theoretically that a fibrillar surface in contact with a substrate can possess both larger adhesion force and larger fracture energy than a corresponding flat interface of the same materials [1,2]. To verify this theory and mimic the benefits observed in biological systems, we constructed microscopic fibrillar arrays using the capabilities available at CNF.

Fibrils were constructed with diameter equal to 10 µm, nearest neighbor spacing between 20 and 120 µm and length between 10 and 100 µm. To make these fibrillar arrays, standard photolithography and deep reactive ion etching was used to produce holes in Si with the desired cross-sections and depths. This Si master surface was then used to mold poly(dimethylsiloxane) (PDMS), which was cured and removed from the master to give fibrils of the same shape and size as the holes in the Si surface. To enable release of the PDMS structures from the master, molecular vapor deposition of (1H,1H,2H,2H-perfluoro octyl)trichlorosilane was used to form a hydrophobic self assembled monolayer on the Si.

We showed in previous experimental work [1] that the fibrillar arrays alone are not sufficient to increase adhesion over the flat control value, due to the large loss of contact area accompanying fibrillation. Hence, a thin film was adjoined to the terminal ends of fibrils in this set of samples. This film was made of PDMS approximately 4 µm thick, formed by spinning PDMS onto a hydrophobic SAM coated Si wafer. While the film was still liquid, the fibrillar arrays were placed on the film. The PDMS liquid wetted the PDMS fibrils and was cured in place to form the terminal film. Later, the sample was removed mechanically from the substrate.

The addition of the terminal film is instrumental in the functioning of the fibrillar adhesion arrays in several ways. First, it increases the available contact area to the maximum possible value, equal to that of the flat control. At the same time, the fibril and thin film hybrid is much more flexible than the bulk flat control sample, allowing contact to form more easily with the adherend. It also turns out that the combination of flexibility and contact allowed by the thin film is responsible for increasing adhesion because of unstable crack propagation and contact pinning [3]. Finally, the film maintains the separation and uprightness of the pillars because there is an energy penalty associated with stretching the film that prevents neighboring fibrils from adhering to each other, a problem with free standing fibrillar arrays.

Results for the adhesion of the film terminated fibrillar arrays are positive. One experiment involved peeling the fibrillar sample from a substrate. The energy per unit area associated with peeling the fibrillar samples was found always to be larger than that for corresponding flat controls, by a factor of 2 to 9. Another experiment involving indentation with a spherical indenter showed that the fibrillar pull off force was also larger than the flat control values, by a factor of 1.5 to 3. The variation depends on the precise geometry of the fibrillar array and film. Although a rigorous study is not yet complete, preliminary experiments indicate that adhesion enhancement increases with increasing fibril spacing and decreasing film thickness and fibril diameter.

References:
Mimicry of Biological Adhesion Through Fabrication of Fibrillar Surfaces

CNF Project # 1225-04
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Users: Nicholas J. Glassmaker, Shilpi Vajpayee
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Primary Funding: National Science Foundation (CMS-0527785)
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- A fibrillar array with terminal film mimics the contact and adhesion enhancement achieved by geckos and insects. (Figures 1 and 2)
- Synthetic fibrillar samples constructed by photolithography, dry etch, and molding.
- Fibrillar samples achieved greater adhesion force than flat controls in indentation experiments. (Figure 3).

Figure 1, top left: Fibrillar surface, lizard. (Anolis carolinensis.)
Figure 2, bottom left: Synthetic poly(dimethylsiloxane) fibrillar surface with terminal film.
Figure 3, above: Fibrillar adhesion force, normalized by flat control values.
Development of Quantum Dot Encoded Polystyrene Beads for Use in an Ultra-Miniaturized Microarray Platform

CNF Project # 1235-04
Principal Investigators: Alexander Couzis, Charles Maldarelli, Lane Gilchrist

Abstract:
An ultra-miniaturized micro-array platform, displaying probe molecules, is conceptualized for high-throughput, multiplexed screening applications. The probes are incorporated in a unilamellar phospholipid bilayer supported on polystyrene (PS) beads of ~1 µm diameter. This whole assembly is called a lipobead. The lipobeads are inserted in wells of corresponding size, etched in a square grid pattern on silicon wafers using photolithography and functionalized with self assembling monolayers (SAMs) with pendant groups designed to bind a single bead to the inside of each well. The area on the substrate surrounding the wells is chemically functionalized with a polyethylene glycol (PEG) terminated SAM to resist nonspecific adsorption in screening assays. An encoding scheme based on the fluorescence of hydrophobically modified zinc sulfide (ZnS) capped cadmium sulfide (CdSe) core-shell semiconductor nanocrystals (quantum dots (QDs)) is used for identification of the receptors hosted by individual lipobeads. QDs with different emission wavelengths (colors) and encapsulation concentrations are used to define a code based on intensity (i) and color (c). The encapsulation is undertaken by copolymerizing the styrene and methacrylic acid with the QDs using a suspension polymerization procedure. The bar-coded PS beads with carboxyl termination are then mixed with lipid films incorporating the probes. The array is assembled by first preparing aliquots of beads, with each aliquot consisting of beads with the same QD barcode, mixing aliquots together and finally depositing the bead mixture onto the microwell grid surface. Confocal laser scanning microscope is used for detecting the bar-codes.

Summary:
This array of optically encoded beads is designed to be used as a protein or chemical microarray. Probe molecules are bound to the bead surfaces (with each probe identified with a QD bar code) and screened against targets present in a solution deposited as a microliter drop on the array surface. Binding of a target to a probe is detected usually by the luminescence of a fluorescent label on the target. By reading the code on the bead lit by this label, the identity of the probe molecule binding the target can be obtained. This design has several advantages over conventional protein and chemical arrays. The micron-scale of the beads and the grid allows a registry density of $10^5$, which is orders of magnitude larger than the density of conventional arrays consisting of dried spots of probe molecules with each spot of the order of approximately 100 µm. The encoding capacity of the QDs (i × c) allows for the bar coding of this large number of registries. In addition, by coating the beads with bilayers and sequestering membrane receptors in the bilayers, this platform can be used for the display of difficult to present membrane receptors which require a lipid environment to retain their biological binding ability.

As a proof of concept, 1 µm diameter carboxyl-functionalized fluorescently tagged PS particles (excitation maxima, 529 nm; emission maxima, 546 nm; obtained from Polysciences, Inc.) were arrayed on the 1.2 µm diameter microwell grid. A flat block of polydimethylsiloxane (PDMS) impregnated with PEG-terminated silane was used to put down a PEG-terminal background phase using contact printing. 3-aminopropyltrimethoxysilane (APS) was used to backfill the ‘bare’ wells. This grid was exposed to 0.0025% (wt) solution of particles for 24 hrs and then rinsed with DI water and imaged under the confocal laser scanning microscope. The negatively charged particles are electrostatically coordinated with the positive amine termination of the wells. The passivating PEG functionality surrounding the wells allowed for the easy removal of the non-coordinating particles.

Reference:
Development of Quantum Dot Encoded Polystyrene Beads for Use in an Ultra-Miniaturized Microarray Platform

CNF Project # I235-04
Principal Investigators: Alexander Couzis¹, Charles Maldarelli¹², Lane Gilchrist¹
Users: Shyam V. Vaidya¹, Nikhil D. Kalyankar¹

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Figure 1, top left: Schematic of conceptualized micro-array platform displaying targets interacting with the probe molecules in the lipobead.

Figure 2, bottom left: Microwell surfaces fabricated using photolithography: diameter of wells = 1.2 µm, depth of wells = 0.55 µm, pitch = 3 µm.

Figure 3, above: (a) Confocal laser scanning microscopy image of fluorescing PS particles electrostatically arrayed on functionalized grid surface, (b) Y-section image along the line shown in (a).
High-Throughput Determination of Mechanical Properties using Microcantilever Arrays

CNF Project #: 1236-04
Principal Investigator: R. Bruce van Dover

Abstract:
We are developing a platform for measuring the composition-dependent mechanical properties of materials using an array of microcantilevers. The goal is to be able to investigate the properties of nearly all compositions in a ternary phase diagram with 1 atomic percent (at.%) resolution in a single experiment, and to use this capability to determine trends and identify unusual and/or useful materials in a wide range of ternary systems [1]. Our approach is to prepare a large array of prefabricated cantilevers on a single wafer, deposit a composition-spread thin film on the array, and interrogate each cantilever as a function of some probe stimulus (temperature change, magnetic field, etc.) and thereby infer the composition-property relationship. Our initial experiments are aimed at measuring magnetostrictive materials, for example in the Fe-Ni-Al ternary system. The composition-spread films are deposited using three spatially-separated sputter guns, yielding a typical composition gradient on the order of 1 at.% per mm. In order to determine the properties with ~ 1 at.% resolution, we therefore require microcantilevers located on ~ 1 mm centers. This approach eliminates the run-to-run variations that can confound one-off experiments. With an automated measurement system that is capable of characterizing in one day the response of a 3,000-cantilever array representing an entire ternary phase diagram, this technique enables investigation of magnetostriction in thin films with unprecedented composition resolution and speed. Our “combinatorial” platform will also enable studies of other mechanical properties of advanced materials such as conventional and ferromagnetic shape-memory alloys.

Summary:
All of our device preparation is done at the Cornell NanoScale Science & Technology Facility (CNF). We choose Si$_3$N$_4$ as cantilever material because it is straightforward to deposit and requires only a single lithographic step. We use the nitride tube furnace to deposit ~ 1 µm of low-stress LPCVD Si$_3$N$_4$ on a <100> silicon wafer. Patterns are transferred by standard photolithography using the GCA 5X stepper. After exposure, MIF 300 base developer is used to develop the patterns and PlasmaTherm72 reactive-ion etcher is used to etch the nitride. After stripping photo resist, KOH is used to wet etch the Si to undercut cantilevers—the Si <111> surface acts as etch stop. A CO$_2$ critical-point dryer is used to dry and release the cantilevers to avoid sticking. With our current design, cantilevers are designed to have dimensions of 500 µm x 50 µm and are spaced 1 mm apart in a square array. These dimensions ensure that each cantilever is small enough that the composition of the film on the cantilever is essentially constant, and that composition-dependent properties can be interrogated with a resolution of ~ 1 at.%. The composition-spread thin films are deposited in a custom-designed sputtering system in our lab.

At the center of our measurement design, the cantilever acts as a base substrate for a deposited thin film whose properties (for example, intrinsic stress, shape memory, or magnetostriction) are to be measured [2]. For magnetostriction measurements, the curvature of the deposited bilayer film-substrate cantilevers are measured as a function-applied magnetic field. The cantilever base has essentially no magnetic response, so the Stoney formula for bending of a cantilever under differential stress can be adapted to the present magnetostriction measurements to give:

$$\lambda = \frac{D}{E_s t_s} \frac{(1+\nu_s)}{9\nu_s(1-\nu_s)}$$

where $\lambda$ is the magnetostrictive response, $D$ is the vertical deflection at the end of the cantilever, $E_s$ is Young’s modulus of the cantilever, $t_s$ is the thickness of the substrate, $t_f$ is the thickness of the film, $l$ is the length of a cantilever and $\nu_s$ and $\nu_f$ are the values for Poisson’s ratio for the substrate and film [3], respectively. The deflection of the cantilever depends only on the differential strain between the film and cantilever base material. Thus this approach can be used for measurement of any source of differential strain, such as differential thermal expansion and, importantly, shape memory materials.

References:
High-Throughput Determination of Mechanical Properties using Microcantilever Arrays

CNF Project # 1236-04
Principal Investigator: R. Bruce van Dover
Users: Noble C. Woo, Bryan G. Ng

Affiliations: 1. Materials Science & Engineering; 2. Chemistry and Chemical Biology; Cornell University
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Figure 1, top left: Fabrication process flow of a Si₃N₄ cantilever.

Figure 2, middle left: Cantilever bending measurement setup. Changes in cantilever curvature due to magnetostriction under an applied magnetic field will modify the optical path of the laser, which is detected by a position sensitive detector, analyzed by software, and reported in terms of the differential strain.

Figure 3, bottom left: (Left) Intrinsic stress in thin films of Ni-Fe-Al, as inferred from cantilever deflections as a function of position in a composition-spread sample. Labels indicate the approximate position of the elemental sources creating the composition spread. (Right) Photograph of the sample.
Scanning Electron Microscope and Focused-Ion Beam Analysis of Corrosion Sites on Lead-Tin Alloys

CNF Project # 1346-05
Principal Investigator: Catherine M. Oertel

Abstract:
Focused ion beam (FIB) was used to cut micron-scale cross-sections through corrosion crusts on acetic-acid corroded lead-tin alloys. The exposed surfaces were mapped chemically using SEM/EDX. The results of the mapping provide insight into the role of tin content in susceptibility of these alloys to organic acid attack.

Summary:
Lead-tin alloys are the most common materials used to construct organ pipes, both in instruments built today and in those remaining from past centuries. Corrosion of these pipes is widely observed, eventually leading to cracks and holes that rob the pipes of their ability to produce sound. Especially for irreplaceable historic instruments, this corrosion is of great concern, and research is needed to determine its causes and mechanisms.

For very lead-rich (> 99% Pb) alloys, organic acids have been found to be a major cause of corrosion [1]. The acetic acid emanating from the wood of organ cases is likely to be a source of damage in lead-rich pipes. Our research has as its goal understanding how the susceptibility to acetic acid attack changes as a function of alloy composition. We carried out laboratory exposure experiments in which lead-tin alloy coupons were exposed to low, controlled concentrations of acetic acid vapor. Alloys with tin contents ranging from 1-10% were studied. We used grazing incidence angle x-ray diffraction to identify the bulk corrosion products that formed on the sample surfaces, but we needed more detailed analytical methods that would allow us to characterize the morphology of corrosion products and to resolve chemical information at corrosion sites.

Using the Zeiss Supra SEM at CNF, we obtained images of sample surfaces at magnifications ranging from 100x-4000x. The low-mag images revealed that the surfaces are not covered with uniform corrosion layers but that corrosion crusts with dimensions of 10-100 µm are interspersed among areas with thin oxide coverage. At high magnification, it was possible to see the individual 1-2 µm crystallites making up the thick crust areas.

Next, we used the FEI 611 FIB to cut cross sections of 10-20 µm through corrosion crusts. Compared with traditional sectioning and polishing, this is a gentler method of preparing cross-sections for SEM analysis. The gallium ion beam does not cause mechanical damage to the corrosion crust or the underlying bulk metal, and by following removal of significant material with a high beam current (2000 pA) with a polishing step at lower beam current (500-1000 pA), it was possible to minimize impregnation of the surface with gallium.

Each cross-section was cut at a 45° angle with respect to the surface normal, creating a surface that is horizontal when the sample itself is tilted at 45° in a microscope. In this orientation, SEM/EDX (using the Zeiss Supra) or SEM/WDS (using the JEOL microscope located in Snee Hall at Cornell) was used to map lead, tin, and oxygen content in the oxide crust and underlying metal. For each alloy, tin was segregated along the oxide-metal interface, allowing us to hypothesize that it has a barrier role in the corrosion process. Current work is being directed at using similar cross-sectional analysis to track evolution of this segregated layer with time and under atmospheres of varying humidity.

References:
Scanning Electron Microscope and Focused-Ion Beam Analysis of Corrosion Sites on Lead-Tin Alloys

CNF Project # 1346-05
Principal Investigator: Catherine M. Oertel
Users: Catherine M. Oertel*, Annika Niklasson**

Affiliation: Cornell Center for Materials Research, Cornell University
Primary Funding: NSF Discovery Corps Fellowship Program
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Figure 1, top left: Lead-tin alloys were exposed to low concentrations of acetic acid, simulating the conditions that lead to deterioration of lead-tin alloy organ pipes. This micrograph shows a surface with discrete corrosion crusts separated by areas of thin oxide coating.

Figure 2, bottom left: A FIB cross-section cut at 45° to the surface normal can be studied with SEM/EDX.

Figure 3, above: An image of the cross-section shows the oxide layer and bulk metal, containing tin inclusions.

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**Pathways to Thin Film Ceramic Porous Membranes for Fuel Cell Applications**

**CNF Project # 1356-05**

**Principal Investigator: Ulrich Wiesner**

**Introduction:**

Proton exchange membrane (PEM) fuel cell technology is considered to be a promising alternative for future energy needs due to a high energy conversion efficiency and lower environmental impact. The fuel cell basically contains an anode and a cathode separated by a PEM. The PEM has two major functions: it serves as a proton conductor and as an electronically insulating mechanical separator between the two electrodes. Current technology uses a PEM that is a composite of highly porous Teflon® backfilled with a perfluorosulfonic acid-containing polymer solution commercially known as Nafion®. The Nafion® polymer is the proton conductor whereas the Teflon® provides structural integrity. A major failure mechanism of the fuel cell is due to the expansion/shrinking of the membrane during temperature and hydration cycles. This expansion/shrinking behavior, especially in the plane of the film, may lead to cracking of the membrane which may cause decreased cell performance or complete failure. Those limitations may be overcome by replacing the Teflon® support by a ceramic material like silicon carbonitride (SiCN). Since the ceramic material has a significantly lower expansion coefficient compared to Teflon®, the expansion of a ceramic-nafion composite membrane in the plane of the film should be significantly reduced. Expansion may still occur by increasing the thickness of the film (the z-direction), but this can be managed easier due to cell design. The lower expansion of the membrane in the plane of the film (x-y direction) should increase the resistance of the membrane to failure.

The ceramic we chose to work with is a polyureamethylvinylsilazane (PUMVS), which is a so-called polymer derived ceramic [1]. As the name suggests, polymer derived ceramics start out as a polymer, which can be easily shaped into complex structures. Heat treatment transforms the polymeric precursors into ceramic materials, while retaining the original (complex) shape. Thus these materials have a polymer-derived structure and ceramic-like properties [2]. Here we show the use of polymer derived ceramics in combination with a stamping technique to develop porous ceramic membranes to replace the Teflon® support currently used in PEM fuel cell technology.

**Results:**

The synthesis of the ceramic porous membranes consisted of several steps. First a direct laser pattern generator was used to generate a 5 by 5 cm mask consisting of a hexagonal array of pillars. The diameter of one pillar was 25 µm and the center to center distance between pillars was 33 µm, to ensure 60-80% porosity of the final membrane. The stamp was made by photolithographic patterning of SU-8 by UV exposure through the mask using the HTG contact aligner, followed by baking and development of the non-exposed SU-8.

The stamp was clamped onto a silicon wafer coated with poly(acrylic acid). This assembly was subsequently dipped into a solution of the liquid PUMVS and a radical initiator. Driven by capillary force, the solution filled the interstitial spaces between the stamp and the coated wafer. Heat treatment of the assembly at 130°C in a vacuum oven crosslinked the PUMVS. The film was released by putting the assembly on a water surface and dissolving the poly(acrylic acid). The resulting film was robust enough to pick up with tweezers without disintegrating. Heat treatment to 50°C transformed the membrane into a porous ceramic membrane without losing its integrity.

**References:**


Pathways to Thin Film Ceramic Porous Membranes for Fuel Cell Applications

CNF Project # 1356-05
Principal Investigator: Ulrich Wiesner
User: Marleen Kamperman

Affiliation: Materials Science & Engineering, Cornell University
Primary Funding: General Motors
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Figure 1, top: Stamp consisting of a hexagonal array of SU-8 cylinders.

Figure 2, middle: Stamp and poly(acrylic acid) coated silicon wafer are clamped together. A mixture of polyurea methylvinylsilazane and thermal initiator is drawn into the interstitial spaces by capillary force. The polysilazane is crosslinked at 130°C. The stamp is removed and the porous polysilazane film is lifted off on water.

Figure 3, bottom: Light microscope image of the resulting porous polyureamethylvinylsilazane film.
Nanofabrication of Multifunctional Nanoscale Materials within Nanoporous Materials and on 2-D Substrates

CNF Project # I448-06
Principal Investigator: Tewodros Asefa

Abstract:

Our research group is interested in developing novel nanostructured materials possessing multifunctional groups; these materials are created by combining “bottom-up” and “top-down” synthetic approaches such as electrodeposition, electroless deposition, chemical vapor deposition, self-assembly, sol-gel processing, wet-chemical synthesis, and reactive ion etching. The two or more functional groups or structures introduced into these materials have resulted in the material possessing two or more properties which allow them to perform either multiple applications or to increase the efficiency of the optical response and the magnetic and catalytic activities of the materials. By virtue of their multifunctional groups as well as structures at the nanoscale, these materials will find future applications in photovoltaics, nanoelectronics, and nanophotonics devices and for catalysis and biomedical applications.

Summary:

The design, synthesis and self-assembly of nanostructured materials are important steps leading to the development of novel miniaturized nanoscale devices for optical, electronics, photonics, sensing, biological or medical applications. To realize the tremendous potential of nanomaterials for diverse applications, we use various “bottom-up” approaches of wet-chemical synthesis and molecular self-assembly combined with “top-down” approaches of engineering nanomaterials to create novel nanoscale materials having multiple functional groups with well-defined nanoscale structures.

In one approach, we prepare nanoporous alumina substrates (0.2 µm diameter and 10 µm width) via electrochemical anodization of aluminum thin films [1] and we use the resulting nanoporous materials as hard-templates to produce silica and organosilica coated conductive metallic nanowires such as copper, silver and gold via electrodeposition.

To perform the electrodeposition in the nanochannels of these materials, we first deposit thin conductive layers of metals on one side of the substrate either via sputtering or thermal evaporation. We have successfully used aluminum sputtered and silver thermal evaporated nanoporous alumina substrates for our electrochemical deposition experiments. Results to date indicate that the Ag thermal evaporated substrates work better for copper electrodeposition inside the nanochannels of the alumina forming well-defined nanowire structures. We speculate this success is because the silver thermal deposition on the nanoporous alumina formed smooth conductive layers. By changing the deposition times and deposition potentials, we were able to collect free standing metal-silica (or metal-organosilica) core-shell nanowires. The silica or organosilica layer in these materials, which has low dielectric constant, provides an insulating layer to the metal and would be an important component when assembling these nanowires on solid devices for future nanoscale electronic components [1]. The nanowire materials we prepared before and after deposition are characterized by methods such as scanning electron microscopy, transmission electron microscopy, atomic force microscopy, UV-Vis-NIR spectroscopy and X-ray diffraction.

In other work, we prepare metal-thiolate precursors by reacting metal chlorides with alkanethiols and we spin-coat the resulting metal-thiolate precursors on noble metal nanopattared Si-wafers. By performing e-beam lithography on the metal-thiolates decorating the substrates, we form nanopatterns and write semiconductor metal-sulfide nanostructures on the substrates by using seeded mediated nanostructure formation. For instance, by depositing Zn-dodecanethiolate on gold nanoparticle containing Si-wafer substrate followed by e-beam writing, patterns of semiconducting ZnS nanoparticles or nanowires preferentially grow around the gold nanoparticles. Electron beams are used to pattern structures of noble metals such as gold nanoparticles from gold-thiolate on solid substrates. We characterize the resulting substrates with UV-Vis-NIR, atomic force microscopy and scanning electron microscopy.

References:


Nanofabrication of Multifunctional Nanoscale Materials within Nanoporous Materials and on 2-D Substrates

CNF Project # 1448-06
Principal Investigator: Tewodros Asefa
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Figure 1, left: Synthetic scheme of nanotube and nanowire deposition within the nanochannel pores of porous alumina “hard-template”. The arrows indicate nanotubes and nanowires that can be deposited within the nanoporous alumina via electroless deposition or electrodeposition, respectively.

Figure 2, below: Copper nanowires electrodeposited inside Ag-sputtered nanoporous alumina channels. This side-view image shows the copper nanowires as well as empty channels of the alumina nanochannels that are not yet filled. By controlling the deposition times and electrodeposition potential, we can grow metallic nanowires of various lengths.
High Quality Factor Nanostring Resonators

CNF Project # 599-96
Principal Investigator: Harold G. Craighead

Abstract:

We have fabricated nanomechanical flexural resonators with room temperature quality factors as high as 207,000 at a frequency of 4.5 MHz, representing the highest quality factor yet obtained for devices with cross-sectional dimensions on the order of 100 nm. We demonstrate that the high quality factors are a result of the high tensile stress present in these doubly-clamped devices, and demonstrate an approach to within an order of magnitude of the thermoelastic limit for mechanical quality factor for devices of this scale. These devices, with the highest ever reported product of quality factor and surface-to-volume ratio, should prove useful as sensitive probes of the environments in which they are operated.

Summary:

Nanomechanical flexural resonators have been fabricated and studied, using different fabrication techniques, and from different materials. Doubly-clamped nanoresonators made in high stress silicon nitride exhibited quality factors as high as 207,000 at a frequency of 4.5 MHz, and at room temperature, the highest $Q$ ever reported for flexural resonators with cross-sectional dimensions on the order of 100 nm. These high quality factor devices were fabricated from electron-beam defined masks, as well as electrospun masks. Resulting $Q$s were shown to be independent of the fabrication steps, implying the high $Q$s are a result of the high stress silicon nitride material. Lower stress resonators have also been fabricated, including doubly-clamped beams made from a lower stress film, and stress-free cantilever beams, yielding for devices of either the same size or the same frequency, quality factors significantly lower than the high stress devices. We have demonstrated that our lower frequency devices, up to 50 MHz, exhibit a variation of $Q$ with frequency consistent with what would be expected from considerations of thermoelastic dissipation. At higher frequency, this trend begins to deviate from the thermoelastic limit, possibly due to the increased importance of clamping losses for the higher frequency, shorter devices. Devices with such small cross-sectional dimensions, and hence large surface-to-volume ratio, with such high quality factor, should prove useful for the sensing of small biological molecules, as well as the probing of the physics of the interaction of such small mechanical devices with their surroundings.

References:

High Quality Factor Nanostring Resonators

CNF Project # 599-96
Principal Investigator: Harold G. Craighead
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Primary Funding: NSF/CCMR
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- Highest quality factors ever reported for flexural resonators with cross sections on the scale of 100 nm with Q above 200,000 at 4.5 MHz and at room temperature.

- Nanoscale devices with such high quality factors have been fabricated using electrospun nanofiber masks, as an alternative to electron beam lithography.

- High quality factors are a direct result of the high tensile stress present in the doubly clamped devices.

Figure 1, left: SEM images of a suspended silicon nitride beam. A 300 nm scale bar is shown.

Figure 2, below left: Optically measured response of a 200 nm wide, 105 nm thick, 60 µm long resonator, with a Q of 207,000 at room temp.

Figure 3, below right: Quality factor data for doubly clamped high stress beams and stress-free cantilevers, plotted with the limit given by thermoelastic dissipation.
Biological Detection via Resonant Nanoelectromechanical Sensors

CNF Project # 599-96
Principal Investigator: Harold G. Craighead

Abstract:
In this work, we have taken advantage of the mass-sensing capabilities of nanomechanical resonant devices and their high sensitivity for the detection of particular biological species. We functionalize these nanoscale devices with the appropriate receptor molecules specific to the species we desire to detect, in order to selectively weigh those analytes. From this information we can calculate analyte concentrations. Extending these devices into multiplexed arrays of separately functionalized detectors would allow for compact screening tests to detect disease markers and harmful biological substances or to track disease progression in a low-cost and rapid way.

Summary:
Nanoelectromechanical systems (NEMS) have attracted much attention as candidates for biological sensors due to their compact size, compatibility with semiconductor processing technologies, and sensitive frequency response to added mass [1]. These devices operate as miniaturized analogues of quartz crystal microbalances, exhibiting shifts in their resonant frequencies upon the addition of mass, such as bound biological species. Using nanoscale oscillators, resonant frequencies, and thus sensitivity to added mass, can be increased sufficiently for the detection of single viruses [2] or molecules [3]. Similar detection schemes include static deflection of microcantilevers resulting from induced surface stress of bound objects [4], but require multiple layers of fabrication, and much larger cantilevers, hindering integration in large scale arrays for detection of multiple analytes.

For precise detection of biological objects, specificity is required, as resonant NEMS will respond to any adsorbed mass. We have worked to functionalize sensor surfaces using appropriate linking chemistries in order to present a surface of bound receptors, antibodies, for example, specific to a particular analyte. Reducing nonspecific binding of proteins to the nanomechanical biosensors is also an important consideration. With sufficient confidence in the specificity of the cantilever surface, in addition to appropriate controls, resonant frequency shifts can be attributed solely to adsorption and thus detection of the desired analyte. So far, we have measured concentrations as low as ~1 ng/mL using nanomechanical resonators.

In this work, we have fabricated NEMS devices from low stress silicon nitride, with thicknesses on the order of 100 nm. Typical devices have lengths and widths on the order of a few microns. Derivatives of the standard cantilever beam shape, in addition to other interesting structures, are being investigated to improve sensitivity to low concentration detection. Resonant structures are fabricated by patterning the nitride device layer on a sacrificial layer of thermally oxidized silicon. Isotropic etching of the oxide releases the devices, making them free to oscillate. We use an optical system in order to thermally excite device oscillation and detect the resonant frequencies from modulation of the light reflected from the oscillating structure [4]. Future work will utilize arrays of NEMS biosensors for multiplexed biological detection from a single sample.

References:
Biological Detection via Resonant Nanoelectromechanical Sensors

CNF Project # 599-96
Principal Investigator: Harold G. Craighead
User: Philip S. Waggoner

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Figure 1: Schematic of nanomechanical biological sensor fabrication. Device stack is grown (a) and patterned via standard lithographic techniques (b). The structures are released by an HF dip (c), then functionalized and used as a highly sensitive mass sensor, shown here to detect an antigen using an antibody-functionalized cantilever (d).

Figure 2: SEMs of two NEMS devices used to enhance sensitivity to single analytes. Both scale bars are 2 µm.
Nanofluidic Devices for Single Molecule Spectroscopy

CNF Project # 762-99
Principal Investigator: Harold G. Craighead

Abstract:
Micrometer and sub-micrometer fluidic devices are used in a variety of applications for analytical biochemistry. Previous work describes the benefits of performing single molecule spectroscopy in small engineered focal volumes with regard to uniform illumination intensity and controllable analyte flow [1]. The goal of this project is to further reduce the engineered focal volume with sub-100 nm fluidic channel dimensions. Reduction of the focal volume will reduce the probability of multiple molecule occupancy and could lead to spectroscopy with higher accuracy. Biophysical interaction at the channel-fluid interface becomes increasingly dominant at smaller dimensions and therefore warrants comparison of the practical benefits of each design [2].

Introduction:
A two-step lithographic process was used to create these nanofluidic devices. Electron beam lithography was used to pattern the nanofluidic channel region and an anisotropic reactive ion etch of the fused silica formed channels of dimension 100 nm by 45 nm, as shown in Figures 1 and 2. A photolithography step defined the reservoirs and analyte supply channels to the nanofluidic channel region. Channels were sealed in a direct bonding process using a fused silica cover wafer, to reduce autofluorescence and allow standard coverslip-corrected microscope objectives to be used.

To provide a comparison with nanofluidic devices, sub-micrometer fluidic channels were created with a similar process involving a single photolithography step. Sub-micrometer channels were patterned with projection photolithography and then created with an anistropic reactive ion etch resulting in channel dimensions of 500 nm by 250 nm, as shown in Figure 3. Direct bonding with a fused silica cover wafer is used again [3].

Fluidic channels have been used in experiments involving fluorescent dye labeled DNA and proteins. In particular, a relatively-new molecular engineered probe, the molecular beacon [4], is under investigation for use with fluidic channels in single molecule spectroscopy.

Summary:
Sub-micrometer (500 nm x 250 nm) and nanometer (100 nm x 25 nm) scale fluidic channels were fabricated in fused silica to conduct single molecule spectroscopy. A nanometer-scale engineered focal volume permits micromolar range sample concentrations, while increased biophysical interactions at the channel-fluid interface warrant comparative study with sub-micrometer channels.

Investigation was conducted using molecular beacons in fluidic channels to provide more robust single molecule spectroscopy, with possibilities for the analysis of heterogeneous analyte solutions. The highly sequence-specific nature of molecular beacons, combined with rapid fluidic channel analysis, presents new possible opportunities for early detection of disease.

References:
Nanofluidic Devices for Single Molecule Spectroscopy

CNF Project # 762-99
Principal Investigator: Harold G. Craighead
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• Rapid analysis of analytes confined within the fluidic channel.
• Materials compatible with several types of biology and conducive to study with conventional microscope optics.
• Straightforward fabrication process requiring 2-step lithography or less.

Figure 1, top left: Optical micrograph of electron beam lithography fabricated nanofluidic channels.

Figure 2, bottom left: Atomic force microscope image of a single nanofluidic channel profile (approximately 100 nm wide x 45 nm deep).

Figure 3, above: Optical micrograph of an array of sub-micrometer fluidic channels. Spectroscopy is performed in the narrow region (500 nm wide x 250 nm deep) of each channel near the numbers.
Ultrasensitive, Magnet-Tipped Cantilevers for Magnetic Resonance Force Microscopy

CNF Project # 863-00
 Principal Investigator: Professor John A. Marohn

Abstract:
Magnetic resonance force microscopy (MRFM) is a developing technology in the family of force microscopy techniques. MRFM detects magnetic resonance as a force on a magnet-tipped microcantilever facilitating three-dimensional, chemically specific subsurface imaging at the nanoscale [1]. If sufficiently high sensitivities can be reached, this technique could achieve atomic scale magnetic resonance imaging, and could be used, for example, to read out the structure of large biomolecules or to study buried semiconductor interfaces. An essential step in achieving the required sensitivity is the development of high sensitivity cantilevers with nanoscale magnetic tips. Our work at the CNF has focused on creating 50-200 nm wide cobalt magnets which extend from the tips of 5 µm wide silicon cantilevers.

Summary:
The purpose for creating overhanging, nanoscale magnets is to maximize the force exerted on the cantilever by each magnetic spin, while minimizing noise in the force signal that arises from non-contact frictional forces between the cantilever and the sample. To achieve single-spin sensitivity, the front of the magnet must be within a few nanometers of the sample. Work by our group [2] has found that, within tens of nanometers of sample surfaces, metal has less friction than silicon, and that narrow cantilever tips have less friction than wider ones. Thus our cantilever design has the magnet extending past the end of the silicon cantilever, and the very tip of the silicon cantilever is narrowed from 5 µm to 1.

The fabrication process starts with <111> oriented silicon-on-insulator wafers. Electron-beam lithography with the JEOL-9300 is used to define the magnets, which are created through thermal deposition of cobalt and subsequent liftoff. A thin layer of oxide is deposited using the GSI PECVD to protect the magnets in subsequent processing. Next, the electron-beam lithography is used to define rectangular etch pits adjacent to the magnets. Plasma etching is used to etch these pits through the device layer of the wafer. The wafer is then etched in heated KOH. The design of the etch pits, the <111> orientation of the silicon, and the anisotropy of the KOH etch combine to undercut the silicon below a portion of the magnets, leaving the magnets extending approximately 200 nm from the post-etch, etch pit edge [3]. The cantilever body is defined with the GCA Autostep, and etched. A second, thicker layer of PECVD oxide is deposited to protect the devices during backside processing. The backside of the wafer is patterned using the EV-620, and etched through to the buried oxide layer using the Unaxis 770. The cantilevers are released in buffered hydrofluoric acid etch, followed by critical point drying.

References:
Ultrasensitive, Magnet-Tipped Cantilevers for Magnetic Resonance Force Microscopy

CNF Project # 863-00
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Users: Steven A. Hickman, Boyan I. Penkov

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Figure 1: SEM of test structure for MRFM cantilever tip magnet fabrication process, with dashed lines indicating the position of the cantilever relative to the magnet.

Figure 2: Before (left) and after pictures showing how overhanging magnets are created by KOH etching.

Figure 3: MRFM cantilever with non-overhanging, 200 nm wide Co magnet. This design is less effective for MRFM than cantilevers with overhanging magnet tips due to noise from non-contact friction.
Highly Compliant Gimbaled AFM Probes

CNF Project # 883-00
Principal Investigator: Frederick Sachs, PhD

Atomic Force Microscopy:

The operating principle of the AFM [1] is simple: a soft probe interacts with the sample, and its deflection is measured by reflecting a laser from the back of a microfabricated cantilever onto a 4 segment position sensitive photodetector (PSPD)-an arrangement termed the “optical lever” [2]. The difference between the sum signal of the top and bottom (T-B) segments of the PSPD is directly related to the probe’s displacement in z. Left-right (L-R) signal has been used to monitor the cantilever’s twisting and these measurements are often related to friction [3]. It has been shown that the sensitivity of the AFM probe (cantilever) is the limiting factor in mechanical resolution of the AFM [4]. The cantilever’s disabilities are especially pronounced when the AFM is operated in fluid. As viscosity of the environment around the cantilever increases, viscous drag becomes significant and additional fluid coupling to the cantilever increases mass. Both viscous drag and mass increase decrease sensitivity and dynamic response of the cantilever [5].

Soft Gimbaled AFM Probes:

We have recently developed torsion AFM levers that have minimal moving areas and lower noise. The fabrication process was developed and optimized at CNF; it requires four contact photolithography layers and allows us to produce a highly compliant and ultra-small (20 x 20 µm) torsion probe [6]. These levers allow more sensitive operation in liquid and better thermal stability than commercially available levers. In addition, our fabrication process allows for design flexibility in three dimensions and we have utilized this fact to produce dual-axis gimbaled levers. These levers have two sets of orthogonally-arranged soft SiN hinges onto which we mount a pad and a gimbaled mount (Figure A). These AFM probes will be useful in a wide range of applications, due to the fact that we can uncouple movement in two directions using a single optical lever (Figure B). The entire structure moves in the z direction and shows up as a 26 kHz resonant peak in the B-T signal (Figure B, left). The inner pad rotates on an orthogonal axis and it has a 95 kHz resonant peak on the L-R channel (Figure B, right).

Dual-beam AFM operation has been shown to eliminate instrumental drift and minimized noise to allow pN force resolution [7]. However, this setup requires a dual optical lever, which is currently not available on commercial AFMs. Our dual axis lever accomplishes the same task with a single optical lever. A single laser beam illuminates both axes, but the two axes of the PSPD (B-T, & L-R) now become useful independent data channels. External z-noise sources can be removed by taking the difference of the two signals (after appropriate scaling for differences in optical gain of the two axes). This cancellation will be effective at least up to the lower of the two resonant frequencies. Noise of the laser system can also be suppressed since both axes are illuminated by the same laser.

Presently, we are optimizing such probes as combined friction/topography AFM probes (Figure A), gimbaled AFM probes that will minimize noise by differential cancellation (Figure A), drift-free/noise-free AFM probes that will measure sample position and noise against substrate position/noise (Figures C & D).

The probe’s specific design can be modified according to the desired application. Our process allows a simultaneous production of soft hinges for the measurement levers and production of either soft SiN hinges (Figure C) or stiff Si hinges for the reference hinges (Figure D). Soft reference hinges maximize force sensitivity while stiff hinges allow stable reference contact and maximum noise reduction.

Acknowledgements:

The results presented in this work are due in part to the knowledge, expertise and physical support provided by the CNF staff. We especially want to acknowledge the help of Meredith Metzler, whole advice on the etching techniques was especially helpful. Supported by NIH and NSF.

References:

Highly Compliant Gimbaled AFM Probes

CNF Project # 883-00
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Primary Funding: NIH, NSF
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Figure A, top: Typical gimbaled AFM lever. Orthogonally-oriented hinges are sensitive in 2-D. This particular lever may be used as a sensitive probe for friction measurements. One can use $\delta(B-T)$ for scanning topology, while simultaneously recording $\delta(L-R)$ as friction at high sensitivity.

Figure B, middle: Resonance peaks for the outer gimbal (left) and inner pad (right) of Figure A. Distinct resonance peaks imply ability to sensitively monitor two movement axes. The typical spring constants for the pad are $\sim 0.01$ N/m.

Figures C & D, bottom: Alternative geometric configurations and additional tips allow production of AFM referencing levers. These levers will use the referencing tip (on the outer gimbal) to monitor position of the substrate, while the measurement tip (on the pad) will record on the orthogonal data channel.
Using Surface Chemistry to Control the Quality (Q) of Micromechanical Resonators

CNF Project # 891-00
Principal Investigator: Melissa A. Hines

Abstract:

MHz-range torsional resonators with Si(111) faces have been fabricated using a combination of lithographic processes and KOH etching. The dissipation of mechanical energy in the resonators is found to be strongly dependent on the chemical nature of the surface. The quality factor and long-term stability of the resonators can be significantly improved by a methyl monolayer directly bonded to the silicon surface. Mechanical energy dissipation in functionalized resonators is shown to be a sensitive function of surface chemistry. At least 18% and 41% of the dissipation in H-terminated and long-chain alkyl-terminated resonators, respectively, is surface-related.

Summary:

The development of stable, high performance micro- and nanomechanical resonators would enable advances in many fields, including chemical and biological sensing. In these applications, an adsorbed mass leads to a small shift in resonant frequency. The sensitivity of resonance-based detectors depends crucially on the resonator’s stability and quality factor, Q. (Roughly speaking, the Q of a resonator is the number of oscillations the resonator will undergo after an impulsive excitation. As a result, Q is inversely proportional to the rate of mechanical energy dissipation.) The development of stable, high performance micro- and nanomechanical resonators would enable advances in many fields, including chemical and biological sensing. In these applications, an adsorbed mass leads to a small shift in resonant frequency. The sensitivity of resonance-based detectors depends crucially on the resonator’s stability and quality factor, Q. (Roughly speaking, the Q of a resonator is the number of oscillations the resonator will undergo after an impulsive excitation. As a result, Q is inversely proportional to the rate of mechanical energy dissipation.)

Our research has shown that mechanical energy dissipation in the MHz-range, micromechanical resonators is often dominated by the chemical state of the surface. By changing a single monolayer of molecules on the surface of a 5-µm-wide, 250-nm-thick silicon resonator—less than 0.07% of the total mass—the quality factor of the resonator can be improved at least 70%. In contrast, the standard commercial coating, a thin layer of silicon oxide, dissipates at least 75% of the mechanical energy in similarly sized resonators.

Interestingly, the inherent mechanical properties of monolayer-thick surface coatings have little effect at this length scale. For example, resonators terminated by thick monolayers [e.g., octadecyl (C_{18}H_{37}) groups] dissipate slightly less energy than those terminated by much thinner monolayers [e.g., ethyl (C_{2}H_{5}) groups.] Instead, the quality factor is sensitive to the density of the monolayer. For example, resonators terminated with long-chain alkyl monolayers, which are limited to 50% coverage by steric effects, have significantly lower quality factors than methyl-terminated resonators. By virtue of their relatively small size, the methyl groups terminate every site on the Si(111) surface.

References:


Using Surface Chemistry to Control the Quality (Q) of Micromechanical Resonators

CNF Project # 891-00
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- Resonator quality factor (Q) affected by one monolayer of molecules.
- Methyl-terminated resonators have high Q and excellent stability.
- The standard oxide coating leads to very low Q.

Figure 1: 7 μm wide, 250 nm thick hexagonal paddle oscillator fabricated from Si(111) wafer and suspended by 450 nm wide silicon wires.

Figure 2: The effect of surface chemistry on the quality factors of 5 MHz resonators.
Fabrication of Nanochannels in Glass

CNF Project # 1096-02
Principal Investigator: Lukas Novotny

Abstract:
Current project has been dedicated to fabricating 500 nm wide and 500 nm deep channels in glass wafers. Each wafer assembly consists of two glass wafers bonded to each other and contains eight flow-cells isolated from each other. Each flow-cell consists of two reservoirs, 3 mm away from each other, and 1 mm wide and 500 nm deep channel which connects these reservoirs. Halfway from the reservoirs, the channel is divided by a 15 µm wide and 500 nm high ridge, in which 500 nm wide channels are made.

In the past years, the nanochannels were used in the project of developing the new method for nanoparticles recognition [1,2]. Nanoparticles are recognized by measuring the optical force acting on nanoparticles in a strongly focused laser beam [3]. Currently, the nano-channels are used in the project for single viruses recognition and classification using light scattering.

Fabrication Procedure:
A borosilicate glass wafer (Schott Glass, Germany) was pre-cleaned in RCA1 solution at 70°C for 20 minutes. The wafer was vapor primed in the YES oven. The wafer was spin-coated with i-line photoresist (OIR 620-7i) at 3000 RPM for 30 seconds with three seconds ramping speed. The nano-sized parts of the flow-cell were patterned using 10X stepper (GCA corp., Andover, MA), and the micro-sized features were patterned using EV620 contact aligner (Electronic Visions, Phoenix, AZ). The channels were etched using a reactive ion etching technique in the Plasma Therm 72. The remaining resist was then removed by soaking the wafer in nano-strip solution at 80°C for 10 minutes. A second glass wafer was used to seal the channels. Holes for liquid delivery were made in the second glass wafer using a sand-blasting tool. Two wafers were cleaned in the RCA1 cleaning solution and then bonded together under 2000N pressure at 550°C for 10 hours. Later, the holes in the top wafer are covered with pieces of parafilm to protect the nanochannels from dust.

Summary:
The fabrication procedure has been developed for repeatable and reliable fabrication of the nanometer-sized channels, sealed between two glass wafers.

References:
Fabrication of Nanochannels in Glass

CNF Project # I096-02
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Figure 1: Optical micrograph of the fabricated glass nanochannels.

Figure 2: AFM topography of a nanochannel.
Directional Microphone Array Fabrication

CNF Project # 1116-03
Principal Investigator: Ronald N. Miles

Abstract:

The goal of this project is to fabricate prototypes of novel silicon microphone arrays. The microphone arrays consist of two differential microphones and one omni-directional microphone on each die. The miniature directional microphone technology is being developed for applications in hearing aids.

Introduction:

Several new microphone designs and fabrication processes were developed and refined this past year. These designs included microphones that utilize conventional capacitive sensing as well as microphones that incorporate optical gratings to allow an optical detection of the diaphragm displacement. The capacitive microphones are composed of polysilicon diaphragms and polysilicon backplates separated by a small gap to permit capacitive sensing. The essential fabrication steps include deep reactive ion etch, sacrificial oxide growth, doped polysilicon deposition, chemical mechanical polishing, phosphorus-containing silica glass deposition, backplate polysilicon deposition, backside cavity etch, and final structure release with buffered HF etch.

The effect of the angle of incidence of the sound on the response of our differential silicon microphones has been measured and shown to be in excellent agreement with theoretical predictions.

Other activities included polysilicon chemical mechanical polishing; development of dry-release technology; development of a backside RIE recipe; experiments on fabrication of sputtered aluminum backplates and gold plated backplates; fabrication of gold backplates using electro-plating technology; and fabrication of conventional capacitive microphones with gold backplates.

Some of the challenges encountered at CNF included the re-characterization of the doped polysilicon furnace, some tool difficulties and equipment down time.

Summary:

Several polysilicon microphone designs have been successfully designed, refined, produced and tested. Acoustic testing of these parts shows a very satisfactory yield and performance. The next step in this project is the refinement of the MEMS prototype microphone circuits and package, which is currently underway. The package will be used to demonstrate the performance and limitations of the MEMS directional microphones and circuitry and allow the team to address issues prior to circuit miniaturization.

References:

Directional Microphone Array Fabrication

CNF Project # 1116-03
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- Goal: Fabricate prototypes of novel silicon microphone arrays.
- The capacitive microphones consist of polysilicon diaphragms and polysilicon backplates.
- Microphone arrays included two differential and one omni-directional microphone as shown in Figure 1.
- The acoustic directivity pattern of the differential microphones corresponds to the “ideal” figure-eight pattern as shown in Figure 2.
- Primary application: hearing aids.

Figure 1: Chip containing an array of two differential and one omni-directional microphone. The chip dimensions are 4.5 x 5.5 mm.

Figure 2: Polar plot showing the response of our differential microphone as a function of the angle of incidence of the sound.
Merging 2-D Ultrasonic Arrays with Microsystems

CNF Project # 1121-03
Principal Investigator: Prof. Amit Lal

Abstract:
Our goal in this project is to develop a flexible platform that can merge a given microsystem with off-chip piezoelectric actuators and drive electronics. Bulk piezoelectric actuation is commonly used in peristaltic pumps and bimorph/unimorph type actuators due to its efficient force generation even at low voltages. Large size and thickness (> 100 µm) of the ceramic is the main challenge towards an integrated approach that does not include manual adhesive bonding. To address this issue, we work on a process in which micromachined PZT pillars can be sandwiched between a circuit or padframe die at the bottom and a MEMS die on top. This can be used for other types of microsystems as well as fluidic pumps [1].

Introduction:
The brief operating principle of the pump which we will use to test the prototype of the architecture can be described as follows: Individual pixels of PZT pillars excite waves on the surface of the MEMS die which will have fluidic channels. If there is only one pixel actuated, this individual source causes standing waves inside the channel. Standing waves creating nodes or antinodes along the channel do not have the capability for continuous flow. On the other hand having a neighboring pixel also emitting acoustic radiation can cause propagating fields between these two actuators if the phase of the latter is properly adjusted. This phased drive scheme can then yield programmable, multidirectional pumps if used in a 2-D array fashion.

Reminiscent of a 2-D ultrasonic imaging array, the device consists of 20 x 20 pixels of PZT-4. The bottom die houses the individual pixels and takes care of the metal routing. It can also have pixel based driving electronics although this is not realized in our device. The Sn/Pb solder bumps electroplated to the pads can be seen in the optical picture in Figure 1. They act as adhesive agent during the flip-chip bonding process. The machining of the PZT pillars is done using a wafer saw. Although for the prototype we stick to 250 µm pitch, we have optimized pre/post-dicing processing along with the dicing recipe to give us 500 µm long PZT pillars down to 150 µm pitch without any bad pixel. Figure 2 shows the SEM micrograph of a diced array. Next step in the assembly is the alignment and bonding of these pillars to the padframe on the bottom. Figure 3 gives a picture taken after this bonding process.

Summary:
We work on a process flow to enable integration of 2-D ultrasonic arrays with microsystems. The current device has problems mainly arising from manual flip-chip bonding process like shorts between individual pixels or non-uniform bonding. This and other problems will be addressed in future runs by process changes as well as using an automatic flip-chip bonder. If the process flow is successful, the architecture will have the flexibility to serve in inkjet heads, programmable fluid pumps and similar applications that can benefit from localized off-chip actuation. It can also be helpful in experiments which help us model the coupling between bulk-PZT and surface micromachined electrostatic resonators [2].

References:
Merging 2-D Ultrasonic Arrays with Microsystems

CNF Project # 1121-03  
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- Bulk PZT Actuation  
- Off-Chip Actuation  
- 2D Transducer Array

**Figure 1**, top left: Optical picture of the solder bumps on top of the aluminum pads. Bonding is done by reflowing after alignment of the pillars.

**Figure 2**, bottom left: SEM micrograph of the diced PZT array leading to pillars of 500 µm long at an array pitch of 150 µm.

**Figure 3**, above: Picture of the pillars bonded on the padframe manually. Some of the pixels are shorted after reflow.
Hybrid MEMS-CMOS Cantilever Switches

CNF Project # I12I-03
Principal Investigator: Amit Lal

Abstract:
A hybrid MEMS-CMOS transistor is proposed to reduce drain-source leakage current. Actuated by the gate voltage to match the on and off states of a transistor, a cantilever replaces the drain on an NMOS transistor. The cantilever-based switching and reduction in drain-source leakage current is reported.

Summary:
As transistor design rules are aggressively scaled, source-drain leakage current (SDLC) has increasingly become the major problem as it increases the static power consumption in SRAMs. By eliminating SDLC, SRAMs can be better suited for ultra-low standby-power applications. We present a way of reduction of source-drain leakage current in MOSFET transistors by using micro cantilevers.

In our implementation of a hybrid MEMS-CMOS inverter, two cantilevers, connected respectively to VDD or GND are used to supply the rail voltages the NMOS and PMOS respectively, when actuated. The inverter input leads to the correct transistor (NMOS if $V_{in}$ = high, PMOS if $V_{in}$ = low) such that the transistor with potential SDLC will be disconnected from the power supply to eliminate leakage current. The cantilever dimensions were designed for CMOS-compatible pull-in voltages. The static power in the cantilever case is limited by the Brownian motion causing displacement currents across the air gap. We have developed a process-flow to post-process NMOS transistors made on SOI [1]. A sacrificial layer of 400 nm of photoresist was used. In order to minimize compressive stress in the resulting thin film, 300 nm of Al was e-beam evaporated at a low rate of 1 Å/sec. We added a 180 nm isolation layer of oxide on top of the gate to prevent the cantilever switch from shorting the gate.

To verify the operation we conducted the following experiments:

1. SDLC elimination in a $W/L = 100 \mu m / 100 \mu m$ transistor: In the very leaky, large transistor, the switching mechanism causes the drain current to remain zero (within noise-limited measurement) until the cantilever is pulled in, whereby the drain current becomes that of a traditional transistor (~ 250 microamps). The dotted line shows sub-threshold SDLC in a traditional transistor.

2. Verification of cantilever control: In a smaller transistor of $W/L = 20 \mu m / 10 \mu m m$, the cantilever is stuck to the source due to adhesion. As the gate voltage is increased, the behavior is that of a traditional transistor. Then this measurement is repeated, except when the gate reaches 4.7 V the die is vibrated by gently tapping the probe chuck. The cantilever desticks from the drain, turning the transistor off. The drain current drops to zero until the cantilever is again pulled-in by the gate voltage and the drain current returns to its normal operating value.

The post-process appears to affect the transistor characteristics, although none of the steps are thermally stressful except for photoresist post-bakes at 115°C for a few minutes. In later processes, it is hoped that this effect will be removed or controllable. We also propose to used an oxide sacrificial layer in the future to address the problem of non-uniformity of the photoresist sacrificial layer over the die.

It is projected by ITRS that at feature sizes around 65 nm, the standby power of microprocessors will exceed the active power. Therefore, although the cantilevers and MOSFETS studied here were very large, the utility of the switching scheme becomes more important at smaller scales. It is especially attractive as the pull-in voltage will decrease as cantilevers scale down with device features, and the limit on the frequency of operation due to mechanical restrictions will increase.

References:
Hybrid MEMS-CMOS Cantilever Switches

CNF Project # 1121-03
Principal Investigator: Amit Lal
Users: Shankar Radhakrishnan, Norimasa Yoshimizu

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- A hybrid MEMS-CMOS transistor is developed to vastly reduce source-drain leakage in CMOS.
- A two-mask post-CMOS MEMS process that causes very low thermal stress on the CMOS was developed.
- Cantilevers designed to be compatible with CMOS switching voltages were designed and fabricated on NMOS transistors.
- Cantilever actuation using gate input voltage was successfully demonstrated, showing a reduction in drain-source leakage current.

**Figure 1**, top left: SEM image of cantilever switch. The area of source-cantilever is much smaller than the gate-cantilever, leading to majority of the cantilever control for the gate electrode.

**Figure 2**, bottom left: Change in drain current of W/L = 100 µm / 100 µm NMOS transistor as cantilever is pulled-in. Drain current below pull-in is noise-limited.

**Figure 3**, above: Drain current of W/L = 20 µm / 10 µm NMOS. Line shows transistor when cantilever is stuck to source. Data points show cantilever unstuck due to applied vibration (1) and pull-in (2).

Figure 1, top left: SEM image of cantilever switch. The area of source-cantilever is much smaller than the gate-cantilever, leading to majority of the cantilever control for the gate electrode.

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Figure 3, above: Drain current of W/L = 20 µm / 10 µm NMOS. Line shows transistor when cantilever is stuck to source. Data points show cantilever unstuck due to applied vibration (1) and pull-in (2).
Fabrication of Thin-Walled and High-Aspect-Ratio Microfluidic Channels

CNF Project # 1176-03
Principal Investigator: Professor R. J. Dwayne Miller

Abstract:
Nanofluidic structures promise to solve the sample preparation problem in various spectroscopy experiments. The thickness of the sample is mostly constrained by the penetration depth of the optical probe. We have designed and successfully fabricated a nanofluidic cell which was used in a 2D-IR spectroscopy experiment to study the nature of the OH bond in water [1,2].

Fabrication:
The building blocks of the cell are two low-stress low-pressure chemical vapor deposition (LPCVD) silicon nitride membranes, created independently on separate wafers using wet etching of silicon. Plasma enhanced chemical vapor deposition (PECVD) of silicon oxide with subsequent photolithography/etching is used to create a buffer layer and the microfluidic channels between the nitride membranes.

In the first generation cell, the membrane thickness was 800 nm, and the cell was filled from the sides through the channels and sealed, trapping the liquid inside. In the latest version, an active control of the sample liquid is realized. Input and output holes are etched into the top wafer and connected to an external pump system. The previously 500 nm thick PECVD silicon oxide buffer layer is now 1500 nm thick in order to achieve a lower flow resistance. Additionally, 70 µm deep wet etched channels are used to connect the ‘thin’ channel area to the input/output holes further reducing the total flow resistance.

The etching of the oxide layer is now done with 6:1 buffered oxide etch (BOE) opposed to previously used reactive ion etching (RIE) providing cleaner and less rough nitride membranes. The BOE etching is highly selective at the nitride/oxide interface leaving almost no residue. A hydrophilic surface is created by high temperature deposition (LPCVD) of ~ 10 nm silicon oxide.

Summary:
Thin-walled fluidic cells with high aspect-ratio containers were fabricated. Active flow of water through 1.5 µm thick channels with 800 nm thick membrane walls is achieved.

References:
Fabrication of Thin-Walled and High-Aspect-Ratio Microfluidic Channels

CNF Project # 1176-03
Principal Investigator: Professor R. J. Dwayne Miller
Users: Alexander Paarmann, Maher Harb

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Web Site: http://lphys.chem.utoronto.ca

- Free standing silicon nitride membranes.
- Bonded substrates with nitride windows enclosing microfluidic channels.
- High-aspect ratio containers.

Figure 1, above: Schematic of nanofluidic device for nonlinear spectroscopy on thin sample liquids.

Figure 2, below left: Nanofluidic cell filled with water. Edge thickness is ~ 1500 nm, center thickness is ~ 400 nm, window size 1 x 1 mm. The interference rings indicate the water layer thickness.

Figure 3, below right: Access hole and wet etched channel. Wet KOH etching is used to create a 70 µm deep channel connecting the access holes to the only 1500 nm thick channels in the window/membrane area.
Ultra-High Density MEMS Based Interconnect for Wafer-Level Ultra-Thin Die Stacking Technology

CNF Project # I260-04
Principal Investigator: Harold D. Ackler, Ph. D.

Abstract:
This work describes a novel Smart Three Axis Compliant (STAC) interconnect targeted to revolutionize chip-to-chip and chip-to-board high-density 3D integration for ultra-thin Si dies (≤ 75 µm) at the wafer level. The STAC interconnect is a three dimensionally compliant interconnect which allows stacked ultra-thin chips to move or flex freely during operation with negligible stress imposed on the die. The work shows that these interconnects can possibly accommodate mismatches of board or package coefficient of thermal expansion (CTE) from chip CTE. STAC interconnects are fabricated using MEMS technologies to support super-fine-pitch (≈ 20 µm pitch) interconnection. These interconnects are batch processed and die containing them can be stacked either at the wafer-level or at the die-level.

Summary:
The constant demand for higher performance and greater functionality from an integrated circuit (IC) package has propelled the semiconductor packaging industry to seek more novel concepts to build and package IC chips. The IC industry has consistently pushed the limits of miniaturization with the advancement in lithographical techniques. Today the IC packaging industry not only has successfully packed more transistors in a square centimeter but also has successfully packaged chips with different functionality into a single module/package. The interconnection of multiple chips in a package has very quickly moved from the x and y-direction to the x, y and z-direction. The later type of chip integration is more commonly known as three-dimensional (3D) packaging or chip stacking technology.

STAC interconnects are MEMS-based electrical interconnects that are microstructurally engineered to accommodate relative displacements between ultra-thin TSV based silicon chip and substrates to which they are bonded without transferring significant stress to the die itself. These interconnects are fabricated from metal films engineered to be released from the substrate when a sacrificial release layer under them is etched away. This is accomplished by using two distinct stresses, compressive and tensile, through the thickness of a single metal film. The alternate stresses in the film will generate a moment causing these interconnect to lift-up/curl. The radius of curvature, $R$ of the interconnect is given by the following equation [1]:

$$ R = Y' h / G $$

where $Y'$ is the biaxial modulus, $h$ is the interconnect film thickness and $G$ is the interconnect film stress range. A more extensive analytical work can be found in the work conducted by Ahmad and Sitaraman [2].

STAC interconnects were formed using a TiW film. The TiW film was sputter deposited in a cryopumped CVC 601 DC magnetron sputter system. The film was deposited by varying the argon partial pressure. The recorded results show the large stress gradient can be achieved with the TiW film. Films can be deposited with stresses ranging from -1GPa to +1Gpa. The STAC interconnect lift-up/curl height can be controlled by varying the stress range built into the TiW film during deposition. The greater the stress range the higher the lift-up/curl. To improve the electrical and thermal properties of the interconnect, a thin layer of Cu is deposited over the TiW film.

Several variations of STAC interconnect designs are being investigated. Two designs are featured in this work. The U-STAC interconnect and a much finer I-STAC interconnect. One end of the strip will connect the bottom bond pad of the top chip and the other end connects the top bond pad of the bottom chip. Total number of I/O’s generated by each design quite easily exceeds the $10^4$ range.

References:
Ultra-High Density MEMS Based Interconnect for Wafer-Level Ultra-Thin Die Stacking Technology

CNF Project # 1260-04
Principal Investigator: Harold D. Ackler, Ph. D.
User: Parthiban Arunasalam

Affiliation: Department of Mechanical Engineering, T. J. Watson School of Engineering and Applied Science, State University of New York at Binghamton

Primary Funding: This material is based upon work supported by Integrated Electronics Engineering Center (IEEC), SUNY at Binghamton

Contact: ackler@binghamton.edu, parthi.arunasalam@binghamton.edu

Figure 1: Successfully fabricated and released U-STAC and I-STAC interconnect. Total interconnect thickness is 1 µm (0.5 µm for each TiW layer). (a) Area array U-STAC interconnects. (b) Closer view of U-STAC interconnect. (c) I-STAC interconnect.
Reducing Idle Power in CMOS Using Cantilever Switches

CNF Project # 1262-04
Principal Investigator: Amit Lal

Abstract:
One of the sources of standby power leakage in MOSFETs is sub-threshold source-drain leakage. Even though the MOSFET is off, a small leakage current flows between its source and drain. We have proposed a solution to eliminate this leakage source by using a cantilever switch. A fabrication process is being developed using aluminum cantilevers over MOSIS dies where the nitride passivation layer acts as the sacrificial layer.

Summary:
The fabrication of the MEMS cantilevers over CMOS is a simple three-mask process that uses the nitride passivation layer as the sacrificial layer for the cantilevers. After a maskless etch thins the nitride layer to the desired cantilever gap, anchors and dimples are defined using two masks. The nitride etches are done using the standard CHF$_3$/O$_2$ process in the CNF. Then aluminum is deposited using e-beam evaporation. The aluminum is patterned for the cantilevers and is etched using wet etchant or, in the future, a dry chlorine-based etch. Finally, the SF$_6$/O$_2$ silicon etch process has been altered to act as an isotropic nitride etch.
Reducing Idle Power in CMOS Using Cantilever Switches

CNF Project # 1262-04
Principal Investigator: Amit Lal
Users: Norimasa Yoshimizu, Shankar Radhakrishnan

Affiliations: Electrical and Computer Engineering; Applied and Engineering Physics; Cornell University
Primary Funding: DARPA
Contact: lal@ece.cornell.edu, ny22@cornell.edu, sr265@cornell.edu

Figure 1, above: Design for reducing idle power consumption. Figure shows design implemented in a CMOS inverter.

Figure 2, top right: SF₆/O₂ undercutting of aluminum cantilevers using nitride sacrificial layer.

Figure 3, bottom right: Nearly-released aluminum cantilevers. The large pad, with etch holes, is pulled down by the gate that lies just beneath it. A slightly misaligned dimple is visible to the left along the beam. Vias down to the source and drain are seen along the sides of the pad.
Prototype Reference Cantilevers for AFM Spring Constant Calibration

CNF Project #1273-04
Principal Investigator & User: Richard S. Gates, PhD

Atomic Force Microscopy Introduction:

Atomic force microscopy (AFM) is widely used today to image surfaces and measure nanoscale forces. To accurately convert the measured AFM cantilever deflection to force, however, requires accurate cantilever spring constant (stiffness) calibration. Large variations in cantilever stiffness can occur even within a processing batch of cantilevers and without calibration, force conversion based on manufacturers nominal specifications can induce unacceptable errors. An accurate AFM test cantilever calibration method is sorely needed.

Reference Cantilever Calibration Method:

There are several methods for calibrating AFM cantilevers but they are usually limited in scope to specific cantilever types or spring constants. The absolute accuracy of the methods is unknown since none of them are currently traceable to the Système International d’Unité (SI). One of the most widely applicable methods (reference cantilever method [1,2]) relies on pushing the unknown cantilever against a cantilever of known stiffness and measuring the deflection. If the stiffness of the unknown cantilever is reasonably close (within a factor of 10) to the stiffness of the reference cantilever, the spring constant of the unknown cantilever can be calculated. Commercial reference cantilevers are available with nominal spring constant values but their accuracy is unknown since they cannot be traced to the SI.

Standard Reference Cantilever Prototype:

The objective of this project is to investigate the feasibility of creating very accurate reference cantilevers that could be used to calibrate the spring constants of AFM cantilevers. This requires very uniform cantilevers that could be calibrated using an SI-traceable technique using statistical sampling. The key to cantilever uniformity lies in careful dimensional control during microfabrication. Since the spring constant \(k\) of an ideal, uniform, rectangular cantilever can be described by an Euler-Bernoulli model [3] (equation 1) that depends on elastic modulus \(E\) and width \(b\) to the first power but the cube of the thickness \(t\) and length \(L\), it is especially important to control these last two characteristics.

\[
k = \frac{Eb^3}{4L^2}
\]

The microfabrication processes included the use of silicon-on-insulator (SOI) wafers in which the device layer thickness is very uniform. Anisotropic back side wet etching was used to define the die on which the reference cantilevers were patterned. E-beam lithography was used to pattern the cantilevers and served two purposes: 1) very high dimensional accuracy when properly calibrated; and 2) careful pattern alignment of the cantilever onto the die by imaging the leading edge of the die through the membrane formed in the back side etching step. Since the cantilever resonant frequency (equation 2) depends on the same key parameters of thickness and length as the spring constant (just to different powers), resonant frequency was used as a measure of uniformity of the cantilevers from different parts of a wafer. The standard deviation of the mean resonant frequency for 22 cantilever arrays was less than 1.0 % suggesting excellent uniformity control. The spring constants of cantilevers made by this process were also measured using a special apparatus designed and fabricated at NIST to provide SI traceable force calibration at the nN scale [4] with a precision of 2 % for a stiffness as low as 26 pN/nm. The results confirm the feasibility of microfabricating SI-traceable reference cantilevers that could be made available to the AFM community.

References:

Prototype Reference Cantilevers for AFM Spring Constant Calibration

CNF Project # I273-04
Principal Investigator & User: Richard S. Gates, PhD

Affiliation: National Institute of Standards and Technology
Primary Funding: National Institute of Standards and Technology
Contact: Richard.gates@nist.gov

- Reference cantilevers of known stiffness can be used to calibrate spring constants of AFM cantilevers.
- Uniform rectangular cantilevers are patterned onto SOI wafers using e-beam lithography.
- Deep reactive ion etching (DRIE) is used to etch the exposed pattern.
- Stiffness of the released cantilevers is validated using an electrostatic force balance, constructed at NIST, capable of nN sensitivity and traceable to the SI.

Figure 1, top left: Schematic side view of cantilever deflections for pushing a test AFM cantilever against a reference cantilever.

Figure 2, bottom left: An initial prototype of reference cantilevers patterned on SOI by e-beam lithography. The cantilevers are 1.4 µm thick and 50 µm wide. The lengths range from 300 µm to 600 µm.

Figure 3, above: Resonant frequency measurement of the prototype cantilevers.
Exploration of Fabrication Techniques for Embedded Strain Sensing Elements

CNF Project # 1348-05
Principal Investigator: Dr. Mohamed R. Mahfouz

Abstract:
We investigated capacitance-based strain sensor arrays and fabrication techniques for the measurement of the load condition of polyethylene spacers in the total knee arthroplasty (TKA) prosthesis. Data from these sensors will be used to give researchers better understanding of component motion, loading, and wear phenomena for a large range of activities. Arrays of capacitive sensors are used to determine the three-dimensional strain within the polyethylene prosthesis component. Arrays of sensors are required to determine the contact area, contact location, and motion between joint replacement components. These sensors implemented on a large scale will give clinicians feedback for individual patient biomechanics without the requirement for patient exposure to x-ray radiation as has been used in the past. Patients will benefit from smart prosthetic components which allow clinicians to monitor biomechanics and loading by applying noninvasive remedies such as orthotics or physical therapy for patients exhibiting poor biomechanics before wear or component failure become issues.

Summary:
In order to match the compliance of the spacer material, ensure biocompatibility, and minimize the modification of existing components; it is desirable to fabricate the multidimensional strain sensor from the same or similar materials as the existing polyethylene component. Prototype electrodes were fabricated at the Cornell NanoScale Facility on four different base materials including silicon dioxide, silicon, parylene, and polyethylene. The primary material used in the bearing between the femoral and tibial components is ultra-high molecular weight polyethylene (UHMWPE). Polyethylene is a low surface energy material, which makes the application of coatings challenging.

A single element of the sensor consists of three unique elements and six total elements. The three different sensing elements are necessary to determine the normal forces, in-plane forces, and shear forces. To fully characterize the strain tensor in three dimensions, six sensors are required to solve for the three normal strains and three shear strains. A parallel plate capacitor is used to determine the strain normal to the plane of the sensors. Two differential translating capacitors indicate the shear components out of the sensor plane, and three interdigitated capacitors function to determine the in plane shear and normal strain.

Two substrates were used for polyethylene fabrication experiments, a 4.0 inch diameter 1.0 mm thick commercial, high-density polyethylene sheet material and a 3.0 inch diameter 4.5 mm thick compression molded medical-grade UHMWPE disk. Obtaining substrates of sufficient flatness and smoothness is a particular challenge. Early attempts to fabricate electrodes on this surface were not successful. A rigorous cleaning and activation process was developed which enabled the successful deposition of electrodes on substrates made of polyethylene. Following the primary cleaning, substrates were exposed to oxygen plasma using an Oxford Plasmalab 80+. An experimental matrix of reactive gases was used to determine the optimum combination of etching gases to both activate and increase surface microroughness. The final mixture was a combination of oxygen and nitrogen for the polyethylene substrates. Following cleaning and surface activation, electrodes were patterned on the surface using the image reversal photolithography technique.

References:
Exploration of Fabrication Techniques for Embedded Strain Sensing Elements

CNF Project # 1348-05
Principal Investigator: Dr. Mohamed R. Mahfouz
User: Boyd M. Evans

Affiliation: Mechanical, Aerospace, and Biomedical Engineering, University of Tennessee
Primary Funding: Internal University Funds
Contact: mmahfouz@cmb.utk.edu, ev3@cmb.utk.edu

Prototype electrodes were fabricated at the Cornell NanoScale Facility on four different base materials including silicon dioxide, silicon, parylene, and polyethylene.

Figure 1: Multiaxial strain sensor with elements for three normal strains and three shear strains.

Figure 2, left: Example of interdigitated capacitive sensors fabricated on polyethylene substrates. Inset is image of interdigitated electrode with 5 µm fingers and 2 µm gaps between legs.

Figure 3, above: 1.2 x 1.2 mm optical profilometer scans of polyethylene substrates. Fig. 3a is of untreated polyethylene surface. Fig. 3b is of oxygen/nitrogen plasma treated surface.
Rotational Oscillators for MEMS Sensors

CNF Project # 1373-05
Principal Investigator: Dr. Cetin Cetinkaya

Introduction:
A rotational oscillator is introduced as a novel approach to the sensing challenges encountered in liquid and gaseous environments. Traditional sensors, such as cantilever beams and membrane-type devices, have a fundamental mode shape out of plane. This motion creates large drag stresses and excessive viscoelastic damping in the device, leading to premature material fatigue and potential device failures. Rotational oscillators would drastically decrease stress and damping in liquids by providing a rotational motion as its second mode shape, and consequently decreasing the stress and damping of the oscillator, while providing accurate chemical sensing comparable to traditional oscillators. The Photo-Acoustics Research (PAR) Laboratory at Clarkson University strives to develop a rotational oscillator for addressing the shortcomings of traditional oscillators in liquid and gaseous environments.

Research Summary:
A class of rotational oscillators have been designed and analyzed for their modal and spectral performances. The dynamics of the device designs are analyzed with the aid of a professional finite element analysis package. The modal properties of rotational mode shapes are determined to define operational bandwidth of the rotational oscillators developed at the PAR Laboratory. Simulation data are examined to ensure good mode separation especially for low order modes and to investigate mass loading sensitivity of the proposed rotational oscillator designs.

In the fabrication of the rotational oscillatory test devices, the PAR Laboratory has been collaborating with the Cornell NanoScale Science & Technology Facility (CNF). The short term objective of the project is to evaluate and test the fabricated devices at the PAR Laboratory using the interferometric and air-coupled excitation characterization facilities. The long term objective is to develop design and fabrication techniques for rotational oscillator sensors for liquid and gas phase sensing applications for homeland security and defense applications.

This project has not been published, however, it formed the basis of the Clarkson University Honors Program undergraduate thesis work for Justin Ricci. Justin is now a M.S. candidate in the Mechanical and Aeronautical Engineering Department at Clarkson University. Jian Chen, Ganesh Subramanian, and Dr. Cetin Cetinkaya have also participated in and supported this work.
Rotational Oscillators for MEMS Sensors

CNF Project #: 1373-05
Principal Investigator: Dr. Cetin Cetinkaya
User: Justin Ricci

Affiliation: Photo-Acoustics Research Laboratory, Clarkson University, Potsdam, NY
Contact: cetin@clarkson.edu

Figure 1: The FEA models provide the first two mode shapes for the rotational oscillator: (a) out-of-plane motion; and (b) in-plane, rotational motion.

Figure 2: SEM images of the rotational oscillator fabricated at CNF. Image (a) shows the overall device, while image (b) shows a close up of the “spoke”. The device was fabricated using an SOI wafer of dimensions 525-2-5 μm.

Figure 3: Frequency modes were found using interferometry equipment. From this, it is clear that the oscillator is free to move. A corresponding mode is found between 350 and 275 kHz.
The Fabrication of Silicon-Based Micromixers for Mixing Enhancement Study

CNF Project # 1376-05
Principal Investigator: Prof. Adeniyi Lawal

Abstract:
In this work, we fabricated a proposed multichannel micromixer for enhanced mixing along with a standard T-junction micromixer for our mixing study. The fabrication of the devices, most especially the multichannel micromixer with its mixing/fluidic features, was realized following a carefully designed approach. For the purpose of ultimately developing an efficient micro-channel mixer for the desired mixing operation, our plan is to validate experimentally our prior numerical data on mixing enhancement study using these fabricated devices.

Summary:
The ineffective mixing in microchannel reactors, primarily due to the inherently diffusion-dominated laminar flow that characterizes such small-volume environment, is one of the current technical issues being addressed by many investigators working in the field of microreaction engineering. Mixing enhancement in microchannel mixers/reactors, most especially for liquid phase reactions, has been recognized as a relevant technical issue critical to the development and application of integrated microchemical processing systems.

The objective of our research study is to investigate mixing enhancement in microchannel mixers, through a theoretical as well as an experimental mixing study of currently utilized as well as proposed micromixing configurations. The proposed micromixers are passive mixers uniquely designed to effect mixing using static or passive mixing structures by creating folding as well as local and global re-orientation of fluid interfaces. The knowledge base necessary for establishing the experimental platform for this mixing study has been created from our prior study using a computational fluid dynamics (CFD) approach [1]. Using the CFD tools and suitable mixing characterization techniques, one of the proposed mixing configurations that we referred to as multilaminated/elongational flow micromixer-4 (MEFM-4) was selected for experimental mixing study along with the standard T-junction micromixer (TjM). The selection of MEFM-4 as the 'best' enhanced-mixing configuration, among the other proposed configurations studied numerically, is based on the set criteria of minimum pressure drop along with high mixing performance.

In this work, the two above-mentioned mixing devices (designated as TjM and MEFM-4) were fabricated from silicon utilizing MEMS microfabrication technology [2]. The fabrication process was achieved using silicon bulk micromachining techniques, which involve two basic steps namely photolithography and deep reactive ion etching (DRIE). With these techniques, silicon wafers (double-side polished, p-type <100>, 4-inch diameter) were structured with fluidic channels of 300 µm deep. Silicon wafers of 500 µm and 800 µm in thickness were used for the fabrication of TjM and MEFM-4 respectively. Thicker wafers were used for fabricating MEFM-4 since deep structures of 300 µm each are required on both the frontside and backside of the wafers.

The three-step lithographic process requirement for the multi-channel MEFM-4, to ensure structural and perfect back-to-frontside-alignment, makes its fabrication, as expected, challenging compared to a two-step lithographic process needed for the single-channel TjM. The DRIE recipe enabled by inductively coupled plasma (ICP) was utilized for the deep etching of the mixing channels with vertical walls as well as the etching of the critical four-through holes in the MEFM-4. These 500 µm etched-through holes would aid the transport of one fluid from the backside inlet manifold to meet and mix with the second fluid coming from the frontside inlet manifold.

The last processing steps required to make the fabricated devices complete for our mixing study are the anodic bonding of the structured silicon substrates with 500 µm thick Pyrex® glass wafers, followed by dicing into individual micromixers of sizes 6.45 cm x 2.54 cm and 3.50 cm x 2.60 cm for TjM and MEFM-4 respectively.

References:
The Fabrication of Silicon-Based Micromixers for Mixing Enhancement Study

CNF Project # I376-05
Principal Investigator: Prof. Adeniyi Lawal
User: John T. Adeosun

Affiliations: 1. Chemical, Biomedical, & Materials Engineering, Stevens Institute of Technology;
             2. New Jersey Center for MicroChemical Systems (NJCMCS)

Primary Funding: ACS PRF and US DOE
Contact: alawal@stevens.edu, jadeosun@stevens.edu

- Silicon MEMS technology was used to successfully fabricate a single-channel T-junction micromixer and a multi-channel micromixer (referred to as MEFM-4).

- Using CFD approach and suitable mixing characterization techniques, MEFM-4 has been shown to exhibit remarkably better mixing performance when compared with the standard T-junction micromixer.

- Future plan is to validate experimentally our numerical study on mixing enhancement using these fabricated devices.

Figure 1, top left: The standard T-junction mixer fabricated from silicon at CNF.

Figure 2, bottom left: The frontside of MEFM-4 fabricated from silicon.

Figure 3, above: The backside of MEFM-4 fabricated from silicon.
Channel-Select RF MEMS Filters  
**CNF Project # 1380-05**  
**Principal Investigator: Sunil Bhave**

**Abstract:**
The vast majority of radio frequency (RF) systems currently in production implement the heterodyne architecture developed by Edwin Armstrong 75 years ago. Today, this architecture relies on discrete components such as quartz crystals and ceramic filters to provide stable references and frequency selection. However, quartz and ceramics are not easily integrated with on-chip circuits, preventing the fabrication of a fully monolithic radio. In recent years, high quality factor (Q) micromechanical resonators have emerged as a possible alternative to quartz and ceramic components. The key benefit of these lateral-mode MEMS resonators is the ability to fabricate multiple frequencies in a single lithography step.

**Summary:**
The OxideMEMS Lab at Cornell University is developing sub-10 µWatt power consumption receivers for sensor network radios operating in the ISM bands. ISM band receivers require narrow channels and are susceptible to nearby strong interferers. To filter out unwanted frequencies, channel-select filter arrays with small bandwidth, good stop-band rejection, and excellent shape factor must be implemented. We have designed and fabricated RF MEMS resonators with high Q (> 7000) and low motional impedance (RX < 60 Ohms) using high-κ dielectric transducers [1]. Channel-select filters are formed by electrically and mechanically coupling these resonators. Electrical coupling is achieved by routing the electrical signal from successive resonators in a ladder configuration. In a typical ladder filter configuration, the parallel frequency fparallel of the shunt resonator is matched to the series frequency fseries of series resonators, defining the filter center frequency ($f_c$). The filter bandwidth is determined by notches on either side of the passband and is twice the pole-zero separation of the series and shunt resonators. The filter array’s frequency characteristics can achieve the best insertion loss reported to date for contour-mode MEMS filters [2,3].

Presently the OxideMEMS Lab is fabricating new resonators with frequencies up to 3.5 GHz and developing a voltage tuning scheme to dynamically tune the filter center frequency and bandwidth for channel agility.

**References:**
Channel-Select RF MEMS Filters

CNF Project # 1380-05
Principal Investigator: Sunil Bhave
Users: Hengky Chandrahali, Dana Weinstein, Chen Chen, Lih Feng Cheow

Affiliation: Electrical and Computer Engineering, Cornell University
Primary Funding: Cornell Center for Nanoscale Systems
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Web Site: http://mems.ece.cornell.edu

Figure 1, top left: SEM of a hafnium dioxide-on-silicon fully released bar resonator.

Figure 2, bottom left: SEM of an array of ladder configuration electrically-coupled thickness shear filters.

Figure 3, above: SEM of 804 MHz thickness shear-mode arc array filter.
Directed Fluidic Self-Assembly of Microscale Tiles

CNF Project # 1396-05
Principal Investigators: Hod Lipson, David Erickson

Abstract:

The aim of this project is to direct the self-assembly of micron-scale units (microtiles) into programmable, reconfigurable structures. Here we detail the fabrication of a first generation of silicon microtiles for directed fluidic self-assembly. We also describe experiments in which single and multiple tiles suspended in various fluids are made to self-assemble on a poly(dimethylsiloxane) (PDMS) substrate using fluidic control.

Summary:

Self-assembly has the potential to provide a scalable alternative to current direct-write or direct-manipulation microfabrication techniques. One of the challenges of self-assembly is the fabrication of arbitrary (non-regular) structures such as those routinely achieved using top-down techniques. Our goal is to direct the self-assembly of microtiles into programmable, reconfigurable structures. We have previously demonstrated stochastic modular self-assembly at the centimeter [1] and millimeter scales. At the micron scale, we direct the self-assembly of silicon microtiles by manipulating local fluidic forces. Here, we describe experiments in which single and multiple tiles are made to self-assemble in a PDMS microchannel.

Fabrication:

Using the L-Edit CAD software, solid silicon microtiles of various sizes (100-500 µm square) with specially-patterned sides for self-alignment were designed. Masks were created using the GCA/MANN 3600F Optical Pattern Generator. The tile patterns were then transferred to SPR positive photoresist spun onto a 30 µm thick silicon-on-insulator (SOI) wafer using the EV 620 Contact Aligner. After developing the resist, the exposed wafer was etched down to the insulator layer using the Unaxis 770 Bosch Etcher. The etched tiles were then released in a 49% HF solution.

Testing chambers of various shapes and sizes were designed separately and again masks were made using the GCA/MANN 3600F. This time the patterns were transferred to 50 µm thick SU-8 negative photoresist spun on to a regular silicon wafer. After development the exposed structures were used to mold PDMS channels (outside the CNF), which were then treated in a plasma cleaner and bonded to glass slides. External tubing was attached to the microchannels for the introduction and removal of assembly fluids. The silicon tiles were introduced into the channels manually through a larger hole in the PDMS.

Experiments:

We demonstrated the ability to attract, bond, and reject a microtile from a patterned substrate by controlling the pressures in multiple 200 µm wide channels connected to the main self-assembly chamber. Both deionized (DI) water and silicone oil proved to be effective media with applied pressures in the range of 1-5 kPa (the more viscous silicone oil required higher pressures than the DI water). We also demonstrated the ability to assemble two microtiles on the substrate by controlling the flow rate in only one outlet. While both assembly media were successful, the advantages of silicone oil were more controllable (slower) dynamics, and more successful self-alignment. In addition silicone oil is non-conductive which will be required in future experiments where power and data will be transmitted across tiles.

References:

Directed Fluidic Self-Assembly of Microscale Tiles

CNF Project # 1396-05
Principal Investigators: Hod Lipson, David Erickson
User: Mike Tolley

Affiliation: Mechanical and Aerospace Engineering, Cornell University
Primary Funding: Cornell University, National Science Foundation
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Web Sites: Lipson Group, www.mae.cornell.edu/ccsl; Erickson Group, www.mae.cornell.edu/erickson

Figure 1, top left: SEM image of 100 µm square silicon tile. After etching down to the insulator layer of an SOI wafer, tiles are released by etching the insulator layer in an HF solution.

Figure 2, bottom left: Two 500 µm square by 30 µm silicon tiles assembled in DI water in a PDMS microchannel. Fluid flow out of microchannel outlets as indicated caused two microtiles to assemble themselves on the patterned PDMS substrate.

Figure 3, above: Flow visualization of a fluid assembly experiment using 3 µm fluorescent silica beads.
Hands-On MEMS

CNF Project # I427-06
Principal Investigator: George E. Georgiou, Ken K. Chin

Abstract:

Hands-on-MEMS is an NSF-funded educational grant. Design and processing of certain MEMS sensors are done by a master’s student. Working sensors are packaged and distributed to universities, for developing a graduate-level MEMS laboratory / design course.

Summary:

The purpose of this project is to develop a simple sensor design and process for educational purposes. Sensor (piezoresistive pressure, electrostatic mirror, capacitive accelerometer and capacitive flow meter) design and processing is part of master’s project.

Packaging is done by a partner university, in this case Columbia University. The packaged samples are then distributed to other universities as part of a MEMS laboratory course under development which ties together design, layout, processing and testing.

Sensors and actuators were designed as part of an “Introduction to MEMS” course in the Physics Department of the New Jersey Institute of Technology (NJIT). Masks were made which combine two sensors with similar processing (mirror + pressure and acceleration + flow). Dimensions were relaxed to allow processing by an inexperienced first year master’s student. Enough insensitivity was also built into the sensor design, to make them robust for a beginning laboratory course.

Enough processing was done after the initial orientation phase at CNF to realize that the combined sensor process integration requires significant process development at CNF, something which is beyond the scope of the introductory nature of the hands-on-MEMS project.

We are now stepping back to simplify processing and to process each sensor individually. Enough process variation is built into processing (for example, timed DRIE of the silicon substrate for the pressure sensing membrane) to allow the laboratory class to also study sensor variability.
Hands-On MEMS

CNF Project # 1427-06
Principal Investigator: George E. Georgiou, Ken K. Chin
Users: George E. Georgiou, Abbas Zaidi

Affiliation: Physics Department, New Jersey Institute of Technology
Primary Funding: NSF
Contact: george.e.georgiou@njit.edu

No pictorial report was submitted for this project
Development of Near Infrared Array Production Method

CNF Project # 509-94
Principal Investigator: Mark D. Morgan

Abstract:
The development of simple, robust, and inexpensive band-pass filter is being pursued. Electron beam lithography and masked ion beam lithography (MIBL) techniques were employed to fabricate optical filters as a critical component of an energy conversion concept utilizing semiconductor photovoltaics (PV). The conversion concept, thermophotovoltaics (TPV), when coupled with MIBL-produced band-pass filters, is capable of converting heat to electrical power with 25% conversion efficiency [1]. We are being supported by contracts sponsored by the US Army and the National Aeronautics and Space Administration.

The goal of the Army program is to demonstrate a diesel fueled TPV power supply via the fabrication of three prototype systems to be tested by Army personnel. The NASA project is a development program aimed at mating a NASA General Purpose Heat Source (GPHS) and a TPV converter to produce a power supply for deep space applications.

The patented EDTEK TPV filter is based on a high-density array of slotted antenna elements etched into a thin gold film. Typically, the cross-shaped mesh elements have line-widths of 80 nm and line lengths of 450 nm. A resonance condition occurs if the dimensions of the elements are comparable to the wavelength of the interacting electromagnetic field. Resonance wavelength, peak transmission and band-pass shape are adjusted by varying element dimensions and geometry.

Summary:
Our work over the past year has continued the prior years’ effort by producing silicon membrane stencil masks used to transfer patterns by MIBL. Fabrication of the stencil masks consisted of patterning silicon membranes (~ 0.65 µm thick) and silicon nitride membranes (~0.4 µm thick) using the CNF VB6 e-beam lithography tool. The work last year was centered on producing silicon masks and silicon nitride masks for use in both our MIBL and aperture array lithography (AAL) system [2,3]. EDTEK and the University of Houston are developing AAL for future mask production aimed at specific applications.

The fabrication of daughter stencil masks allows the printing of one square centimeter of nano-scaled features on a single substrate in less than 20 second exposure time. Using a 4 mm²-printed-area silicon stencil mask, patterned at CNF, we employed MIBL to print a larger nano-scale area onto another silicon or silicon nitride membrane thereby producing a 2nd generation mask used to print areas of 100 cm² per substrate. The substrates were processed to produce IR band-pass mesh filters for application in the TPV power generator. To date, our group has fabricated 1st generation stencil masks at CNF of 2 mm x 2 mm. From these master masks, 2nd generation masks have been fabricated with dimensions 10 mm x 16 mm. The EDTEK MIBL system has employed daughter masks and master masks to print a total of 30,000 cm² of nano-scaled features.

References:
Development of Near Infrared Array Production Method

CNF Project # 509-94
Principal Investigator: Mark D. Morgan
Users: Mark D. Morgan, Vasan Sundaram

Affiliation: EDTEK, Inc.
Primary Funding: U.S. Army, NASA
Contact: m.morgan@edtekinc.com, v.sundaram@edtekinc.com

- Prototype electric generator design requires an IR band-pass filter with 1800 cm² of nano-scaled features.
- The IR band-pass filter was fabricated using masked ion beam lithography (MIBL) - 80 nm linewidth.
- MIBL allowed exposure of entire required area (4.4 x 10¹¹ elements) in 20 hours.

Figure 1: Photomicrograph of 2nd generation silicon nitride stencil mask fabricated using masked ion beam lithography.
Waveguide Fabrication and Optimization in Potassium Titanyl Phosphate

CNF Project # 764-99
Principal Investigator: Gregg Switzer

Abstract:
AdvR’s work involves the fabrication and characterization of optical waveguides in potassium titanyl phosphate (KTP) coupled to semiconductor lasers. These waveguides allow second harmonic generation (SHG) to frequency double the output of a narrow bandwidth semiconductor laser. The accuracy of the final waveguide is influenced by each step of the photolithographic process. The focus during 2005-2006 has been to explore the viability of depositing silicon dioxide as a mask for the pattern to allow in-process interrogation and optimization of the optical transmission of the waveguides. Future efforts will include refining the process of silicon dioxide depositions as well as an exploration of using other non-metallic dielectrics.

Summary:
Periodic waveguides embedded in KTP wafers provide a novel approach to high efficiency QPM of laser light. A mask containing the periodic pattern is transferred onto the surface of the KTP wafer using 5x photolithography at CNF. Although the single layer pattern transfer seems straightforward, wafer-to-wafer variations in both property and geometry require non-standard deviations from the traditional photolithography process. Once the wafer is patterned, the wafers return to AdvR for dicing, polishing and dipping onto a hot bath of rubidium nitrate salts. The rubidium ions that pass through the holes in the pattern exchange with potassium ions within the wafer causing a slight increase in the index of refraction of that region. This gives rise to total internal refraction of light as it travels down the pattern-defined optical waveguide. If a segmented waveguide is patterned with an appropriate pitch, type 1 quasi-phase matching (QPM) is possible through domain reversal in the exchanged regions allowing for highly efficient frequency doubling of laser light. The systematic errors introduced throughout the process from reticle manufacture to final lithography on to the KTP wafer critically impact the desired properties of the waveguide.

AdvR has recently designed segmented waveguides for type 1 QPM for frequency doubling 1060 nm light to 530 nm. Patterns with several distinct periods, duty cycles, and widths of waveguides were designed to compensate for variations in modal confinement (and thus the modal index) of the waveguide that can arise from substrate differences and exchange depth. Wafers have patterned with aluminum in the past, but this time it was decided to pattern the wafers with SiO₂ instead.

The ability to use SiO₂ instead of our traditional aluminum coating is advantageous because it allows us to characterize the properties of the waveguide without removal of the transparent pattern. The chip can therefore undergo repeated ion-exchanges in order to tune its optical behavior, thereby greatly improving yields. SiO₂ also acts as an electrical insulator and optical buffer between the waveguides and micro-electrodes used for electro-optic (EO) control of the index of the waveguide. EO control enables active control of the waveguide properties, opening the door for new innovative laser-based applications.

In July 2005, AdvR utilized the CVC 4500 Evaporator to deposit ~ 700Å silicon dioxide (SiO₂) on KTP wafers at CNF for the first time. Our first attempt at SiO₂ deposition yielded only moderate resolution of the elements due to chipping that occurred during the lift off process. Although the waveguides were not ideal, some did produce successful frequency double output at 530 nm from a 1060 nm source and could be characterized with the SiO₂ pattern still in place as proof of concept. Future trips to CNF are expected in order to improve the SiO₂ deposition and lift off process.

AdvR greatly appreciates the extra effort the CNF staff made in response to our specific needs.
Waveguide Fabrication and Optimization in Potassium Titanyl Phosphate

CNF Project # 764-99
Principal Investigator: Gregg Switzer
Users: Philip Battle, Tim Fry, Gregg Switzer, Will Suckow
Primary Funding: SBIR
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Figure 1: Contrast of a well defined SiO₂ patterned waveguide vs. a poorly defined pattern. A majority of the waveguides from this initial test resulted in poor quality, prompting a future return to CNF to improve the photolithography process.

Figure 2: Frequency tunable 1064 nm input was coupled into a newly patterned QPM waveguide and frequency doubled for scanning an iodine absorption line at 532 nm.
Tunnel Coupled Quantum Well-Quantum Dots Active Medium for High-Frequency Semiconductor Lasers

CNF Project # 780-99
Principal Investigator: Serge Oktyabrsky

Abstract:
The project is focused primarily on materials, technologies and components for the III-V optoelectronic devices integrated with a silicon platform. The goal is to provide design and technology for light emitters and photodetectors with high thermal stability and high bandwidth suitable for hybrid microintegration on Si electronics into massively parallel arrays [1]. The major focus is on the microcavity optoelectronic devices, such as vertical cavity surface-emitting lasers (VCSELs), microcavity light-emitting diodes, and resonant cavity photodetectors, which are anticipated to play the major role in the future chip-level optical interconnect technology. In 2005/06, structures of tunnel coupled pairs consisting of InGaAs quantum wells grown on top of self-assembled InAs quantum dots (QW-on-QDs) were employed to improve the gain medium in thermally stable semiconductor QD laser diodes. We have developed a tunnel QW-on-QDs structure with a QD resonance transition which is red-shifted ~ 35 meV relative to the QW ground state (GS). Edge-emitting lasers utilizing this active medium were developed and characterized. All-epitaxial vertical cavity surface emitting lasers with triple-pair tunnel QW-on-QDs medium demonstrated continuous wave mode lasing with 5.7 mA minimum threshold current at QD ground state emission wavelength, 1131 nm [2].

Summary:
QD-on-QW active medium was developed and characterized in edge-emitted lasers. Our approach is to use multiple pairs of tunnel coupled QWs grown on QDs structures (QW-on-QDs) as opposed to multiple QD layers for a single QW [2]. Optimized energy separation between ground energy states of QW and QDs within a pair was found to be ~ 35 meV, which is close to the energy of LO phonon. This transition with narrow linewidth, 21.6 meV at T = 77K, indicates an efficient LO-phonon assisted tunneling of carriers from QW into QD ensemble states. The highest gain ( > 50 cm⁻¹ in waveguide lasers) was achieved with a QW-on-QDs active medium with GS relative separation of c 35-40 meV. VCSELs with 3 x (QW-on-QDs) active medium were designed, grown, and processed. The design was based on AlGaAs/GaAs all-epitaxial distributed Bragg reflectors (22 and 34 pairs in the top and bottom reflectors, respectively) intracavity p-contact, and single selectively oxidized current aperture. The devices demonstrated relatively high operating voltage ( > 5V), likely because of high resistance of intracavity contact. Nevertheless, CW lasing mode of first QD-based VCSEL with tunnel-coupled medium was demonstrated with oxide aperture sizes from 5 to 17 µm at room temperature. Best measured differential efficiency of ~ 6.2% was observed in small-aperture lasers [3].

References:
Tunnel Coupled Quantum Well-Quantum Dots Active Medium for High-Frequency Semiconductor Lasers

CNF Project # 780-99
Principal Investigator: Serge Oktyabrsky
Users: Jobert van Eisden, Rama Kambhampati

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• Active medium consisting of InGaAs Quantum Wells grown on top of self-assembled InAs Quantum Dots (QW-on-QD) coupled by tunneling was developed.

• Damping-limited bandwidth of QW-on-QD active medium is estimated at 30 Ghz.

• VCSELs with tunnel-coupled QW-on-QD were fabricated.

• Threshold current density of ~ 4kA/cm² at 1130 nm was measured.

Figure 1, top left: Schematic band diagram of tunnel coupled Quantum Well-Quantum Dot medium.

Figure 2, bottom left: TEM (200) dark field cross-section of QD-on-QW medium.

Figure 3, above: Light-current characteristic of all-epitaxial tunnel coupled QD-on-QW VCSEL.
Preparation of Silicon Based Photonic Materials

CNF Project # 810-99
Principal Investigator: Philippe Fauchet

Abstract:
By forming a lattice of macroscopic dielectric media, an optical analogy of a crystal can be fabricated, called a photonic crystal. This investigation seeks to use silicon fabrication techniques to construct photonic bandgap structures with the capability of manipulating light for potential all-optical silicon based optoelectronic circuits. The optical properties of silicon photonic structures are also used to build biosensors. To achieve these goals, various thin-film deposition, etching, annealing, and photolithographic processes were performed at CNF. Equipment used in this project includes the MOS dry oxide and nitride LPCVD furnace, the Applied Materials RIEX etcher, JEOL 9300 e-beam writer, Plasmatherm 770 chlorine silicon etcher, HTG System III-HR Contact Aligner, Zeiss SEMs, Oxford PlasmaLab 80+ RIE, GaSonics Aura 1000 Asher, Heidelberg DWL 66 Laser Pattern Generator, PlasmaTherm 720 aluminum etcher, GSI PECVD nitride deposition, CAD tools, and the GCA PG3600F optical pattern generator.

Summary:
In the first approach we define a variety of photonic crystal resonators/waveguides on SOI wafers by e-beam lithography and fabricated by RIE. The structures are integrated with tapered ridge waveguides for light coupling. All coupling facets are polished after fabrication. To enable for electric field tuning, electrodes are integrated with photonic crystal resonators, and liquid crystals are infiltrated throughout the photonic structures [1-4]. In Figure 1, a 1-D photonic crystal cavity, with a “defect” lattice in the matrix and a photonic crystal waveguide, is integrated with a vertical contact. We have also fabricated resonators of other geometries, like ring resonator, [5,6] and the preliminary measurement shows their discrete resonance peaks with quality factors as high as 30,000.

Fabricated photonic crystals can also be used in biosensing applications. Their optical properties are highly sensitive to environmental variations (e.g. ambient refractive index changing, biological molecules binding etc.). Photonic crystals therefore are an attractive platform for label-free biosensing applications. Binding of the target biological molecules is monitored by observing a red shift of the transmission resonance.

References:
Preparation of Silicon Based Photonic Materials

CNF Project # 810-99
Principal Investigator: Philippe Fauchet
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Figure 1, top left: 1-D photonic crystal cavity, with a “defect” lattice in the matrix and a photonic crystal waveguide, with an integrated vertical contact.

Figure 2, bottom left: 2-D PBG microcavity integrated on SOI wafer for biosensing.

Figure 3, above: Normalized transmission spectra of the photonic crystal microcavity. Curve (a) indicates the initial status, curve (b) is measured after glutaraldehyde is applied and curve (c) is obtained by infiltrating BSA (Bovine Serum Albumin) molecules.
Etched Facet Technology for Blue-Violet Lasers

CNF Project # 924-01
Principal Investigator: Alex Behfar

Abstract:
A blue-violet (405 nm) emitting laser was fabricated using a new etched facet technology for GaN-based material. The technology can allow the fabrication of short-cavity lasers with higher yield than possible in cleaved facet lasers.

Summary:
Nichia Chemical first demonstrated GaN-based blue lasers on sapphire substrates in 1995 and has subsequently been able to produce commercially available CW lasers [1]. Nichia uses cleaving to form the facets of its blue lasers, but prices of such lasers have remained very high. There is tremendous interest in fabricating inexpensive 405 nm-emitting GaN based lasers for the next generation of DVD applications.

Cleaving to form mirror facets is the standard process for making edge-emitting lasers. An alternative to cleaving is to etch the laser facets. Etched facets with high quality were formed in GaAs through a process based on chemically assisted ion beam etching (CAIBE) at Cornell University [2]. These laser devices are characterized by precisely located mirror facets with quality and reflectivity equivalent to those obtained by cleaving.

The EFT allows lasers to be fabricated on the wafer in much the same way that integrated circuit chips are fabricated on silicon. Etched facet lasers are monolithically integratable with other photonic devices on a single chip [3] and can be tested inexpensively at wafer-level [4]. Facet reflectivity modification (FRM) can be used to modify the reflectivity of the etched facets through deposition of dielectric coatings with the wafer intact.

Early on in the development of GaN based blue lasers, CAIBE was applied to the fabrication-etched facets for blue lasers on sapphire [5], however, the reflectivity obtained from such etched facets were smaller than cleaved facets. The lower reflectivity was blamed primarily on roughness of the facet [6] as well as the deviation of the facet from vertical [7].

A key to obtaining high quality etched facets is high selectivity between the etch mask and the semiconductor material in the etching process. The etch facets formed in GaN were formed with a selectivity of better than 10:1 and the etch rate was higher than 0.25 µm/min.

Formation of a cavity in the GaN system through cleaving does not easily allow a cavity length below 500 µm. Since the material system has very high defect density (presently around 1E5 defects/cm^2 for the best available material), assuming a ridge width of 2 µm, a cavity of such length will on average contain at least 1 defect. As such, the GaN system provides additional incentive for using etched facets since much shorter cavities can be formed with relative ease. Assuming a cavity length of 50 µm with the same ridge width of 2 µm, the probability of a cavity with a defect is a factor of 10 lower. This leads to significantly increased yield.

Acknowledgements:
Alfred Schremer, Malcolm Green, Alan Morrow and the rest of the BinOptics technical team contributed to this work.

References:
Etched Facet Technology for Blue-Violet Lasers

CNF Project # 924-01
Principal Investigator: Alex Behfar
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- Low-cost blue-violet emitting lasers for next generation DVD players.
- High defect density in GaN material severely impacts laser diode yield.
- Improved yield with short cavity etch facet lasers.

Figure 1: SEM image of an etched GaN facet.

Figure 2: Spectral characteristics of a blue-violet laser.
Sub-Wavelength Confinement in Integrated Metal Slot Waveguide on Silicon

CNF Project # 980-01
Principal Investigator: Michal Lipson

Abstract:
There is a growing research interest in optical circuits at the nanometer scale for future integration of optical, optoelectronic and electronic devices on-chip. For this goal, however, the typical dimensions of conventional dielectric waveguides are dictated by diffraction, therefore limiting dense on-chip integration. Plasmonic waveguides such as nanoparticle chains [1], nanorods [2], in contrast, guide light through the interaction of photon and electron oscillation around the metal surface, and are potential candidates for nanoscale optical elements with sizes much smaller than the diffraction limit. The tradeoff between the confinement level and propagation loss, however, is a fundamental limitation of such waveguides. Therefore, structures offering both high confinement and relatively low loss are desired. To overcome the traditional limitations of plasmonic waveguides, we use the inverted metal slot waveguide with a dielectric core sandwiched between metal cladding [3]. As is general for surface plasmon, the electric field polarized perpendicular to the metal-dielectric interface is bound around the interfaces due to the high dielectric discontinuity between metal and dielectric. For narrow slot, the plasmonic waves around the two interfaces interact and result in a symmetrical mode where the light is almost completely confined in the dielectric slot, enabling extreme sub-wavelength confinement across the slot far beyond the diffraction limit. The effective index of the slot increases due to the coupled plasmonic waves. Therefore, for three-dimensional realistic structures the light is vertically confined via the index confinement mechanism. Using the metal slot with silicon, we show experimentally a low loss plasmonic waveguide on silicon substrate with very high confinement, and we also show high coupling efficiency between these waveguides and dielectric silicon waveguides using very compact tapers.

Summary:
We fabricate the metal slot waveguide on an SOI wafer. The pattern is defined using electron beam lithography and etched by RIE. Then a photolithography step is used to define the window for metal evaporation and a bilayer resist structure is used for successful liftoff of 300 nm Au. Experimentally we show a low loss plasmonic waveguide on silicon with high confinement. The measured loss is about one order of magnitude lower than previously demonstrated structures with a similar level of lateral confinement. We also show for the first time experimentally high coupling efficiency between these waveguides and dielectric silicon waveguides using very compact tapers. The realization of deep sub-wavelength confinement and efficient coupling with standard dielectric silicon waveguides has attractive applications in nanoscale circuits and on-chip integration of optical, optoelectronic and electronic devices.

References:
Sub-Wavelength Confinement in Integrated Metal Slot Waveguide on Silicon

CNF Project # 980-01
Principal Investigator: Michal Lipson
User: Long Chen
Affiliation: Electrical and Computer Engineering, Cornell University
Primary Funding: Air Force Office of Scientific Research (AFOSR)
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Figure 1, top left: Schematics of (a) cross section of the metal slot with silicon (b) integration of the metal slot waveguide with silicon wire waveguides.

Figure 2, bottom left: Microscope image of the metal slot integrated with a dielectric silicon wire waveguides.

Figure 3, above: Propagation loss of the metal slot waveguide with different slot width.

Figure 1

Figure 2

Figure 3
Evolutionary Photonics

CNF Project # 980-01
Principal Investigator: Michal Lipson

Abstract:

We simulate an evolutionary process in the lab for designing a novel high confinement photonic structure, starting with a set of completely random patterns, with no insight on the initial geometrical pattern. We show a spontaneous emergence of periodical patterns as well as previously unseen high confinement sub-wavelength bowtie regions. The evolved structure has a Q of 300 and an ultra small modal volume of $0.112(\lambda/2n)^3$. The emergence of the periodic patterns in the structure indicates that periodicity is a principal condition for effective control of the distribution of light.

Summary:

We demonstrate and fabricate the design of a sub-diffraction limit mode volume optical resonator. The device is in a single layer slab, with a center bow tie cavity and surrounded by distributed Bragg layers. The bow tie creates a small modal volume cavity. The Bragg layers act to confine light in the bowtie cavity and increase Q of the structure.

The device geometry was generated with an evolutionary algorithm from random. There was no initial seeding for the device geometry. The merit function for device design was the field intensity in the structure center. Repeated runs of the algorithm generated the same basic geometry. Structure was limited to 250 nm thick silicon substrate, 4 by 5 µm, and surrounded by either oxide or air lower index material.

The device was analyzed and found to have sub-diffraction limited mode volume of $0.112(\lambda/2n)^3$. Excitation was provided by 1.5 µm wavelength continuous wave. Device simulation was run until a steady state was achieved. The unusual shape of the resonator has not been previously reported in the literature and opens a potential for future optical structure design. The small mode volume could be used to offset low Q values and increase bandwidth of resonators which need high bandwidth and high nonlinear effects.

The algorithm demonstrates a completely different approach for ground up structure design. It could be used to explore photonic devices with unusual properties, which are difficult or impossible to design efficiently “by hand”.

The CNF nanocluster was used for evolutionary simulations for device design. Devices were fabricated with JEOL e-beam lithography on SOI wafers.

References:

Evolutionary Photonics

CNF Project # 980-01
Principal Investigator: Michal Lipson
Users: Alexander Gondarenko, Stefan Preble, Jacob Robinson, Long Chen

Affiliations: Electrical & Computer Engineering; Applied & Engineering Physics; Cornell University
Primary Funding: NSF
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- An evolutionary algorithm on a distributed computing platform at CNF nanocluster was used to design a new class of optical resonators.
- The new resonators show a sub diffraction limit optical mode volume $0.112(\lambda/2n)^3$.
- We attempted to design structures by hand, similar to the ones produced by the algorithm, but in simulations, our devices performed poorer.
- We fabricated hand and computer evolved devices to show fabrication feasibility.
- The devices shows a narrow central slot (10s of nanometers), surrounded by distributed Bragg layers.

Figure 1: Device “hand designed” from results of evolutionary algorithm. 1.5 µm excitation field. Designed for sub diffraction optical mode volume.

Figure 2: Device designed by evolutionary algorithm, theoretically out-performs hand designed device. 1.5 µm excitation field. Designed for sub diffraction optical mode volume.
Ultra-High Resolution Imaging of Highly Confined Optical Modes in Sub-Micron Scale SOI Waveguides

CNF Project # 980-01
Principal Investigator: Michal Lipson

Abstract:
Highly confined optical modes, made possible by the large index contrast in silicon on insulator (SOI) or semiconductor air-bridge platforms, has allowed for the development of a variety of compact and efficient photonic devices. Recent SOI photonic devices include optical switches and modulators in microring resonators [1] and photonic crystal cavities [2]. Optical buffers based on the large group index in photonic crystals [3] and electromagnetically induced transparency (EIT) [4] have been shown experimentally. Raman gain [5] and lasing [6] as well as efficient wavelength conversion [7] have also been recently demonstrated. The efficiency of these processes relies on the high concentration of optical intensity in the submicron-sized silicon core.

With this recent success of highly integrated sub-micron-scale photonic structures, comes the need for improved measurement and characterization techniques to better understand and design future devices. Of particular interest is the measurement of local near field properties of these devices. Here the highly confined nature of these devices makes local field measurements particularly challenging. Because light is confined to non-radiating guided modes and mode features are often smaller than the free space diffraction limit, conventional far field microscopy can not resolve the local characteristics of guided modes. Therefore much interest has been shown in the use of near field scanning optical microscopy (NSOM) to observe the local nature of guided modes in photonic devices. NSOM measures the optical near field by scanning a sub-wavelength-sized probe in the vicinity of an evanescent field near the sample’s surface.

We develop and implement a new type of NSOM: Transmission NSOM (TraNSOM) which has higher resolution than aperture NSOM techniques [8] and higher collection efficiency than existing apertureless techniques [9]. We implement this new technique on a commercial AFM by measuring small changes in transmission as the waveguide is scanned by a metallic AFM probe. With this technique we measure optical decay lengths as small as 85 nm in highly confined SOI waveguides.

Summary:
We perform ultra-high resolution imaging of the optical near field of sub-micron-scale silicon on insulator (SOI) waveguides using a transmission near field scanning optical microscopy (TraNSOM) technique we developed and implemented on a commercial AFM. We report resolution 18 times smaller than the free space wavelength ( ~ 85 nm) limited only by the size of the AFM probe. We implement this technique by measuring small changes in transmission through the waveguide as we scan it with PtIr coated AFM probe. The change in transmission at each point is related to the local intensity of the optical near field. We fabricate these waveguides to operate at a wavelength near 1.55 µm using electron beam lithography and ICP etching. The Si waveguides are approximately 500 nm wide and 250 nm tall on a 3 µm buried oxide. The waveguides are covered with about 160 nm of thermal oxide which we use as a hard mask for etching. Measured optical mode profiles agree well with theory.

References:
Ultra-High Resolution Imaging of Highly Confined Optical Modes in Sub-Micron Scale SOI Waveguides

CNF Project # 980-01
Principal Investigator: Michal Lipson
User: Jacob Robinson

Affiliation: Electrical and Computer Engineering, Cornell University
Primary Funding: Air Force Office of Scientific Research (AFOSR)
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- Optical mode imaged by measuring changes in optical transmission as a sub-micron scale SOI waveguide is scanned by a metallic AFM probe.

Figure 1, top left: Topographical AFM image of silicon on insulator (SOI) waveguide.

Figure 2, bottom left: Simultaneously recorded TraNSOM image of the optical mode (free space wavelength of 1.532 µm) confined to the waveguide shown in Figure 1. Optical standing wave pattern is a result of reflections at the input and output interfaces.

Figure 3, above: Solid line: Cross section of the mode profile measured along the line from point P to point Q in Figure 2. Dotted markers: Mode profile simulated with finite element mode solver. Dashed line: Simultaneously recorded topography measured from point P to Q.
Tunable Optical Delay with On-Chip Analogue to EIT

CNF Project # 980-01
Principal Investigator: Michal Lipson

Abstract:
Recent theoretical analysis of coupled micro resonators has revealed that coherence effects in coupled resonator systems are remarkably similar to those in atoms. Similar to the atomic systems, where electromagnetically induced transparency (EIT) occurs due to quantum interference effects induced by coherently driving the atom with an external laser [1], induced transparency can also occur in a photonic resonator system where coherent interference between two coupled resonators is instead enforced by the geometry of a nanophotonic structure. In particular, for particular configurations of waveguides side-coupled to resonators, there exists an all-optical dark state that can be asymptotically decoupled from the waveguide for proper tuning of the resonator frequencies. The existence of such an all-optical dark state, which gives rise to an EIT-like transmission spectrum, is critical for on-chip coherent manipulation of light at room temperatures, including the capabilities of stopping, storing and time-reversing of an incident pulse [2]. We provide the first experimental observation of structural tuning of the EIT-like spectrum in integrated silicon optical resonator systems. Our results demonstrate that the resonant interference required for coherent manipulation of light can indeed be achieved on-chip without the use of atomic resonance. Consequently, many of the basic limitations on bandwidth and decoherence that result from the fragility of the electronic coherence may be fundamentally overcome.

Summary:
The device is fabricated on SOI substrate using e-beam lithography and plasma dry etching. The device consists of two serially coupled ring resonators. The diameters of the two rings are 10 µm. The center-to-center distance between the two rings is 15.69 µm. Both the waveguide coupled to the rings and the one forming the rings have a width of 450 nm and a height of 250 nm. A slight difference in perimeter between the two rings (8 nm) is introduced to detune the ring resonances.

The transmission spectrum for the quasi-TM mode shows two dips at from the low-Q resonances of the two ring resonators (Q = 770). Due to the coherent coupling between these low-Q resonances, a narrow transmission peak appears with a quality factor Q = 11,900. Like the transparency peak in EIT spectrum, this high-Q transmission peak corresponds to a large group delay of 17.9 ps which corresponds to an effective group index of 207, considering the physical length of the device. Both the transmission spectrum and the group delay spectrum can fit very well with a theoretic model.

This delay is determined by the detuning between the two ring resonances. If the cavity is lossless, the delay will approach infinite when the detuning approaches zero. In the fabricated device, the maximal delay is limited by the scattering loss in the ring resonators. In the experiment, the detuning is controlled by tuning the resonance of each resonator thermally, and peak group delays between 7.8 ps to 25.0 ps are measured, corresponding to variable group index from 90 to 290. Higher group delay results in higher scattering loss, and therefore lower peak transmission.

References:
Tunable Optical Delay with On-Chip Analogue to EIT

CNF Project # 980-01
Principal Investigator: Michal Lipson
Users: Qianfan Xu, Jagat Shakya

Affiliation: Electrical and Computer Engineering, Cornell University

Primary Funding: Semiconductor Research Corporation;
National Science Foundation’s CAREER award

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Figure 1: Top view microscopic picture of the serially coupled silicon micro-ring resonators as an on-chip all-optical analogue to the electromagnetically induced transparency. Arrows show the direction of light in the cavity.

Figure 2: Measured optical delay in the device (squares) at different wavelengths, which matches well with theory (solid line). A peak delay of 17.9 ps observed at the center of the EIT-like transmission peak (dashed line, right y-axis).
Terahertz & Infrared Photonic Crystal Structure

CNF Project # 1183-03
Principal Investigator: Yujie J. Ding

Abstract:
Photonic crystal structure is a promising candidate for manipulating light. Previous works are most focused on infrared photonic crystal. For the Terahertz (1 Thz = 10^{12} Hz) far infrared range, which is of great interest to people for its application in bio-sensing, imaging etc., only a few results has been obtained [1]. In this project, we are working on designing and fabricating 1 D (Bragg reflector) and 2 D photonic crystal structures for THz frequency range. Apart from THz photonic crystal structures, we are also interested in fabricating 2 D photonic crystal slab structures for 1.55 µm infrared range. This structure is designed to demonstrate a low loss sharp bending waveguide to be applied for optical interconnect on single Si chip.

Summary:
In the previous year, we worked on optimizing both the design and fabrication of our photonic crystal structures. For our THz 2 D photonic crystal structures, we selected high resistivity Si wafer with a thickness of around 300 µm, which is thinner than the wafer we used before. We also selected new patterns for the 2 D photonic crystal. Besides etching circular air holes to form either the square lattice or hexagonal lattice which we tried before, we tried some new structures with square shaped air holes arrayed in square lattice and hexagonal shaped air holes arrayed in hexagonal lattice. We also optimized the parameter of the hole diameter and the lattice constant to achieve a broader photonic bandgap. The fabrication process of our THz photonic crystal remains almost the same; the major steps are thermal oxidation to grow SiO_2 mask layer, HTG photo lithography, PT72 SiO_2 etching, and Unaxis 770 Si through wafer deep etching.

For the 1.55 µm infrared photonic crystal slab waveguide, we selected silicon on insulator (SOI) wafers with a thicker SiO_2 insulating layer. This will help to reduce the leakage loss of the SOI slab waveguide. In fabrication, we used PECVD to deposit the SiO_2 mask layer. Unlike the thermal oxidation method, deposition will not change the top Si device layer thickness. Since the feature size of this structure is sub-micron (around 500 nm), electron-beam lithography was selected to write the patterns on the wafer. For SiO_2 mask etching, we used the PT72 and for top Si layer etching the PT770 is selected.

From our experimental characterization, we have identified the photonic bandgap for both of our 2 D THz photonic crystal and infrared photonic crystal structures. Further measurement and optimization of structures are still expected.

Reference:
Terahertz & Infrared Photonic Crystal Structure

CNF Project # 1183-03
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Users: Hongqian Sun, Wei Shi

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Near IR Quantum Dot Lasers for Ultrashort Pulse Generation

CNF Project # 1190-04
Principal Investigator: Prof. Farhan Rana

Abstract:
In recent years quantum dot (QD) lasers have become an active field of research. They offer a number of advantages over their quantum well laser counterparts, exhibiting lower threshold currents and improved temperature stability. Furthermore, a gain medium which comprises of self assembled quantum dots grown by MBE exhibits a broader gain spectrum along with a lower line-width enhancement factor compared to a quantum well based gain medium. These two factors make the prospects for producing pulses with widths approaching 100 fs via mode-locking quite lucrative.

Summary:
Our group is actively investigating QD lasers for generating ultra-short pulses via mode-locking. Recently, passively mode-locked quantum dot lasers have successfully produced pulses with pulse widths approaching 400 fs and peak powers of 500 mW [1,2].

A monolithic passively mode-locked semiconductor laser is comprised of a ridge waveguide etched into the semiconductor. On this ridge, two sections are defined of which the longer one is forward biased and acts as the gain section. Electrically isolated from the gain section is the saturable absorber section whose length is usually kept much shorter than the gain section. This segment is reverse biased, and for a suitable reverse bias voltage and forward bias current, the laser begins to emit a train of pulses at a repetition rate which depends on the length of the ridge waveguide. Feedback in the laser is provided by the cleaved facets which can be coated with various dielectric films to increase or decrease reflectivity.

One of the most promising samples consisted of three stacks of self-assembled InAs QDs sandwiched between AlGaAs cladding layers grown on a GaAs substrate. Waveguides with widths ranging from 2-5 µm were patterned using standard lithography techniques. Etching of the waveguides was performed on the newly installed III-V PlasmaTherm-770 ICP etcher. A combination of BCl₃/Ar with the right RF parameters was able to provide a smooth, vertical waveguide profile. The waveguides were then planarized using a thermally curable polyimide before the top metal contact was evaporated. The samples were then thinned to 110 µm and after evaporating the back contact, the laser bars were cleaved with lengths varying between 250 µm and 3 mm. Unfortunately only laser bars whose lengths were below 1 mm demonstrated lasing at wavelengths just below 1200 nm. Several tens of mW of power was collected from one side of the device. The shorter waveguide lasers exhibited threshold currents around 100 mA. So far we have been unable to confirm mode-locking in these devices. Their short lengths would lead to repetition rates exceeding 50 GHz and would be a step forward towards high repetition rate devices. Facet coating by evaporating dielectric layers resulted in the reduction of the threshold currents as well as more output power.

References:
Near IR Quantum Dot Lasers for Ultrashort Pulse Generation

CNF Project # 1190-04
Principal Investigator: Prof. Farhan Rana
User: Faisal R. Ahmad
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Primary Funding: NSF
Contact: fr37@cornell.edu, fra3@cornell.edu
Web Site: http://people.ece.cornell.edu/rana/

Figure 1, top left: A schematic representation of a modelocked semiconductor laser. The waveguide is surrounded by low index polyimide. The gain and the saturable absorber section are electrically isolated. Light is extracted from one of the facets.

Figure 2, bottom left: A SEM showing the cleaved facet of a fabricated laser diode.

Figure 3, above: Coating one of the facets with a high reflector leads to an increase in the output power as well as reduction in the threshold current.

Figure 3
High Sensitivity Uncooled Microcantilever Infrared Imaging Arrays

CNF Project # l202-04
Principal Investigator: Gregory Simelgor

Abstract:
Multispectral Imaging is developing an infrared imaging detector based on an advanced bimorph microcantilever design. The bimorph design utilizes a combination of metallic and dielectric materials to create a temperature-sensitive structure that serves as the moving element in a variable-plate capacitor. The microcantilevers are integrated directly onto a CMOS integrated circuit, and all microcantilever materials are compatible with standard silicon IC foundry processing. Sensitivity modeling and measurements indicate an order-of-magnitude improvement over the current industry standard, vanadium oxide microbolometers.

Summary:
Uncooled vanadium oxide (VO$_x$) and amorphous silicon (a-Si) microbolometers are presently the technologies of choice for thermally sensing and imaging long wave infrared radiation. However, the performance of these devices has not improved significantly in recent years and studies indicate that these technologies may be reaching their performance limits [1,2].

Our proposed technique makes use of MEMS structures that respond mechanically to the absorption of infrared radiation. These structures were invented at the Oak Ridge National Laboratory (ORNL) [3] in the mid-1990s. Multispectral Imaging has licensed the ORNL technology [4-6] and is pursuing its commercialization using the resources at CNF.

Each pixel of our sensing array comprises the following components: an anchor, which elevates the sensing element above the substrate; a thermally isolating dielectric element of the paddle support arm; a thermally sensitive bimorph element of the paddle support arm; and the paddle itself. The gap between the paddle and the substrate serves as a resonant cavity for infrared radiation, and enhances the absorption of energy by the paddle. Heat flows from the paddle to the relatively cooler substrate through the thermally sensitive part of the support arm, causing the support arm to bend and the paddle to change its height relative to the substrate. The temperature-sensitive paddle height is measured capacitively by the underlying CMOS integrated circuit.

Although the process of integrating the MEMS structures on top of the CMOS circuit has not been completed, extensive modeling and measurements of both the CMOS and the MEMS structures separately indicate an order-of-magnitude improvement over microbolometer performance.

References:
High Sensitivity Uncooled Microcantilever Infrared Imaging Arrays

CNF Project #: 1202-04
Principal Investigator: Gregory Simelgor
User: Lijun Jiang

Affiliation: Multispectral Imaging, Inc.
Primary Funding: Venture capital
Contact: gsimelgor@multispectral.net, ljiang@multispectral.net
Web Site: http://www.multispectral.net

FUNDAMENTAL NOISE LIMITS and NEDT (mK)

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Figure 1, top left: Schematic diagram showing the operating principle of the bimorph microcantilever IR sensor.

Figure 2, bottom left: SEM images of single pixel. Cantilever bending response to changes in the tensile and compressive stresses in the thin film layers used to fabricate the microcantilever sensor structure.

Figure 3, above: SEM images of pixels from small arrays. To date, we have fabricated test structures with single pixels and small arrays of up to 64 x 64 pixels.
Integration of Nanophotonic Devices Based on Silicon & Polymer Waveguides

CNF Project # I227-04
Principal Investigator: Roberto Panepucci

Abstract:

We are developing integrated photonic devices based on silicon and polymer materials. Micro-electro-opto-mechanical systems (MEOMS) fabricated on a silicon-on-insulator technology are integrated with wavelength selective filters to develop complex sensor arrays and switches. Polymer materials have been investigated as a platform for incorporating passive as well as active functions in integrated photonic devices. CNF’s capabilities serve to complement FIU’s Motorola Nanofabrication Facility inaugurated in 2005.

Summary:

We are investigating the fabrication of suspended silicon waveguides, or micro-electro-opto-mechanical systems (MEOMS), that can be used to detect displacement in fully integrated fiber-optic devices. As part of the development of this device we have also investigated the fabrication of ultra-small silicon ring resonators. These ring resonators act as filters for on-chip wavelength division multiplexing (WDM) and demultiplexing. The ability to perform on-chip WDM is critical for querying the large arrays of integrated sensors we are developing.

Silicon waveguides are fabricated using silicon-on-insulator wafers with a Si device layer of 200-400 nm thickness over a 3 µm buried oxide layer. The devices are patterned using HSQ resist at 100 kV using the Leica VB6. Waveguide lengths vary from 5-10 mm and incorporate a previously developed nanotaper coupling structure that allows efficient coupling of > 90% of light from a tapered fiber. The waveguides are etched in the PT770 using Cl₂:BCl₃:Ar ICP-RIE. Typically devices are clad in SiO₂ using the GSI PECVD, diced and polished for testing. We are developing release methods to allow a suspended section of the device to interact with the environment. Electrostatic actuation of the device is implemented with on-chip electrodes. Figure 1(a) shows an SEM of the gap between cantilever waveguide and receiving waveguide in such a device. Figure 1(b) shows an optical micrograph of a 2 µm radius ring resonator used to multiplex the optical signal between different parts of the photonic circuits. This device has a free-spectral-range (FSR) of 47 nm, the largest for single ring resonator reported to date.

Polymer devices are of interest due to the ease of integration with nanocrystals, chromophore molecules and other dopants. We are investigating the use of 1D and 2D photonic crystal structures integrated with polymer waveguides. We are using PMMA/DR1 mixtures which have allowed us to directly pattern the waveguide layer in a single electro-beam lithography step using grayscale to achieve 3D structures. Lithography is carried out with the Leica VB6 at 100 kV using thin Au overlayer for charge dissipation. The polymer is deposited over a 30 nm Si₃N₄ capped 4 µm SiO₂ lower cladding. A wet etching step is used to release the structure. Figure 2(a) shows a SEM of cross section of the resulting 2D photonic crystal layer. Figure 2(b) shows a SEM top view of the 1D cavities inserted in a ridge waveguide.

Simulation of these structures is performed with a commercial package from RSOFT. The characterization of these devices is carried out in our labs through a fiber-optic test setup which includes piezoelectric nanopositioners and infrared imaging.

References:

Integration of Nanophotonic Devices Based on Silicon & Polymer Waveguides

CNF Project # 1227-04
Principal Investigator: Roberto Panepucci
Users: Xuan Wang, Roberto Panepucci

Affiliation: Electrical and Engineering Department, Florida International University
Primary Funding: NSF, AFOSR
Contact: roberto.panepucci@fiu.edu

Figure 1(a): SEM of the gap between cantilever waveguide and receiving waveguide in a released device.

Figure 1(b): Optical micrograph of a 2 µm radius ring resonator with a free-spectral-range (FSR) of 47 nm, the largest for single ring resonator reported to date.

Figure 2(a): SEM of cross section of the PMMA/DR1 2D photonic crystal layer.

Figure 2(b): Shows a SEM top view of the 1D cavities in a ridge waveguide.
Super Compact Gratings for DWDM Wavelength Mux/Demux Applications

CNF Project # 1366-05
Principal Investigator: Seng-Tiong Ho

Abstract:
There are current needs for various monolithically integrated photonic devices and subsystems with dense wavelength division multiplexing (DWDM) capabilities. While making enormous progress, the current integrated DWDM Mux/Demux devices are still large in size (typically ~ 100-1,000 mm² for AWG). Meanwhile, the typical size of active devices, such as laser diodes, is < 0.5 mm², which is > 1,000x smaller. Thus the size of wavelength Mux/Demux will dominate all other active-passive components when integrated. This makes the wavelength Mux/Demux among the most expensive components to integrate.

Research Summary:
We have devised a monolithically integrated ultra-compact wavelength Mux/Demux on InP platform that can have a size of ~ 0.5-5 mm² at DWDM resolution, which will open up various opportunities that could lead to WDM-On-Chips that are substantially more compact than current technology. As the costs of integrated devices are proportional to the chip size, this will mean substantial reduction in the chip costs as well as increase in the integrated on-chip functionalities.

The ultra-compact wavelength (λ) Mux/Demux is based on a monolithically integrated curved diffraction grating on InP chip, which provides wavelength dispersion and beam focusing. Our approach results in high spatial resolution for the grating dispersion and hence high wavelength resolution at small physical size.

Summary:
In the current year, we fabricated some initial wavelength demultiplexer structures based on the super compact grating. The device fabrication process can be summarized as follows: we start with epitaxial grown InP/InGaAsP wafers. First, a layer of SiO₂ is grown by PECVD to act as InP/InGaAsP etch mask later. Then, the device pattern is written by e-beam lithography using PMMA as resist. The PMMA pattern is etched in RIE and transferred to the SiO₂ layer. The SiO₂ layer then acts as etching mask for InP/InGaAsP ICP etching, with typical etching depth of ~ 4 µm.

The initial fabrication run shows promising results. With a mm-size device, we are able to demonstrate wavelength resolution of 50 GHz. We are continuing to optimize the grating design as well as the fabrication process, and will push the device size and wavelength resolution further.

References:
Super Compact Gratings for DWDM Wavelength Mux/Demux Applications

CNF Project # 1366-05
Principal Investigator: Seng-Tiong Ho
User: Yingyan Huang

Affiliation: Department of Electrical Engineering and Computer Science, Northwestern University
Primary Funding: NSF
Contact: sth@ece.northwestern.edu, yingyan@ece.northwestern.edu

- Fabricated a novel super compact grating with minimal aberration, thus allow narrow slit size and large diffraction angle.
- Achieved 50 GHz wavelength resolution with device size < 1 mm².

Figure 1, top left: SEM picture of the fabricated wavelength demultiplexer with detector.

Figure 2, bottom left: SEM picture of the super compact grating.

Figure 3, above: Initial measurement result of the device transmission spectrum, showing 50 GHz pass band. The different height of the peak comes from the quantum well absorption at different wavelength.
Metal-Semiconductor-Metal Ultraviolet Photodetector on AlGaN

CNF Project # 1390-05
Principal Investigator: William R. Donaldson, Roman Sobolewski

Abstract:

Metal-semiconductor-metal photodiodes on AlGaN substrates are being developed to measure ultra-fast, deep-UV pulses on the OMEGA Laser System.

Summary:

This work involves the fabrication of metal-semiconductor-metal photoconductive detectors and photodiodes on aluminum gallium nitride (AlGaN). These photon detectors have finger widths and spacings between 150 to 5000 nm and are expected to have response times in the picosecond (ps) regime. At the University of Rochester, we have demonstrated that metal-semiconductor-metal (MSM) photodetectors have response times as short as 0.8 ps when fabricated on GaAs and 5.0 ps on Si. These devices have been used for the ultra-fast detection of photons not only because of their superior speed performance, but also because their planar geometry makes them suitable for use as either a discrete devices or as part of an optoelectronic integrated-circuit. AlGaN will allow us to extend this performance into the deep ultraviolet. Fundamental properties of these devices will be characterized at the University of Rochester, using an electro-optic sampling system. The devices will then be packaged for use on the OMEGA inertial confinement fusion (ICF) laser system as UV and x-ray detectors. Unlike GaN devices, which were fabricated at CNF in previous years, the AlGaN device should be blind to the OMEGA operating wavelength of 351 nm.

Two graduate students, Allen Cross and Shuia Wu, associated with the project, have been trained at CNF in the past year and have fabricated the first AlGaN devices. The finger widths of the MSM structure ranged from 0.15 µm to 5 µm and two different active areas (25 x 25 and 50 x 50 µm square) were patterned. The smaller device have higher speed but lower sensitivity. To couple the electrical signal out of the devices, contact pads are fabricated adjacent to the interdigitated optically active region. For the ultra-fast characterization, the contact pad are rectangular regions equal to the area of the inter-digitated region. However, for applications on the OMEGA laser system, a smooth transition to a 50-Ohm transmission line is needed. In this case, an exponentially tapered pad is used to match the device to a 1-mm wide micro-strip transmission line.

The current-voltage characteristics of these two different geometries have been measured. The dark current increases by a factor of 10 between the small rectangular pads and large exponentially tapered pads. Thus the dark current is dominated by leakage between the pads, which are as much as 50 µm apart, rather than the fingers, which are 0.5 µm apart. We are exploring additional processing steps to reduce the pad leakage current.
Metal-Semiconductor-Metal Ultraviolet Photodetector on AlGaN

CNF Project # 1390-05
Principal Investigator: William R. Donaldson, Roman Sobolewski
Users: Allen Cross, Shuai Wu

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Primary Funding: U.S. Dept of Energy Office of Inertial Confinement Fusion under Cooperative Agreement No. DE-FC52-92SF19460, the University of Rochester, and the New York State Energy Research and Development Authority
Web Site: http://www.lle.rochester.edu/

Figure 1, top left: The MSM photodiodes are fabricated on AlGaN. The MSM structure size shown here is 25 x 25 µm. The finger width is 0.3 µm (a) and 1.0 µm (b).

Figure 2, bottom left: The large exponentially tapered pads used for impedance matching to a 50-Ohm transmission line.

Figure 3, above: The current-voltage characteristics of these two different geometries shown in Figures 1 and 2 have been measured. The dark current increases by a factor of 10 between the small rectangular pads and large exponentially tapered pads.
CoFeB Spin Valve Nanopillars for Spin Transfer Studies

CNF Project # III-80
Principal Investigators: Robert A. Buhrman, Daniel C. Ralph

Abstract:

We have fabricated spin valve nanopillars with Co$_{60}$Fe$_{20}$B$_{20}$ magnetic layers in order to study both magnetic and spin-transfer torque behavior in this ferromagnetic material. Due to the very high tunnel magneto-resistance that has been demonstrated in magnetic tunnel junctions with CoFeB electrodes and MgO tunnel barriers, this material is a strong candidate for spin-transfer torque switched magnetic memory.

Summary:

The transfer of spin angular momentum from spin-polarized conduction electrons to the local moment of a nanoscale ferromagnetic element can be used to manipulate the magnetic orientation of that element through the application of spin-torque [1-3]. The possibility that this can form the basis of a write mechanism for magnetic random access memory that is simultaneously fast, dense, nonvolatile, and consumes low power was significantly advanced by the demonstration of very high tunnel magneto-resistance in CoFeB/MgO/CoFeB magnetic tunnel junctions [4,5] which enables fast access times.

In order to study the magnetic properties of this CoFeB as well as its response to spin-polarized current, we have fabricated spin valve nanopillars from continuous films consisting of (in nm): non-magnetic underlayers/CoFeB 20 / Cu 60 / CoFeB 3.5 / Cu 50 / Pt 30, where CoFeB is Co$_{60}$Fe$_{20}$B$_{20}$. The pillars are lithographically formed into elliptical shapes by electron beam lithography followed by argon ion mill etching. Typical nanopillar dimensions are ~ 60 x 160 nm wide and ~ 70 nm tall. The pillars are electrically isolated with SiO$_2$, and a combination of photolithography, etching and ion-beam deposition are used to form the top and bottom electrical contacts.

Samples are studied by sweeping either the external magnetic field or the DC current while measuring differential resistance. The resistance state reflects the magnetic configuration of the sample through the giant magneto-resistance effect [6], where the resistance is higher when the magnetizations of the two magnetic layers are aligned antiparallel and lower when aligned parallel. In addition to switching the magnetic layers with an external magnetic field, a sufficiently large spin-polarized current can exert enough torque on the thinner nanoscale magnetic element that precession is induced that can ultimately reverse the magnetic orientation of that layer. Initial studies indicate critical current densities of $J_c \sim 4-8 \times 10^7$ A/cm$^2$ for magnetic reversal which is consistent with the saturation magnetization, $M_s = 1180$ emu/cm$^2$.

Future studies include measurements of magnetic damping in the CoFeB free layer as well as the effect on non-uniform magnetization states on switching, both of which are critical for utilization of CoFeB in magnetic memory.

References:

CoFeB Spin Valve Nanopillars for Spin Transfer Studies

CNF Project # III-80
Principal Investigators: Robert A. Buhrman, Daniel C. Ralph
User: Gregory D. Fuchs

Affiliation: Departments of Applied Physics, Cornell University
Primary Funding: Center for Nanoscale Systems
Contact: gdf9@cornell.edu

- Spin valve nanopillars with magnetic layers composed of Co$_{60}$Fe$_{20}$B$_{20}$.
- This material is amorphous as deposited, but can crystallize after annealing.
- Very high magnetoresistance has been demonstrated in MgO magnetic tunnel junctions using CoFeB electrodes. These devices allow us to study the magnetic properties and spin-transfer torque behavior of this material.
- Measurements of differential resistance (dV/dI) vs. current (a) and magnetic field (b).
Atomic Scale Characterization of Magnetic Multilayers

CNF Project # III-80

Principal Investigators: Robert A. Buhrman, John Silcox

Abstract:
We have used a combination of scanning transmission electron microscopy (STEM) and electron energy-loss spectroscopy (EELS) techniques to study the nature of interfaces in sputtered, permalloy (Ni$_{81}$Fe$_{19}$) based magnetic multilayers used in nanopillar spin valves. In particular, we have investigated the atomic structures of permalloy (Py)/Cu interface used in a spin valve configuration, Py/AlO$_x$ interface used as a surface oxide passivation and a Ta/CuO$_x$ stack as a smooth seeding layer for such magnetic multilayer growth. Ta/CuO$_x$ stack improves the interfacial roughness via oxygen diffusion along grain boundaries of Cu to react with Ta to form TaO$_x$. Analysis of the Py/Cu interface subject to moderate temperature treatments displays short-range interdiffusion of Ni and Cu. An AlO$_x$ capping provides a sharp oxygen concentration profile (~1 nm) across Py/AlO$_x$ interface accompanied by Fe segregation into the alumina to form iron oxide.

Summary:
The asymmetry in the conduction probabilities of different electron spins forms the basis for a number of interesting physical phenomena such as giant magnetoresistance effect (GMR) [1], tunneling magnetoresistance effect (TMR) [2] and their inverse effect, spin-momentum transfer (SMT) [3]. All of these quantum mechanical effects are not only important for fundamental science but also play a big role in determining the direction of future information technologies.

The increasing demand for ultra-high information storage density necessitates a better understanding of the nano-scale physics of all the aforementioned phenomena. This can only be achieved by a thorough investigation of the complex microstructure including interfacial interactions and roughness. In the present study, we use scanning transmission electron microscopy (STEM) and electron-energy loss spectroscopy (EELS) to probe the atomic-scale interactions of one of the most commonly used soft magnetic materials, Py. Py with its ultra-low coercive fields and high spin polarization (45%) proves to be an ideal choice for magnetic sensing applications via the GMR and TMR effects. Its relatively low saturation magnetization makes it easier to manipulate by spin-polarized currents via the spin-torque effect [4].

The improvement in interfacial roughness by using a Ta/CuO$_x$ bilayer is achieved by grain boundary diffusion of oxygen along the CuO$_x$ which reacts with Ta and forms an interfacial TaO$_x$ layer. Energy-dispersive x-ray (EDX) measurements of this transition layer suggest an O/Ta ratio of 1.6 ± 0.2. In addition, the observation that Ta grown on CuO$_x$ being a chemically sharp surface as opposed to CuO$_x$ grown on Ta displaying significant Ta/Cu intermixing is consistent with their surface free energies at room temperature calculated from their internal free enthalpies of atomization with that of Cu (1934 mJ/m$^2$) being significantly lower than that of Ta (3018 mJ/m$^2$).

For a Py/Cu spin valve deposited on such a smooth substrate a significant short range interdiffusion between Ni and Cu can be seen at both interfaces within 1 nm with traces of Ni present almost everywhere inside the Cu spacer layer. Although molecular dynamics simulations suggest, that Py grown on Cu is more chemically diffuse then Cu grown on Py, it is hard to come to this conclusion from the EELS measurement since the uncertainty of the position of the interface is determined by the Py/Cu interfacial roughness (about 1-1.2 nm).

A significant portion of the Fe segregation into the AlO$_x$ occurs within 1.5 nm of the Py/AlO$_x$ interface. Thin Al passivation layer does successfully eliminate Ni oxidation and also increases the metal to oxide ratio for iron component. The remaining iron in oxidized form is a result of Fe segregation into the AlO$_x$ layer and oxidation therein. Having such an antiferromagnetic oxide in a Py/AlO$_x$ magnetic tunnel junction can have an adverse effect in device performance.

References:
Atomic Scale Characterization of Magnetic Multilayers

CNF Project # III-80
Principal Investigators: Robert A. Buhrman, John Silcox
Users: Ozhan Ozatay, Andrei Mkhoyan
Affiliation: Department of Applied and Engineering Physics, Cornell University
Primary Funding: NSF, DARPA
Contact: oo24@cornell.edu, rab8@cornell.edu, js97@cornell.edu

• STEM Bright Field image of a Py/Cu/Py trilayer with a smooth Ta/CuO$_x$ substrate capped with AlO$_x$ passivation layer.
• Bright Field image of Ta/CuO$_x$ interface shows contrast for the existence of TaO$_x$ filled grain boundary gaps.
• EELS spectra taken across Py/Cu/Py trilayer with 0.92 nm steps.
• Concentration profiles of O, Ni and Fe across Py/AlO$_x$. 
Magnetic Vortex Dynamics Driven by DC Spin-Polarized Current

CNF Project # III-80
Principal Investigators: Robert A. Buhrman, Daniel C. Ralph

Abstract:
We fabricated nanopillar spin-valves with a thick spin-polarizing layer. In the presence of a spin-polarized current, we observe microwave dynamics, which correspond to precession of a magnetic vortex core in the thick magnetic layer, coupled to oscillations of the thinner layer. Dynamics are observed for in-plane and out-of-plane applied fields. The oscillation frequency varies weakly with the applied field strength and current amplitude. The oscillations have quality factors of up to 3400 and high power densities.

Summary:
In the presence of a magnetic field and a spin-polarized current, a magnetic moment obeys a modified version of the Landau-Lifshitz-Gilbert (LLG) equation with an additional term describing the torque exerted by the spin current [1,2]. The spin transfer torque (STT) effect provides a new handle for manipulating magnetic particles at the nanoscale and could have possible applications for current-switched non-volatile memory and as DC-driven microwave sources. Experiments have demonstrated that STT can be used to induce current-controlled hysteretic switching [3], as well as persistent microwave dynamics [4] in magnetic trilayers, and can drive domain wall motion [5].

An important issue, both theoretically and for technological applications, is understanding how to increase the coherence of STT-driven microwave oscillations. Previous experiments focused on uniform precession modes [4]. In our experiment, we use STT to drive oscillations of a non-uniform magnetic structure, a magnetic vortex formed in a nanopillar spin valve.

The samples were fabricated by depositing (Ta (5nm)/Cu (2 nm)/CuO (20 nm)) x 2/Ta (20 nm)/Py (60 nm)/Cu (40 nm)/Py (5 nm)/Cu (20 nm)/Pt (30 nm) on a thermally-oxidized silicon wafer, using DC-magnetron sputtering. Electron-beam lithography was used to define ~160 nm x 75 nm ellipses. Then, ion milling was used to etch to the bottom of the thick Py layer, defining a pillar-shaped structure. Contact is made by bonding Au ribbon to lithographically-patterned Cu leads. The sample is DC-current-biased to generate STT on the magnetic layers. The resulting oscillations of the magnetizations of the Py layers produce a time-varying voltage via the giant magneto-resistance (GMR) effect. Simulations indicate that these oscillations are consistent with the precession of a magnetic vortex in the thick Py layer (gyrotropic vortex mode). Such vortex oscillations have previously been observed in experiments on single Py dots where a magnetic field was used to excite them [6]. We find that the frequency of the gyrotropic mode increases with increasing perpendicular-to-plane applied magnetic fields and decreases with increasing in-plane fields applied along the major axis of the ellipse, consistent with a tightening and, respectively, distortion of the magnetic vortex. Pinning introduces anomalies in the general trend. We observed constructive and destructive interference of the gyrotropic mode with other modes, likely resulting from coupling between the two magnetic layers. Additionally, we have determined that the gyrotropic mode is highly coherent, with measured quality factors of up to 3400 at room temperature.

References:
Magnetic Vortex Dynamics Driven by DC Spin-Polarized Current

CNF Project # III-80
Principal Investigators: Robert A. Buhrman¹, Daniel C. Ralph²
Users: Vlad Pribiag¹, Patrick Braganca², Gregory Fuchs², Ozhan Ozatay², Jack Sankey¹

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- We study magnetization dynamics in nanopillar spin valves that contain a magnetic vortex (Figure 1).

- Spin-torque drives vortex precession in the thick Py layer, which couples to oscillations in the thin Py layer (Figure 2 (vertical axis is power in units of Johnson noise at 300 K)).

- Oscillations have high quality factors of up to 3,400 (Figure 3).

- We observe frequency modulation driven by an oscillating applied magnetic field (Figure 3).

![Figure 1](image1.png)

![Figure 2](image2.png)

![Figure 3](image3.png)
Enhanced Magnetic Damping in Spin-Transfer Excitation

CNF Project # II-80
Principal Investigator: Robert A. Buhrman

Abstract:
Terbium doping in permalloy spin valves dramatically increase the critical currents needed for switching and dynamic behavior onset while reducing the microwave noise, without significantly affecting other properties. Furthermore, the critical currents of terbium doped sample increases linearly with decreasing temperature, while undoped samples show little variation.

Summary:
Recent experiments on Co(fixed)/Cu/Co(free) magnetic nanopillars [1,2] have shown that a spin-polarized current (I) applied perpendicular-to-plane (CPP) can change the relative alignment of the two magnetic layers. The prevailing theories [3,4] indicate a transfer of spin angular momentum at the normal metal/ferromagnet interface from the conduction electrons to the magnetic moments, giving rise to a mutual torque. Upon reaching a critical current, the torque becomes great enough to overcome the intrinsic Gilbert damping and flip the thinner layer ferromagnet (free) either parallel (p) or anti-parallel (ap) to the thicker layer ferromagnet (fixed), depending on the direction of the current. The so-called ‘spin transfer’ switching has stimulated interest as a potential write mechanism for non-volatile memory elements, although it would act as a parasitic effect for CPP field sensors such as those used in modern hard drive read heads. Further work has shown that high frequency dynamics can also be excited by the spin-torque [3].

While magnetic damping is understood to play a fundamental role in spin-torque phenomena, little experimental work has been done to study the effect of varying the damping parameter $\alpha$. Recently, light terbium (Tb) doping in thin films of permalloy (Py) has been shown to increase $\alpha$ by several orders of magnitude [4]. To directly study the effect of increased $\alpha$ on spin-transfer systems, we have fabricated 0.05 square micron Py/Cu/Py nanopillar spin valves with Tb-doping between 0 and 2% in the free layer. Electron beam lithography and ion beam etching techniques were used to define the nanopillars, while a number of oxide deposition methods were used to electrically isolate the top and bottom leads of the device.

We find that, while the gigantic magneto-resistance (GMR) varies less than 20%, the critical currents for reversibly switching the free layer (proportional to $\alpha$) are two to three times larger in the 2% Tb samples than in pure Py samples at room temperature, and eight times larger at 150 K. This substantial increase is still considerably less than the increase in $\alpha$ observed in the bulk film measurements of similar composition samples, suggesting that processes other than intrinsic spin-orbital coupling can dominate $\alpha$ in spin-transfer nanopillars. As temperature is increased from 4 K to 300 K, the effect of Tb doping decreases linearly. The Tb doping also increases the critical current for the onset of processional dynamics. These results suggest Tb doping as one approach to reducing the negative impact of spin torque effects on nanoscale spin valve and tunnel junction read head sensors.

References:
Enhanced Magnetic Damping in Spin-Transfer Excitation

CNF Project # III-80
Principal Investigator: Robert A. Buhrman
User: Eric M. Ryan
Affiliation: Applied and Engineering Physics Department, Cornell University
Primary Funding: CNS under NSF - Engineering grant
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- 0.05 square micron Py/Cu/Py nanopillar spin valves fabricated with Tb-doping 0 and 2% in the free layer to studying increased damping in spin-transfer systems.
- The Tb doping increases the critical current for switching and for the onset of processional dynamics.
- The effects will be beneficial for next generation CPP hard drive read heads.

Figure 1, above: Cross-sectional schematic and SEM image of a nanopillar.

Figure 2, top right: Tb-doped samples require higher currents to switch.

Figure 3, bottom right: The effect of Tb doping is stronger at lower temperatures, as expected.
Optical Studies of Single Molecule Transistors

CNF Project # 598-96  
Principal Investigator: Daniel C. Ralph

Abstract:
We are studying the effect of applied light on electron transport in single molecule transistors. Our three-terminal devices are fabricated using electron-beam lithography followed by electromigration. The experimental set-up for illuminating the devices consists of a tunable Ar/Kr laser coupled into an optical cryostat. Light-induced changes in molecular conductance can be studied as a function of the gate and bias voltages.

Summary:
We are working to extend the study of single molecule transistors [1,2] to include light-induced effects. Electronic excitations in an optically active molecule can be probed using tunneling electrons provided the flow of electrons through the molecule is comparable to the relaxation time of the excitation. We will be looking for features in the electron tunneling spectra which, in addition to being a function of bias voltage and gate voltage, should depend on the wavelength and power of the incident light. Because the nature of the response should be molecule-specific, light studies should also provide clear confirmation that the device contains the molecule of interest, rather than some other species which might produce artifacts.

To make a single molecule transistor, a molecule must be connected within a nanometer-scale gap between two conducting electrodes. Gaps of this size cannot be made using conventional lithography techniques, so we fabricate a continuous wire and then create the gap using electromigration [3]. The fabrication is done by starting with an insulating SiO$_2$ layer on Si. A thin Al gate electrode is deposited where the devices will be, and a native oxide is allowed to form. We then deposit Au or Pt wires (150 nm wide, 500 nm long, and 10 nm thick) on top of the gate electrode with e-beam lithography and liftoff. These wires are contacted by larger Au leads.

We clean each chip in an oxygen plasma and then deposit molecules from dilute solution. The sample is loaded into an optical cryostat which enables studies at liquid helium temperatures (4.2 K) for improved energy resolution in electron-tunneling spectroscopy. We break the wires in situ using electromigration with low-resistance contacts-ramping the voltage bias until the wire breaks, to leave a gap of order 1 nm between the resulting electrodes. In some of the devices, we find that a molecule then bridges this gap. The presence of a molecule can be detected by Coulomb blockade, in which current turns on above a gate dependent threshold bias voltage.

The molecule we have chosen for initial experiments is Ru$_2$(tppz)$_3$, synthesized by the Abruña group at Cornell. This molecule was chosen because it is redox active at low potentials, stable in both air and acetonitrile, and has a large optical cross section. In addition to this ruthenium dimer, we may also try optical experiments involving semi-conducting quantum dots.

References:
Optical Studies of Single Molecule Transistors

CNF Project # 598-96
Principal Investigator: Daniel C. Ralph
Users: Jacob E. Grose, Janice Wynn Guikema

Affiliation: Department of Physics, Cornell University
Primary Funding: NSF/MRSEC and NSF/NIRT through the Cornell Center for Materials Research
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Objective: Study conductance changes in single molecule transistors (SMTs) upon application of laser light at low temperature.

Figure 1, above: SEM images of (a) an unbroken wire and (b) a wire broken by electro-migration. In some devices a molecule deposited on the surface can be trapped in the gap, creating a SMT.

Figure 2, below: Schematic of experimental setup. (Inset) Photograph of optical cryostat.
Single Electron Transistors made using Chemically Synthesized Metal Nanoparticles

CNF Project # 598-96
Principal Investigator: Daniel C. Ralph

Abstract:
By combining electron-beam lithography with chemical self-assembly, we fabricate single-electron-transistor devices incorporating individual chemically-synthesized metal nanoparticles, and use the devices to perform low-temperature electron tunneling spectroscopy of the electron-in-a-box states of the nanoparticle. This allows us to determine the discrete electronic spectra of individual nanoparticles which are-by virtue of chemical synthesis-well defined in their composition, size and shape.

Summary:
Electron tunneling at low temperatures (\(< 100 \text{ mK}\)) has been used in the past to measure electron-in-a-box quantum states of evaporated metal nanoparticles formed by surface tension on a non-wetting substrate. Although a variety of electronic interactions could be observed, including superconducting pairing, magnetic exchange forces, and spin-orbit interactions, the size and shape of the evaporated particles could not be well-controlled.

In this project, we use chemical techniques to both synthesize and assemble monodisperse gold colloid of nearly spherical shape into a single-electron-transistor geometry. Chemical synthesis enables us to control a particle’s size-and hence its mean level spacing—and organic molecules with appropriate end groups allowing us to tether the particles to the electrodes with symmetric tunnel barriers. The transistor geometry consists of two lateral gold electrodes (source and drain) bridged by a nanoparticle, on top of a gate electrode, and yields good electrostatic coupling of the nanoparticle to the gate [1]. This coupling is essential to study electron interaction effects as it allows one to change the number of electrons in the particle and see how this modifies the particle’s spectrum.

Source and drain electrodes are fabricated by defining a 16 nm thick gold wire with a 100 nm wide constriction (electron-beam lithography on bilayer PMMA) on top of an oxidized silicon substrate, which serves as a gate electrode in the final device. By running a current through the constriction, a nanometer-sized gap between source and drain is formed via electromigration [2]. To incorporate metallic nanoparticles from a colloidal solution into this gap, we first assemble a monolayer of [(aminoethylamino)propyl]trimethoxysilane on top of all three electrodes, and then immerse the chip into a colloidal solution whose pH has been adjusted to yield an attractive electric force between the negatively charged gold particles and the positively charged amino groups of the organic monolayer. The organic monolayer also serves as a tunnel barrier between the particles and the source/drain-electrodes.

After assembly, the chip is rinsed, dried and cooled in a dilution refrigerator to measure the tunnel current I as a function of the source-drain bias V and the gate voltage \(V_{\text{gate}}\). When plotting the differential conductance \(dI/dV\) versus V and \(V_{\text{gate}}\) for a device where a single particle bridges the gap, we observe diamond shaped areas of zero conductance due to Coulomb blockade, and sets of parallel lines of higher conductance due to the particle’s discrete level spectrum. When we apply a magnetic field, each level Zeeman-splits into two levels, and different levels avoid each other at higher magnetic fields due to spin-orbit interaction. Performing these measurements on gold particles of various sizes allows us to investigate how g-factors and spin-orbit interaction depend on the mean level spacing.

References:
Single Electron Transistors made using Chemically Synthesized Metal Nanoparticles

CNF Project # 598-96  
Principal Investigator: Daniel C. Ralph  
Users: Ferdinand Kuemmeth, Kirill Bolotin

Affiliation: Physics Department, Cornell University  
Primary Funding: NSF/DMR and ARO  
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Figure 1, top left: Chemically-synthesized gold nanoparticles (10 nm diameter) assembled into a broken gold wire by means of an organic monolayer, to make a single-electron transistor.

Figure 2, bottom left: Differential conductance of metal nanoparticle transistor as a function of gate voltage and source drain bias, showing one particle’s discrete energy-level spectrum.

Figure 3, above: A particle’s spectrum as a function of magnetic field. We observed Zeeman splitting and avoided level crossings due to spin and spin-orbit effects.
The Kondo Effect in Mechanical Break Junctions

CNF Project # 598-96
Principal Investigator: Daniel C. Ralph

Abstract:
Our group recently demonstrated the fabrication of single-molecule transistors that combine the ability to mechanically adjust the spacing between two electrodes and to shift the energy levels of a molecule with a gate electrode [1]. Here we use this device geometry to study the Kondo effect, a many-body phenomenon that arises from the coupling between a localized spin and a sea of conduction electrons. By mechanically adjusting the electrode spacing, we are able to tune the Kondo effect.

Summary:
Our devices are fabricated on thin (200 µm) silicon wafers, which are flexible and allow for mechanical bending. We begin with a thermal growth of silicon oxide, followed by a wet etch to form trenches, and a re-growth of thin oxide in the trenches over which gold bridges will later be suspended. Using a series of photolithography and electron-beam lithography steps, we define the bonding pads as well as the critical features, namely 50 nm-wide gold wires. These wires are then suspended 40 nm above the silicon substrate using a combination of dry and wet etches. To incorporate molecules in our devices, 50 microliters of a dilute solution of C$_{60}$ or [Co(Tpy-SH)$_2$]$^{2+}$ are deposited onto a chip. These molecules are of interest because the Kondo effect has been previously observed with them [2,3]. The chip is subsequently cooled down to 1.5 K in a cryostat, and a nanoscale gap is created in the gold wires by electromigration [4]. After electromigration, we find that one or a few molecules can sometimes be found bridging this gap. Our cryostat is equipped with a motorized mechanism that enables controlled bending of the silicon substrate, which in turn allows us to adjust the inter-electrode gap with very fine precision. We measure transport characteristics of our devices as a function of bias voltage, magnetic field, electrode spacing, and temperature.

Measurements on individual C$_{60}$ and [Co(Tpy-SH)$_2$]$^{2+}$ molecules in our device geometry show signatures of the Kondo effect in a quantum dot, namely a zero-bias peak in $dI/dV$ which splits in a magnetic field and is suppressed as a function of increasing temperature. This zero-bias peak reflects the formation of a spin-singlet state between a localized unpaired electron on the molecule and the conduction electrons of the leads at the Fermi energy. We are able to tune both the height and width of the Kondo resonance in our devices as a function of electrode spacing. The former gives us information about the asymmetry in coupling of the molecule to each electrode, and the latter tells us about the Kondo temperature, which is the many-body energy scale characteristic of the binding energy of the spin-singlet state. While we have been able to tune the Kondo effect and modulate current flow through single molecule devices by varying electrode spacing, we have not yet identified the dominant mechanism for the observed changes. Varying the electrode spacing could both alter the molecule-lead coupling as well as induce a gating effect, either of which could modify the Kondo effect.

References:
The Kondo Effect in Mechanical Break Junctions

CNF Project # 598-96
Principal Investigator: Daniel C. Ralph
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Figure 1, top left: Scanning electron micrograph of a gold bridge suspended 40 nm above a silicon substrate.

Figure 2, bottom left: Differential conductance traces for a C_{60} molecule in a gold break junction at various temperatures, after subtraction of a small background. Inset: A fit to theory yields a Kondo temperature of 23 K.

Figure 3, above: Differential conductance traces for the same device at several electrode spacings. Inset: Conductance map showing the evolution of the Kondo peak as the electrodes are pulled apart.
Spin-Transfer-Driven Ferromagnetic Resonance of a Single Nanomagnet

CNF Project # 598-96
Principal Investigators: Daniel C. Ralph, Robert A. Buhrman

Abstract:
We have developed a new form of ferromagnetic resonance (FMR) spectroscopy which uses the spin transfer torque from an RF current to probe the normal modes of individual nanoscale magnets. We measure the magnetic dynamics of samples smaller in volume by more than a factor of 50 compared with other techniques. Studies of the resonance frequencies, amplitudes, line-widths, and line shapes as a function of microwave power, DC current, and magnetic field provide detailed new information about the exchange, damping, and spin-transfer torques that govern the dynamics in magnetic nanostructures.

Summary:
Spin-polarized current passing perpendicularly through magnetic multilayers can exert torques on the magnetic moments through direct transfer of spin angular momentum at magnetic interfaces. This spin-transfer phenomenon allows small magnetic samples to be manipulated far more efficiently than is possible through the traditional methods involving applied magnetic fields. Here we show that it is possible to use spin transfer to drive FMR in a single nanoscale magnet. We can detect the resulting magnetic dynamics by measuring a DC mixing voltage generated by the precessing nanomagnet [1].

We study magnetic multilayers patterned into nanoscale pillars. We fabricate these nanopillars by first sputtering a multilayer of 120 nm Cu / 20 nm Py (permalloy) / 12 nm Cu / 5.5 nm Py$_{0.65}$Cu$_{0.35}$ / 2 nm Cu / 30 nm Au and then evaporating 50 nm of carbon on top. Electron beam lithography is used to define an elliptically-shaped etch mask of dimensions 90 x 30 nm that is used for ion milling the pillar structure. PECVD-grown SiO$_2$ is then deposited to insulate the pillars before electrical contact to the top is made using photolithography.

The PyCu layer has a smaller magnetic moment than the Py layer, so if we apply an appropriate magnetic field perpendicular to the layers, the PyCu layer saturates out of plane while the Py layer will rotate only slightly. Applying an RF current through the pillar then induces an RF torque to the PyCu moment, exciting precession at the normal mode frequencies. The modes that we observe shift in frequency linearly with field above the saturation field of the PyCu layer, in parallel to the shift that would occur for spatially-uniform precession, but offset to higher frequencies. The offsets are likely due to the exchange field from the non-uniform character of the modes. As expected from modeling, the line shape of the lowest-order mode’s FMR signal is Lorentzian with a linewidth proportional to the dissipation. This allows an efficient measurement of the damping in a single nanomagnet. Applying DC current in addition to the RF drive, we observe a linear decrease in the effective damping, in agreement with predictions.

Previous experiments have shown that large DC currents passing through such devices can excite steady-state dynamical modes, and the resulting magnetoresistance oscillations cause the pillar to emit microwave power, making these devices candidates for field- and current-tunable microwave sources. As a function of DC current, the frequencies of the peaks in the emitted power spectrum often jump in discontinuous steps, and it has previously been a topic of much debate as to why. From the FMR measurements, we are able to identify these jumps in the DC-driven spectra as transitions between different normal modes of a magnetic layer.

This new FMR technique should have immediate utility in academia and industry for efficient probing and optimization of magnetic material parameters on the nanoscale. It should also be possible to scale the technique to much smaller magnetic volumes, approaching the molecular limit.

References:
Spin-Transfer-Driven Ferromagnetic Resonance of a Single Nanomagnet

CNF Project # 598-96
Principal Investigators: Daniel C. Ralph, Robert A. Buhrman
Users: Jack Sankey, Patrick Braganca, Ilya Krivorotov, Andrei Garcia, Kiran Thadani

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- We have developed a new form of ferromagnetic resonance (FMR) spectroscopy using spin transfer torques.
- We are able to characterize in detail the magnetic normal modes in single nanoscale devices.

Figure 1, above: Geometry of the nanopillar device.

Figure 2, right: (a) FMR peak for the fundamental mode of a 30 x 90 nm² sample. (b) Damping parameter as a function of DC current. (c) FMR scans showing many normal modes. (d) Magnetic-field dependence of these modes.
Linewidths of Spin-Transfer-Driven Precession in Magnetic Nanopillars

CNF Project # 598-96
Principal Investigators: Daniel C. Ralph, Robert A. Buhrman

Abstract:

In a magnetic multilayer device, spin-transfer torque from a spin-polarized DC current can drive magnetic layers into steady-state precessional modes [1,2]. We investigate how the linewidths of these modes depend on the angle and magnitude of an applied magnetic field. We find that the linewidths can change dramatically, decreasing by a factor of 50 in some devices, as the field is rotated away from the easy axis. We also find that the angular dependence of the linewidths depends on the device geometry.

Summary:

In a magnetic multilayer, a spin-polarized current generated by one magnetic layer can deposit spin angular momentum into a thinner magnetic layer and generate a steady-state precessional excitation of the thinner layer. This effect is of technological interest because it may allow the production of high-quality nanoscale frequency-tunable microwave sources and resonators. It has been shown previously that the coherence time of the spin-transfer-driven precession is strongly temperature-dependent—it increases with decreased temperature [2]. From a practical standpoint, it is desirable to attain long coherence times even at room temperature. To this end, we have investigated the dependence of the coherence time on another parameter: the direction of the applied magnetic field.

Our devices consist of two magnetic layers [typically permalloy (Py), Co, or CoFe] separated by a Cu spacer. One of the magnetic layers, called the free layer, is very thin (about 5 nm), thereby making it susceptible to the spin-transfer torque. The other magnetic layer, called the fixed layer, remains stationary. This is accomplished by either: a) making it very thick (40 nm), b) exchange-biasing it with an antiferromagnet, such as IrMn (8 nm), or c) making it an extended film instead of a patterned ellipse. We call the three different kinds of devices “patterned”, “exchange-biased patterned” and “unpatterned”, respectively. The free layer in all three types of devices is etched using electron-beam lithography and ion milling to give a cross section that is approximately elliptical, with an aspect ratio of 2:1 or 3:1 and a minor axis of 50-70 nm. We use photolithography to pattern bottom leads and to make top contacts.

In our measurements, we vary the angle between the applied magnetic field and the energetically-favored magnetically easy axis of the free layer. As the applied field is rotated away from the easy axis, shape anisotropy changes the effective field on the moments of the fixed and free layers, thereby altering their orientation and current-induced precession dynamics. We use a spectrum analyzer to measure the resistance oscillations arising from spin-transfer-driven magnetic precession.

We find that the field direction studied most commonly, in-plane along the magnetic easy axis of the ellipse, generally gives the largest linewidths, corresponding to the least coherent precession. As the field is rotated away from the easy axis, the linewidths can change dramatically, decreasing by a factor of 50 in some devices. We have observed the largest angular dependence in exchange-biased devices and the smallest dependence in unpatterned devices. We are currently exploring several possible mechanisms to try to understand these dramatic variations.

References:


Linewidths of Spin-Transfer-Driven Precession in Magnetic Nanopillars

CNF Project # 598-96
Principal Investigators: Daniel C. Ralph¹, Robert A. Buhrman²
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- We measure DC-current-driven precession dynamics in magnetic-multilayer nanopillars.
- The linewidths can change dramatically, by a factor of 50 in some devices, as the field is rotated away from the easy axis.

Figure 1, above: The geometry of the devices.

Figure 2: Resistance versus magnetic field sweeps.

Figure 3, right: Power spectrum of DC-driven resistance oscillations for an applied field at 0 and 75 degrees from the easy axis. (inset): Linewidth versus field angle.
Infrared Systems Progress at CNF:
Tunable and Static Infrared Frequency Selective Surfaces

CNF Project # 659-97
Principal Investigator: Glenn Boreman

Abstract:
Using e-beam lithography, frequency selective surfaces (FSS) were fabricated for operation from 3-14 \( \mu \)m. These structures consist of periodic arrays of metallic antenna elements with critical dimensions on the order of 200 nm. The antenna elements are built on a dielectric substrate with a ground plane. A process for the fabrication of a tunable FSS was developed based upon a reversed biased Schottky contact built on a Si membrane.

Summary:
Several new types of static FSS were built. These include the spiral antenna elements and square slot FSS. The circular spirals were proposed to achieve circular polarization from an FSS surface. The square slot FSS are a Babinet’s Principle conjugate to the square loop FSS that were built previously. A paper has been submitted to IEEE on their design, fabrication, and measured results.

A tunable FSS has been proposed with the use of a Schottky junction and Si membrane. A silicon on insulator (SOI) wafer is used with the buried SiO\(_2\) as an etch stop. LPCVD is used to deposit a silicon nitride etch mask on the SOI wafer. An array of square holes in the silicon nitride etch mask is used as the etch mask for a deep Si etch. The Si etchant is KOH, and the SiO\(_2\) layer is used as an etch stop. The nitride is then removed and Ohmic and Schottky contact metals are deposited on the Si. Electrical connections are made to the membrane to bias the Schottky contact. This portion of the project is still underway and publication is still forthcoming.

References:

Infrared Systems Progress at CNF:
Tunable and Static Infrared Frequency Selective Surfaces

CNF Project # 659-97
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Figure 1: Circular Spiral FSS antenna elements fabricated using e-beam lithography.

Figure 2: Square Slot FSS antenna elements fabricated using e-beam lithography.
Controlled Patterning of Nanotubes and Novel Processing

CNF Project # 786-99
Principal Investigator: David M. Tanenbaum

Background:
Low device yield and placement issues are critical for the development of carbon nanotube devices. We are focusing on ways to improve yield and reduce cost of nanotube device fabrication.

Abstract:
In collaboration with both the Davis group from BYU and the McEuen group based at Cornell, we are working on new approaches for fabricating suspended nanotube oscillators, using wet etching chemistries in place of reactive ion etching. This approach will allow for nanotubes to be released and suspended microns above gating electrodes in hopes of dramatically increasing yield of these types of devices. Adhesion forces between nanotubes and SiO₂ surfaces were quantitatively measured [1]. This year we have focused on characterizing CsOH etching of bulk silicon with thin ( < 200 nm) SiO₂ masks. The mask patterns are made with maskless photolithography [2] and transferred into thermal oxide via an HF vapor etch. The selectivity of these masks in the silicon etch process can be > 10,000:1 allowing us to do deep etching into the bulk silicon, even through wafer. This technique combined with the growth of ultralong ( > 100 µm) carbon nanotubes should enable a class of nanotube mechanical oscillators to be produced and characterized with a minimum of facilities, even in our undergraduate environment. In the area of growth of ultralong nanotubes, we have followed the path of using Argon bubbled through liquid ethanol (C₂H₅OH) to achieve growth yields of longer tubes, following reports in the literature. We have used a variety of patterned catalysts as well, including iron chloride, iron nanoparticles, and evaporated thin films of iron. Carbon nanotubes over 200 nm long have been grown under a variety of conditions, which could be suitable for our device geometry, although the literature suggests we should be able to do even better. We have modeled gas flows and created nozzles for varying the gas dynamics in the furnace tubes. In parallel with the CsOH etching studies we have been building an interference lithography system based on a 50 mW, 405 nm diode laser used to create sub micron gratings in photoresist, that can be transferred into our substrates to create large areas of periodic trenches to replace our previous samples which were written in small fields by electron beam lithography.

Summary:
A wide variety of approaches are being used to lower the cost and increase the yield of devices made with carbon nanotubes. Process development for hybrid mechanical and fluidic devices is in progress.

References:
Controlled Patterning of Nanotubes and Novel Processing

CNF Project # 786-99
Principal Investigator: David M. Tanenbaum
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Affiliation: Department of Physics & Astronomy, Pomona College
Primary Funding: NSF
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- We have characterized a variety of carbon nanotube CVD processes.
- We are using CsOH wet etches with thin thermal oxide masks for deep etching of silicon.
- We have developed an interference lithography system for writing large areas of sub micron gratings with a 405 nm diode laser.

Figure 1, top left: Carbon nanotubes suspended above a grid patterned in HSQ. The tubes were grown from ferric nitrate particles embedded in the e-beam patterned HSQ film. Charging is evident where tubes are in contact with the HSQ.

Figure 2, bottom left: Close up of trenches etched in bulk silicon wafers using a thermal oxide as an etch mask for a CsOH etchant.

Figure 3, above: An AFM image of a very long single walled carbon nanotube grown from an Argon source gas through an ethanol bubbler.
Nanofabrication of Quartz Cylinders

CNF Project # 868-00
Principal Investigator: Michelle D. Wang

Abstract:
Optical tweezers help scientists study and manipulate biological structures at the single molecule level. Recently, a variety of methods have been proposed for torque generation with optical tweezers via the transfer of spin angular momentum. Performing rotation experiments with these modified tweezers necessitates specialized, anisotropic trapping particles. We have nanofabricated a large quantity of crystalline quartz trapping particles in order to facilitate such optical rotation experiments.

Summary:
Our design consists of a circular cylinder made from crystalline quartz, such that the extraordinary susceptibility of the crystal lies in a plane parallel to the cylinder’s radial direction. Quartz cylinders were nanofabricated from a single-crystal quartz wafer using standard photolithographic techniques. Resist was spun onto the quartz surface, patterned, and developed. The wafer was etched, and the cylindrical posts were removed from the surface.

We calibrated the optical properties of the quartz cylinders, including the maximum force and torque that can be exerted on a cylinder, and assessed heterogeneity using a single beam optical trap. A 100x, NA 1.3 objective is mounted in an inverted microscope. The quartz cylinder is rotated in the trap by controlling the direction of polarization of incident light. The laser’s polarization angle is controlled with a response time of a few microseconds using acousto-optic modulators. External torque exerted on the particle is measured by monitoring the angular momentum change of transmitted light using detectors that measure the difference between right and left circular polarizations. Cylinders fabricated in the same batch have a narrow distribution of optical properties.

Future experiments will attempt to couple these cylinders to biological structures in order to apply and measure force and torque. Other experiments might consider the microfluidics of cylindrical structures.

References:
Nanofabrication of Quartz Cylinders

CNF Project # 868-00
Principal Investigator: Michelle D. Wang
User: Christopher Deufel
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Primary Funding: Keck Foundation
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- Fabrication of crystalline quartz trapping particles for optical rotation experiments.
- The quartz cylinder can be rotated in an optical trap by controlling the direction of incident light polarization.
- Very small torques can be generated and measured.
- Cylinders have a narrow distribution of optical properties.

Figure 1: Array of 0.5 µm diameter circular posts etched into a single-crystal quartz wafer.

Figure 2: Quartz cylinder after removal from the wafer surface.
Capacitance Measurements of Individual Carbon Nanotubes

CNF Project # 900-00
Principal Investigator: Paul L. McEuen

Abstract:
We have measured the capacitance of individual carbon nanotubes. The capacitance is measured as a function of the carrier density over a wide range including both electrons and holes. Our measurements detect the electrostatic part of the capacitance as well as the quantum corrections arising from the electronic density of states and electronic correlations. These measurements allow us to determine this central quantity of nanotube transistors as well as to study the fundamental physics of electrons in one dimension.

Summary:
The electronic capacitance of a one-dimensional system such as a carbon nanotube is a thermodynamic quantity that contains fundamental information about its quantum ground state. It is composed of an electrostatic component describing the interactions between electrons and their correlations, and a kinetic term given by the electronic density of states. In this work we have measured for the first time the capacitance of individual carbon nanotubes. We use these measurements to study the ground state of interacting electrons in one dimension.

The devices used in this study utilize a metallic top gate separated from a NT by a thin layer of oxide to form a capacitor. The NTs were grown using chemical vapor deposition from patterned pads of evaporated Fe on degenerately-doped silicon with a 200 nm SiO$_2$ top layer. We used a C$_6$H$_4$ / H$_2$ growth recipe, which we found to produce predominantly single-walled NTs with small diameter (1-4 nm). After growth, the NTs were contacted with source and drain electrodes made of 50 nm of palladium, followed by aligned deposition of 10 nm SiO, dielectric and a 50 nm aluminum top gate. This geometry allows us to perform transport as well as capacitance measurements on the same device.

Our measurements detect the geometrical part of the capacitance as well as the quantum corrections arising from the density of states and electronic correlations. The measured geometrical capacitances are consistent with the classical expression for the capacitance between a gate and a wire. The observed quantum corrections demonstrate a symmetric one-dimensional sub-band structure of electrons and holes and give the first thermodynamic measurement of the Luttinger parameter in the NT. Deviations of the measured capacitance from a simple theory points to the importance of electron correlations in the one-dimensional ground state.

References:
Capacitance Measurements of Individual Carbon Nanotubes

CNF Project # 900-00
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Figure 1: AFM image of a carbon nanotube capacitor device: An individual single-wall NT grown on conductive Si substrate with 200 nm SiO$_2$ cap layer is contacted with Pd contacts. An aluminum top gate forms a capacitor with the nanotube segment beneath it separated by 10 nm of SiO dielectric. This design allows to measure both transport and capacitance on the same device.

Figure 2: Measured capacitance of nanotube devices with different capacitor length. For each device we plot a single point representing its measured capacitance at $T = 77K$ and the length of the nanotube under the gate (the capacitor length) extracted from the AFM images. The two lines show the theoretically predicted capacitance for nanotubes with diameters $d = 1$ nm and $4$ nm.
Probing Electrostatic Potentials in Solution with Carbon Nanotube Transistors

CNF Project # 900-00
Principal Investigators: Paul L. McEuen, Hector Abruña

Abstract:
We have used single-walled carbon nanotube transistors to measure changes in the chemical potential of a solution due to redox-active transition metal complexes. The interaction of the molecules with a gold electrolyte-gate wire changes the electrostatic potential sensed by the nanotube, which in turn shifts the gate-voltage dependence of the nanotube conductance.

Summary:
Carbon nanotubes (NTs) are promising candidates for nanoscale electrochemistry experiments. Semiconducting NTs can be used as field-effect transistors (FETs) in an electrolyte environment [1], and their high sensitivity makes them excellent sensors of molecules in solution (e.g., [2]). In previous sensing experiments, the analyte caused a shift in gate-voltage dependence of the nanotube conductance, which has been attributed to charge transfer from adsorbed molecules to the nanotube. We have explored the response of single-walled NT FETs to redox-active transition metal complexes, and have found that the signal stems from the changing chemical potential of the solution and does not depend on the proximity of the molecules to the NT [3].

We fabricated CVD-grown NTs in a FET geometry at the CNF, with 10 µm between the gold source and drain electrodes. A 60 by 25 µm microfluidic PDMS channel was sealed over the NT, and a gold wire in a reservoir on the end of the channel was used to set the electrochemical potential of the solution [1]. We introduced different redox-active molecules into the reservoir and studied the electrical response of the NT FET.

We found that oxidizing molecules generally caused a positive shift in the NT threshold voltage, whereas reducing molecules caused a negative threshold voltage shift. We explored this more carefully by making simultaneous measurements with a traditional electrochemical cyclic voltammetry setup, and we found that the threshold voltage shift varied with the change in chemical potential of the solution, as determined by the Nernst equation. By controlling the flow in the channel, we also determined that the response depended only on whether the molecules were near the electrolyte-gate wire, and not on their proximity to the NT.

To understand these data, we recall that the voltage applied to the electrolyte-gate wire sets the electrochemical potential of the solution, which is the sum of the chemical potential (set by the molecules in solution) and the electrostatic potential. If the nanotube senses only the electrostatic potential, then the shift in threshold voltage will simply be the change in the chemical potential, which agrees with our data. The source of the signal is the electrochemical interaction between molecules and electrolyte-gate wire, and the proximity of the molecules to the NT is irrelevant, as we observed.

To confirm this model, we measured the nanotube conductance versus both the voltage on the electrolyte-gate wire, which sets the electrochemical potential, and the potential of a Ag/AgCl reference electrode, which measures only the electrostatic potential. As expected, when we added a redox-active molecule, the conductance shifted versus the gate voltage, but it remained unchanged versus the reference electrode potential. We therefore conclude that NT FETs can be used as nanoscale reference electrodes to measure the electrostatic potential of a solution, which could lead to new kinds of nanoscale electrochemistry experiments.

References:
Probing Electrostatic Potentials in Solution with Carbon Nanotube Transistors

CNF Project # 900-00
Principal Investigators: Paul L. McEuen, Hector Abruña
Users: Lisa Larrimore, Sudhasattwa Nad, Xinjian Zhou

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Figure 1, top left: Electrolyte-gated NT FET. The gate wire establishes the electrochemical potential of the solution, which is the sum of the chemical potential set by the molecules and the electrostatic potential.

Figure 2, bottom left: Threshold voltage shift varies with change in chemical potential (oxidizing molecules cause a positive shift, reducing cause a negative shift).

Figure 3, above: The NT measures only the changing electrostatic potential, acting as a nanoscale reference electrode.
Suspended Carbon Nanotubes for Transfer to Secondary Substrates

CNF Project # 900-00
Principal Investigator: Paul McEuen

Abstract:

We have grown suspended carbon nanotubes and carbon nanotube bundles spanning etched windows in Si wafers as wide as 100 µm. Au evaporation through shadow masks onto the growth chip allows optical resolution of the carbon nanotubes in the Au-coated regions, useful for subsequent manipulation during transfer onto other substrates.

Summary:

The exceptional electronic and mechanical properties of carbon nanotubes have been extensively investigated in a number of device configurations such as field effect transistors, quantum wires, and sensors. In general, these studies have been carried out on material grown or deposited onto an oxidized Si wafer. An interesting alternative is to use the ferroelectric field effect configuration, where the standard gate dielectric is replaced by a thin ferroelectric film. However, because many ferroelectric materials cannot withstand the elevated temperatures during the growth of carbon nanotubes, a method to transfer carbon nanotubes from a silicon based “growth chip” to the target ferroelectric sample is necessary.

We have followed the procedure developed by X.M.H. Huang et al. [1] for Si to Si transfers. Oxide and nitride layers were grown onto 400 µm thick Si wafers, and photolithographic patterning was used to wet-etch 1 mm x 10-100 µm windows through the wafers and define catalyst pads at the edges of these windows. Using an aqueous suspension of the catalyst developed by Kong et al. [2], we optimized the growth process to produce suspended carbon nanotubes, and carbon nanotube ropes spanning trenches as wide as 100 µm. We subsequently use commercial TEM grids as shadow masks, and evaporate Au onto the growth chips, allowing optical resolution of the carbon nanotubes in the Au-coated regions.

These suspended nanotubes can be transferred with high precision onto the ferroelectric oxide samples. The ability to access the nanotubes from both sides of the wafer, and over large regions, also makes these structures interesting for studies of the interaction of carbon nanotubes with fluids, when integrated into a microfluidic delivery system.

References:


Suspended Carbon Nanotubes for Transfer to Secondary Substrates

CNF Project # 900-00
Principal Investigator: Paul McEuen
Users: Patrycja Paruch, Samantha Roberts

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- Suspended carbon nanotubes and nanotube bundles as long as 100 µm have been grown across windows patterned in silicon wafers.
- Gold evaporation through simple shadow masks allows the suspended nanotubes to be optically resolved.
- These structure can be used to transfer the nanotubes to different substrates, and to investigate their interaction with fluids.

Figure 1: SEM of suspended carbon nanotubes.

Figure 2: Dark field micrograph of carbon nanotubes with Au-coated regions.
Carbon Nanomechanics

CNF Project # 900-00
Principal Investigator: Paul L. McEuen

Abstract:
Single walled carbon nanotubes (SWNT), rolled up graphene sheets, have been intensely studied in recent years due to their extraordinary electrical and mechanical properties. Our group has fabricated a SWNT electromechanical resonator, the smallest NEMS device to date [1]. Recent work has concentrated on understanding the losses in SWNT mechanical resonators, as well as looking at their low temperature behavior in the regime where single charging effects become important.

Summary:
Due to their very small mass ( ~ Attogram) and high Young’s modulus ( ~ TPa), SWNT resonators have the potential to be systems which are extremely sensitive to external perturbations, making them potentially very good sensors. We fabricated suspended doubly-clamped SWNTs in transistor geometry, to use as nanomechanical resonators. The gold source and drain electrodes were defined photolithographically and a conducting Si substrate was used as a gate. SWNTs were suspended between the source and drain electrodes, 500 nm above the gate. Mechanical vibrations were induced in the NT by applying a high frequency AC voltage relative to the gate [1]. We detected the SWNT’s motion by using the SWNT’s transistor properties and employing it as a mixer. The resonance frequencies of SWNT devices at room temperature were found to be in the MHz and could be tuned by varying the DC voltage of the back gate, and thus the tension in the NT.

Recent work has gone into studying the energy loss mechanisms in mechanical resonators. The quality factor, an inverse of the normalized width of the resonance peak, is an important measure of the loss. It also is a measure of the sensitivity of a resonator to external perturbations. At room temperature, the SWNT resonators displayed quality factors from 30 to 200, and exhibited a strong gate voltage dependence. At low temperatures, quality factors as high as 4000 were observed, with roughly a temperature dependence [2]. There are many possible sources of loss in the resonators. Losses could come due to extrinsic sources, such as clamping, residue from fabrication, ohmic joule heating in the contacts from electrons hopping on and off the SWNT, and out-of-phase coupling to external systems. The importance of the first three loss mechanisms was tested by changing the clamping geometry and the fabrication method, and correlating with the measured quality factors. No significant changes in quality factors were observed. Losses could also be due to intrinsic sources, for example, the thermoeelastic effect. This mechanism predicts the observed temperature dependence, but its applicability is still being investigated. At low temperatures, the SWNT resonators have the potential of being the most sensitive mass detection system to date.

As another experiment on a more fundamental science front, we have cooled the resonators down to liquid helium temperatures to investigate the coupling between mechanical vibration of the SWNT and single electron charging effects. At temperatures of 0.3K to 4.2K, the SWNT behaves as a quantum dot. The NT displays Coulomb oscillations in the conductance as a function of the gate voltage. We find that the mechanical resonances of the SWNT are influenced by the Coulomb oscillations. We observe a dip in the resonance frequency and in the quality factor as the gate voltage is swept through a Coulomb oscillation. We attribute these dips to the influence of the single electron charging events.

References:
Carbon Nanomechanics

CNF Project # 900-00
Principal Investigator: Paul L. McEuen
Users: Vera Sazonova, Arend van der Zande

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Figure 1, top left: A top view SEM image and pictorial side view of suspended SWNT devices.

Figure 2, bottom left: A plot of current measured through the SWNT as a function of AC driving frequency and DC gate voltage. The dip around $V_g = 0$ is the electronic mixing signal of the SWNT. The small parabolic curves are due to the motion of the nanotube.

Figure 3, above: A plot of the quality factor vs. temperature for many different SWNT resonators.
Carbon Nanotube Diodes and Transistors with Multiple-Gate Geometry

CNF Project # 900-00
Principal Investigator: Paul L. McEuen

Abstract:

Single-walled carbon nanotubes are configured into diodes and transistors with multiple gate geometry. Diodes are fabricated in a split-gate geometry with electron (N) and hole (P) regions separated by a central region that can be gated to be either P, N, or intrinsic. Nearly ideal diode behavior is observed when the central region is intrinsic: the current as a function voltage increases exponentially in forward bias. Carbon nanotube transistors are also fabricated with top gate geometry on high-resistivity silicon substrate. Devices are shown to be operated as microwave mixers up to 50 GHz.

Summary:

Individual semiconducting carbon nanotubes have been shown to make excellent channels for both P and N type field effect transistors (FETs). A P-N interface can be created within single carbon nanotubes through electrostatic gating [1]. By adapting split-gate geometry, we demonstrate the fabrication of nanotubes P-I-N diodes with nearly ideal diode behavior.

In addition, semiconducting carbon nanotubes have been shown to have room temperature carrier mobility exceeding 10000 cm²/Vs [2]. As a result, they offer promise as very high frequency transistors. We fabricate top-gated carbon nanotubes transistors on high-resistivity silicon substrate, and demonstrate that they can function as mixers up to 50 GHz [3].

Our carbon nanotubes diodes are fabricated on highly doped Si wafers with 50 nm of Mo as split-gates in the 100 nm PECVD silicon oxide layer (Figure 1 inset). A P-I-N diode can be formed with the split gates oppositely biased and the global back gate grounded. Figure 1 shows a typical I-V curve from a device at T = 1.5 K. The device is strongly rectifying, turning on at less than 1 V in the forward bias direction, but with no significant current under reverse bias until about -8 V. Temperature dependent experiments further reveal that the carbon nanotubes P-I-N diodes have an ideality factor of 1.2, suggesting a nearly ideal diode behavior. These results are crucial for evaluating the use of nanotubes for both bipolar and optoelectronic devices.

Our top-gated carbon nanotubes transistors are fabricated on intrinsic silicon substrate (resistivity 12-39 Kohm-cm) with 1 µm thermal oxide. 16 nm SiO₂ is evaporated on the top of nanotubes as dielectrics for the top gate. When operated as a mixer, the nonlinearity of G vs. V results in a DC mixing current proportional to dG/dV (Figure 2). Despite the attenuation due to the measurement circuit, even at 50 GHz, the mixing signal is nearly identical in shape to the signal at 10 MHz. This means that the nanotube device still operates as a mixer at 50 GHz. Further study of eliminating parasitic capacitances from the setup is currently undergoing in order to explore the intrinsic high-frequency limit of carbon nanotubes.

References:

Carbon Nanotube Diodes and Transistors with Multiple-Gate Geometry

CNF Project # 900-00
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Figure 1, above: Carbon nanotube P-I-N diode with split-gate device geometry.

Figure 2, right: Carbon nanotube top-gated transistor working as mixer up to 50 GHz.
Anomalous Magnetic Nanostructural Evolution in Annealed Cu$_{80}$Co$_{20}$ Granular Thin Films and Its Effect on Extraordinary Hall Effect

CNF Project # 1001-01
Principal Investigator: Jian-Qing Wang

Introduction:

Our research investigated the evolution of magnetic Co nanoparticles in annealed Cu$_{80}$Co$_{20}$ thin films deposited by magnetron-sputtering. SQUID measurements of as-deposited films showed typical single-peak magnetic susceptibility curve. Magnetic analyses by Curie-Weiss and Langevin curve fittings showed a uniform nanoparticles size of 3.0 nm in diameter [1]. In samples annealed up to 200°C, no significant changes were observed. At annealing temperature ($T_a$) of 300°C, two significant changes were observed: downshift in position of the ZFC peak to ~ 30 K accompanied an emergence of another peak around 300 K. These features, observed over a wide range of film thicknesses (40-200 nm), are consistent with the coexistence of two particle sizes in annealed samples where the first and second peak corresponds to the size of the smaller and the larger nanoparticles. From $\chi$-$T$ curves, the estimated size of the two particles are approximately 3.0 and 6.0 nm in diameter [2].

Summary:

The two particle-size distribution strongly implies coalescence is an unlikely mechanism for the formation and growth of Co nanoparticles in our annealed samples as evidenced by the virtual absence of intermediate-sized particles that results in the distinct two-peak structure in $\chi$-$T$ curves. Furthermore, the quasi-linear decrease in EHE resistivity with $T_a$ is inconsistent with much more dramatic decrease predicted by Berger’s theory for systems with a uniformly sized particle distribution [3]. This disagreement can be explained by asserting that annealed Cu-Co systems are comprised of two particle sizes where the most of the extraordinary Hall effect (EHE) contribution arises from quantum mechanical side-jump but dominant scattering events contributing to the EHE are those off the smaller Co particles.

Based on our magnetics data and with interfacial energy considerations, rather than uniform reduction in size upon annealing at lower $T_a$, we believe nanoparticles undergo random dissolution into the surrounding Cu matrix which would cause the population of the nanoparticles to decrease, thus weakening the dipole interactions within the film, causing the observed downshift in $T_b$. Concurrently, gradual smoothening of the Cu-Co boundary causes the initial decline of EHE due to reduced interfacial scattering. Upon reaching 300°C and beyond, there is a substantial reduction of smaller particle population. At this point, it becomes energetically favorable for the dissolved Co atoms in the surrounding Cu matrix to nucleate on to the surface of the existing smaller particles rather than on new sites. Further reduction of small particle population occurs at higher $T_a$’s. At 400°C it was estimated that less than 20% small particles still remain. This could explain about 90% reduction in EHE from 100°C to 400°C [4].

References:

Anomalous Magnetic Nanostructural Evolution in Annealed Cu$_{80}$Co$_{20}$ Granular Thin Films and Its Effect on Extraordinary Hall Effect

CNF Project # 1001-01
Principal Investigator: Jian-Qing Wang
Users: Jian-Qing Wang, Nam H. Kim
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Primary Funding: Research Corp., NSF
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- Both Cu & Co are of fcc structures in nanophase → Similar atomic numbers & lattice spacings render TEM and XRD analyses rather ineffective.
- Examined magnetron sputtered granular Cu$_{80}$Co$_{20}$ thin films from 40-200 nm in thickness for as-deposited as well as up to 400°C.
- Quasi-linear decrease in extraordinary Hall Effect resistivities supports two particle-size distribution of the Co nanoparticles in Cu$_{80}$Co$_{20}$ annealed at high temperatures.

Figure 1, top left: (a) TEM cross-section of a magnetron sputtered granular Cu-Co thin film on Si substrate. (b) XRD of the Cu-Co film.

Figure 3, bottom left: Hall resistivity curves for 40 nm thick Cu$_{80}$Co$_{20}$ series annealed at $T_a$; (a) $\rho_{xy}(H)$ for films annealed at 100°C and 400°C. (b) and at 300°C.
Fabrication of Nanoscale Sensors Using Divining Rod Carbon Nanotubes

CNF Project # 1192-04
Principal Investigator: Michael J. Naughton

Abstract:
This is a project to develop scanned probe sensors using branched nanostructures, such as Y-junction carbon nanotubes (YCNTs). The goal is to improve SPM sensing, including magnetic (i.e. MFM), by employing intrinsically nanoscale sensing elements. As the dimensions of these are smaller than the wavelength of light, conventional optical sensing is precluded. We’re using piezoresistance sensing, in “divining rod” geometries.

Summary:
Our branched YCNTs have a mostly non-crystalline structure. Previously, we fabricated devices using samples of diameter 20 to 50 nm, and several microns in length. The typical electrical resistance of such YCNTs is of the order of 100 kΩ/µm. The YCNTs are both theoretically estimated and experimentally proven to have a significant piezoresistive effect. However, we found that the ductility of our YCNTs is low, and they broke rather easily, such as via electrostatic shock or under AFM manipulation.

To address this, we report here another round of fabrication process using larger diameter YCNTs, ~ 100 nm, with lengths easily exceeding 10 µm. These are anticipated to be much stronger in structure than our previous samples, yet still have substantial piezoresistive effect, which means they could probably survive the shocks that previously broke the thin YCNTs, and live to tell us the story of nanoscale displacement sensing.

With that, we designed Cr/Au nano-wires placed via e-beam lithography (we used CNF’s Leica VB6) across two branches of the new Y-junction CNT, and make the 3rd branch suspended over a trench etched by BOE (see figure for the new sample fabricated). We’re presently working on measurements of these new YCNT samples, including magnetically-driven piezoresistivity.
Fabrication of Nanoscale Sensors Using Divining Rod Carbon Nanotubes

CNF Project # 1192-04
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Primary Funding: NSF NIRT Grant 0210533
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Figure 1: SEM picture of a Y-junction CNT (diameter ~ 100 nm) suspended above a trench ( ~ 400 nm deep, etched by BOE); the nanotube is contacted by 4 metal leads (50 nm Cr + 30 nm Au). Substrate underneath the structure is Si with 1 µm of thermally-grown silicon oxide. This structure will be used to sense nanoscale displacement via piezoresistive effect.
Squashed Carbon Nanotubes by Metal Contacts and Consequences for Contact Resistance

CNF Project # 1246-04
Principal Investigator: David Anthony Muller

Abstract:

We report the contact geometry between metal contacts and carbon nanotubes using electron tomography [1,2] which provides three-dimensional information at the spatial resolution of 2 nm [3]. Investigating the three-dimensional interfacial structure between the contacts and carbon nanotubes is important since the contact resistance, or the barrier height at the contact, can be changed greatly depending on the contact geometry [3-5]. We observe that high-surface-energy contacts, like gold, deform carbon nanotubes in the radial direction by creating a flat contact surface, and induce changes in the local energy gaps of the nanotubes. The changes in the local energy gaps of the deformed carbon nanotubes were probed with spatially resolved electron energy-loss spectroscopy (EELS) [6]. On the other hand, wetting contacts, like titanium, preserve the shape of nanotubes. For titanium contacts, a high Schottky barrier still remains which makes titanium not a desirable contact material.

Experimental Details:

Carbon nanotubes were dispersed in a solvent and the solution was sonicated to isolate individual nanotubes. A drop of the solution was applied to a transmission electron microscope (TEM) grid that is coated with a thin holey carbon film. Afterward, approximately 10 Å of Au, 15 Å of Au-Pd, 15 Å of Pd, or 25 Å of Ti were deposited by e-beam evaporation at 10^{-6} torr. The nanotubes were examined using a Tecnai F20 scanning electron transmission microscope (STEM) and a Fischione high-tilt holder. During electron tomography, the metal-deposited carbon nanotubes were tilted over ±70° with acquisition of projected images at 2° increments. Simultaneous iterative reconstruction technique (SIRT) [7] was used to reconstruct a three-dimensional representation of the metal-deposited nanotubes from the tilt series. The carbon K-edge of the carbon nanotubes were examined with EELS to probe the conduction band of the nanotubes modulated by the core hole effect.

Conclusions:

Investigating the three-dimensional interfacial structure between the metal contacts and the carbon nanotubes using electron tomography and spectroscopy has been successfully achieved. The experimental results show high-surface-energy contacts deform nanotubes in the radial direction to induce changes in the local energy bands of the nanotubes. Wetting contacts, on the other hand, preserve the shape of the nanotubes and eliminate the geometrical contribution to the contact resistance.

References:

Squashed Carbon Nanotubes by Metal Contacts and Consequences for Contact Resistance

CNF Project # 1246-04
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- Gold clusters facet on carbon nanotubes and deform the nanotubes. The contact surface between the cluster and the nanotube is flat.
- Carbon K-edge, which reflects the conduction band of the carbon nanotube modulated by a core hole effect, shows changes in the local energy bands in the deformed region of the nanotube.
- Wetting contacts preserve the shapes of carbon nanotubes.

Figure 1, above: Tomographically reconstructed gold clusters on a carbon nanotube shows flat contact surface.

Figure 3, below: Tomographically reconstructed titanium clusters on carbon nanotubes shows wetting contact preserves the shape of nanotubes.

Figure 2: Carbon K-edge from deformed region (gold cluster) of carbon nanotubes confirms changes in the local energy bands due to deformation.
Sub-Micrometer Sized Hall Sensors for High-Resolution Scanning Hall Microscopy

CNF Project # I294-04
Principal Investigator: Dr. Maxim Marchevsky

Abstract:

Scanning Hall Microscopy (SHM) is a powerful tool for studying fundamentals properties of magnetic and superconducting materials. Spatial resolution of the technique is defined by the lateral dimensions of the Hall sensor active area; the latter is about 1.5 square µm in our existing setup. The aim of this project is to fabricate Hall sensors of sub-micron dimensions; that would dramatically widen the application scope of Hall microscopy. Reduction of the sensor lateral dimensions to 0.3-0.5 µm should make it possible to image individual flux lines in superconductors up to magnetic field induction of ~ 80-100 Gauss and directly visualize interesting dynamic phenomena and structural transitions in the vortex lattices. Sub-micrometer sized sensors would allow to resolve dynamics of magnetic domain walls in ferromagnetic materials [1] down to nanometer scale. The high-resolution SHM technique has also a great promise for non-invasive magnetic imaging of nanostructures.

Research Summary:

First, we use photolithography to define a pattern of ~ 1.5 x 1.5 µm-sized Hall sensors and their current and voltage leads in the layer of photoresist on a square semi-insulating GaAs substrate wafer. Four independent Hall “crosses” are patterned in the center of the wafer. Next, a polycrystalline Bi film of the 100 nm thickness is deposited using RF magnetron sputtering. A lift-off process is done subsequently. Choice of the Bi sensor material is due to its low carrier density and long electron mean free path that contribute to its high Hall coefficient. In the second fabrication step, the active area of the Hall crosses is reduced to sub-micrometer dimensions using focused ion beam (FIB) etching. With FIB, we cut four ~ 0.1 µm wide trenches, inwards from the corners of the sensor active area. Occasionally, the trenches were further extended after an initial cut, with sensor resistance being checked in between the steps. Finally, the wafer is separated in four parts using an additional lithography-etching-cleaving procedure, leaving actual Hall crosses at ~ 7 µm distance from the etched corner of the wafer chip. In the actual scanning microscope, the sensor chip is installed on the top of the piezoscanner, facing sample surface at a ~ 2 degree angle. When the corner of the sensor chip comes into a mechanical contact with the sample, the actual fabricated Hall cross remains separated from the sample surface by ~ 0.2 µm gap.

The active area of the FIB milled Hall “crosses” is ~ 0.5 x 0.5 µm. Resistance of the sensors was measured as ~ 5-8 kΩ after the initial lift-off step and increased up to ~ 10-15 kΩ after the milling procedure. The room temperature Hall coefficient of the fabricated sub-micron sized Hall sensors is typically (2-5) x 10^-5 Ω/G. Further testing of the sensor performance at low temperature is required.

Summary:

Sub-micrometer sized bismuth Hall sensors have been fabricated using a combination of photolithography and focused ion beam milling. The devices show promise as magnetic nanosensors for scanning Hall microscopy application.

References:

Sub-Micrometer Sized Hall Sensors for High-Resolution Scanning Hall Microscopy

CNF Project # l294-04
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Figure 1: Four bismuth Hall sensor devices fabricated on a GaAs wafer. Active area of ~ 2 x 2 µm is formed using conventional photolithography.

Figure 2: Hall “crosses” patterned using focused ion beam milling. Active area of the devices is reduced to ~ 0.5 x 0.5 µm. Resistance of 10-15 kΩ at 300 K and Hall coefficient of 2-5 x 10⁻⁵ Ω/G.
Fabrication of Nanoscale Josephson Junctions for Quantum Coherent Superconducting Circuits

CNF Project # 1314-05
Principal Investigator: Britton L. T. Plourde

Abstract:
We are developing a process for producing quantum coherent superconducting circuits involving nanoscale Josephson junctions. Such circuits have shown great promise in recent years for explorations of quantum mechanics at the scale of circuits on a chip and for forming qubits, the elements of a quantum computer. The superconducting qubit, where the entire device has two fundamental basis states, can be manipulated with resonant radiation and placed in an arbitrary quantum superposition of the basis states. Success in this area requires a fabrication process for making nanoscale junctions reproducibly with an architecture which allows for the placement of many qubits on a chip.

Summary:
The unique properties of nanoscale Josephson junctions enable a wide range of novel superconducting circuits for investigations in many diverse areas. One such circuit, the superconducting flux qubit, has emerged as a promising candidate for the element of a quantum computer, due to the low intrinsic dissipation from the superconducting electrodes and the possibility of scaling to many such qubits on a chip [1,2]. This circuit consists of a thin-film superconducting loop interrupted by several Josephson tunnel junctions. When the applied magnetic flux is adjusted within a certain range and the temperature is below ~ 50 mK, these circuits have two states—all of the screening supercurrent flowing clockwise around the loop or counterclockwise. Resonant microwave radiation drives transitions between the two states and microwave pulses of well-defined lengths can be used to generate arbitrary superpositions of the two states. To engineer the energy level spacings in a convenient range, the junctions must have capacitances of only a few femtofarads, thus driving the junction sizes to be of the order of 100 nm x 100 nm. The sense of the circulating supercurrent can be measured with another superconducting device involving more Josephson junctions, a dc Superconducting QUantum Interference Device (SQUID).

Currently we are developing a process for patterning these circuits at the CNF and we are attempting to integrate the patterning of photolithographically-defined large-scale features with 100 nm-scale structures defined with electron-beam lithography to lead to scalable qubit architectures. This will allow us to work on improvements in the quantum coherence in these devices, evaluate approaches for generating entanglement between multiple qubits, and investigate other novel quantum coherent circuits.

The junctions are fabricated using the standard double-angle shadow evaporation technique [3], in which a resist bilayer of copolymer and PMMA is used to produce a narrow PMMA airbridge suspended above the substrate. Evaporation of aluminum from two different angles with an oxidation step in between forms a small Al-AlOx-Al tunnel junction from the deposition shadow of the airbridge. We have developed a process for defining these junctions on the JEOL9300 and we perform the aluminum evaporations in a dedicated chamber at Syracuse. We pattern the large-scale features using the Autostep 200 and reactive ion etching of Nb films and wet etching of Pd films. Measurements of these circuits will be performed in cryogenic systems at Syracuse University, including a custom dilution refrigerator which should reach a temperature of 20 mK, currently under construction in the investigator’s lab.

References:
Fabrication of Nanoscale Josephson Junctions for Quantum Coherent Superconducting Circuits

CNF Project # 1314-05
Principal Investigator: Britton L. T. Plourde
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Figure 1: The superconducting flux qubit, a possible element for a quantum computer, with two fundamental basis states—all of the screening supercurrent, $I_s$, flowing clockwise around the loop or counterclockwise.

Figure 2: SEM image of suspended PMMA airbridge. Image taken after double-angle aluminum evaporation, but before liftoff process.

Figure 3: SEM image of completed Al-AlO$_x$-Al tunnel junction. Future measurements to be performed at temperatures down to 20 mK.
Probing Quantum Coherent Vortex Dynamics in Superconductors with Nanoscale Weak-Pinning Channels

CNF Project # 1314-05
Principal Investigator: Britton L. T. Plourde

Abstract:
We are fabricating nanoscale superconducting structures for guiding the motion of vortices. Vortices are quantized bundles of magnetic flux which thread many different superconductors over a particular range of applied magnetic field. Such investigations may allow us to probe fundamental parameters of vortices in superconductors, including their effective mass and intrinsic damping. In addition, we will perform experiments to search for the quantum interference and tunneling of a vortex.

Summary:
For technological applications of superconductors in large magnetic fields, controlling the dynamics of magnetic flux vortices which penetrate the superconductors is important, as the motion of many vortices can cause unwanted dissipation. The addition of defects to the superconductor can pin the vortices in place and hinder their motion. In this case, each vortex can be treated as a classical particle interacting with a potential energy landscape generated by the pinning defects and the other vortices. However, at low temperatures, and in the absence of pinning and dissipation, a vortex can be described quantum mechanically and may exhibit phase coherence, allowing for the possibility of a vortex interfering with itself or tunneling between two different positions. Studies of such behavior of a collective excitation, such as a vortex, would guide fundamental understanding of quantum mechanics at large scales and could provide a new route for implementing a quantum computer.

Nanofabricated superconducting structures have been used previously for studying classical vortex dynamics. Much of the pioneering work was performed at Leiden University in the research group of Peter Kes [1] where devices were patterned with narrow thin-film channels with weak pinning surrounded by banks of a different superconductor with much stronger pinning. Such a structure allowed easy motion of vortices in the weak-pinning channels, while the vortices in the strong-pinning banks remained immobilized. Many different classical phenomena have been explored in this system, including recent studies of vortex mode locking [2]. In our research program, we are fabricating similar weak-pinning channels for guiding vortices around various paths at low temperatures to explore quantum coherent effects, including the possibility of vortex interference [3].

We fabricate our channels from bilayer films deposited at Leiden University. The lower weak-pinning film consists of an amorphous layer of NbGe, while the upper film is a 50 nm-thick layer of reactively-sputtered NbN which has strong pinning. We produce channels as narrow as 150 nm with electron-beam lithography and reactive ion etching using CF$_4$. Because of the high etch rate of the PMMA resist, we must use a relatively thick layer of PMMA, while still allowing for the definition of narrow structures. We pattern large-scale features into the films photolithographically. Future devices may include an insulating layer followed by a subsequent superconducting wiring layer for producing local voltage probes and gate electrodes. Measurements of these circuits will be performed in cryogenic systems which are currently under construction at Syracuse University.

References:
Probing Quantum Coherent Vortex Dynamics in Superconductors with Nanoscale Weak-Pinning Channels

CNF Project # 1314-05
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Figure 1: Weak-pinning channel fabrication process. (a) Deposition of weak-pinning amorphous NbGe layer (200 nm thick) followed by strong-pinning NbN layer (50 nm thick), (b) Channels etched into NbN with RIE. (c) Vortices in channels move easily, while vortices in NbN banks remain pinned in place, enabling future explorations of quantum coherent dynamics of vortices in a superconductor.

Figure 2: 150 nm-wide weak-pinning a-NbGe channel arranged in 1 µm-diameter ring geometry for vortex interference experiments.
Josephson Junction Quantum Computing

CNF Project # 1362-05
Principal Investigator: John Matthew Martinis

Abstract:

The laws of quantum physics provide intriguing possibilities for a tremendous increase in speed compared to classical computation [1]. Circuits made of superconductors and Josephson junctions are promising candidates for scalable quantum computation because of their compatibility with integrated-circuit fabrication technology [2-5]. The Josephson phase qubit [5] stands apart from other superconducting qubits because it does not require an optimal operating point [4]. Coupling of phase qubits is thus straightforward, allowing for multiple control methods. With recent improvements in coherence times and amplitudes, and the ability to measure both qubit states simultaneously, it is possible to use phase qubits to produce entangled states and measure them with high fidelity. We believe that demonstrations of quantum algorithms are also feasible.

Summary:

Josephson quantum bits (qubits) are constructed from superconducting integrated circuits. These “microwave atoms” can be thought of as non-linear microwave resonators formed from the non-linear inductance of a Josephson tunnel junction and its self-capacitance. Quantum behavior may be seen in these devices because of the extreme non-linearity of the Josephson junction—a single microwave photon has enough energy to significantly change the resonant frequency of the oscillator.

Single qubit logic operations are performed by injecting current pulses through the Josephson junction. Microwave pulses with a frequency resonant with the qubit quantum level spacing produces transitions between the quantum states, whereas quasi-DC pulses adjusts the quantum phase between the ground and excited states. The qubit state is measured by applying a strong pulse so that the excited state selectively tunnels to an external ground state. Once tunneled, the state can be easily distinguished by an on-chip superconducting quantum interference device (SQUID) amplifier.

A simple two-qubit logic gate may be constructed by simply coupling the two resonators with a capacitor [6]. This coupling produces a swap-type gate that, along with single qubit operations, has been shown to be a universal gate for quantum computation. This simple gate may be tested with a gate sequence that first places one of the qubits in its excited state, and then performs a swap operation such that the excited quantum state oscillates between the two qubits. Simultaneous measurements of both quantum states show that the occupation of the excited state is observed in only one qubit at a time (i.e. anti-correlated), as expected from quantum mechanics. It is also possible to apply a phase rotation to one of the qubits so that the oscillation halts; the measurements however remain anti-correlated.

Experiments are now under way to reduce the error rate of the gates by using improved materials and new circuit designs. We believe larger circuits can be constructed, possibly up to 5-10 qubits with current technology, enabling the demonstration of more complex quantum algorithms.

References:

Josephson Junction Quantum Computing

CNF Project # 1362-05
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- The goal of the project is to understand the basic physics for building a Josephson junction quantum computer.
- Initial experiments test simple quantum logic gates.

Figure 1, above: Photomicrograph of a device with two Josephson junction quantum bits.

Figure 2, right: Coherent operations on coupled phase qubits. (a) Sequence of operations. A 180° pulse is first applied to qubit B, populating the 01 state. Following a free evolution period in which the qubits interact, the state occupation probabilities are measured using a current pulse that induce selective tunneling of the 1 state. For data in (c) and (d), a 90° [180°] pulse is applied to qubit B after 16 ns. (b) Plot of measurement probabilities of the states 01, 1, and 11 as a function of the free evolution time. (c) Plot of measurement probabilities for a sequence that creates the eigenstate of the coupling Hamiltonian. After the eigenstate is formed by the 90° pulse, it ceases to evolve with time. (d) As in (c), but with an 180° pulse. Here, the phase of the oscillation changes by 180 degrees.
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