2006-2007
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Introduction to the
2006-2007 CNF Research Accomplishments

This year marks the 30th anniversary for
the Cornell NanoScale Science and
Technology Facility (CNF).

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 intensive staff support. In this mission, CNF has been singularly successful.

Our success is measured by the success our users experience as extensive staff and equipment resources strengthen their publications and products. Each year, over 700 new and returning users take advantage of CNF’s resources. In this publication, we summarize the range of work carried out in CNF in the past year. We believe that you will find this collection of work impressive, and more so because only a fraction of the projects can be 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, to serve 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 comprise facilities at Cornell, Stanford, Michigan, Georgia Tech, Washington, Penn State, UCSB, Minnesota, New Mexico, Texas, Harvard, and Howard, with NCSU as an affiliate. NNIN is managed from Cornell by 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 enhance our effective operation.

30th Anniversary Celebration

We celebrated our 30th anniversary by hosting the Future of Nanotechnology symposium, which was held on June 14th. A central aim of the symposium was to take a critical look into the shape of things to come. Renowned experts from around the world shared their vision of (i) the future of nanotechnology, (ii) the emerging field of nanomedicine, and (iii) the social and ethical issues associated with nanotechnology. Plenary lectures by Stanley Williams (HP), Tejal Desai (UCSF), and Sheila Jasanoff (Harvard) during the morning session introduced three parallel tracks on these three topics. Finally, a poster session gave an opportunity to our users to present their work. Over 400 people attended the symposium. The complete program with videos of the presentations can be found at http://www.cnf.cornell.edu/cnf_nanofutures.html.
New People
We are pleased to announce that David Botsch is our newest Computer Support Specialist. Born in Illinois, Dave grew up in South Carolina where he developed a taste for grits, barbecue, and Waffle House. Since receiving a Bachelor of Arts in Computer Science from Cornell University, he has resided in Ithaca. His previous jobs include working for the Savannah River Site in New Ellenton, SC; Hubbell Power Systems in Aiken, SC; Cornell Information Technologies; and the Cornell Center for Materials Research where he has worked on everything from remote camera applications to security and authentication infrastructures to large database applications. At CNF, one of his primary responsibilities is implementing CORAL, our new laboratory management system.

New Equipment
During the last year, CNF continued to upgrade its equipment resource base in Duffield Hall with a number of acquisitions, both expanding our capabilities and replacing older instruments to maintain the base capabilities of the facility.

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. With these new additions, we currently have a total of 56 nodes in support of nanoscale science calculations.

This year the Cornell NanoScale Facility also purchased two licenses for the Atomistix Toolkit from Atomistix for use on the CNF cluster. This density functional code provides users with the ability to model electronic transport through nanotubes, molecular junctions, and other nanoscale structures. This code adds to the suite of over 15 nanoscale simulation tools already available on the CNF cluster.

In the fall of 2006, CNF acquired a materials ink jet printer from Dimatix. This simple device uses inexpensive disposable “ink” cartridges to print materials on wafers, glass, rigid plastic, and even flexible materials. The “inks” can be custom formulations of organic chemicals, nanoparticles, nanotubes, of suspensions of metallic or semiconductor particles or precursors. The minimum feature size is approximately 25 µm. This tool opens a variety of new avenues for processing, including the fabrication of organic electronics and bio sensors.

To enhance its capabilities in nanostructure characterization, CNF obtained a set of four Zyvex nanomanipulators. These mechanical probes can be positioned, in vacuum, with a resolution of 1 nm. They are placed inside our Zeiss Ultra 55 SEM, allowing sample probing with the Zyvex manipulators while imaging with the SEM. They can be used for either electrical probing or mechanical manipulation, and can thus be used for electrical probing of nanostructures such as nanotubes, or for mechanical actuation of MEMS devices under test.

To continue our upgrading of our photolithography capabilities, we replaced an ailing Suss contact aligner with a new 200 mm capable contact aligner from ABM. We are continuing to look for opportunities to upgrade some of our existing heavily used equipment in this area with newer, more capable systems.

During the Spring of 2007, we initiated a joint development agreement with Oxford Instruments related atomic layer deposition. Our Oxford FlexAL plasma assisted ALD tool arrived in August. CNF staff, users, and Cornell faculty and students will be working with Oxford process engineers to develop new processes and implement existing processes of interest to CNF on this new ALD tool.

For more than one year, CNF has been evaluating the available next-generation instruments for deep silicon etching via the “Bosch™” process. An extensive designed experiment was conducted to evaluate the performance and process window of the available commercial tools. As a result of this exercise, CNF has placed an order for an Oerlikon VERSALINE deep silicon etch tool. This continues our relationship with Oerlikon, and its predecessors PlasmaTherm and Unaxis, a relationship which has been ongoing for almost our entire 30 years.

In July, we were informed that we were funded by NSF through a special equipment proposal to purchase and install a system for the growth of carbon nanotubes. We have specified a system that will grow single walled nanotubes on 100 mm wafers. We will be working with Prof. Paul McEuen’s group in Applied Physics to develop processes on this instrument. This will be the first such open facility for the growth of nanotubes within NNIN. We expect there to be considerable demand.
Broader Impacts

Here at the CNF, we conduct and participate in numerous educational outreach activities, both alone and as part of NNIN. One network-wide program is the Research Experience for Undergraduates Program (NNIN REU). During the summer of 2007, 69 undergraduate students participated in the 10-week NNIN REU internship program of focused research in micro and nanotechnology. Eleven of those interns spent their summer in Ithaca, working with Cornell faculty, graduate students and CNF staff.

Nanotechnology is extremely popular with the undergraduate population, evident in the fact that NNIN received over 600 applications and many of the interns noted that ours was the only program they applied to. The research accomplishments can be found at the NNIN REU web site, http://www.nnin.org/nnin_reu.html. In reading through the reports, you will find their research was of exceptional quality; reflecting the breadth of disciplines and providing a sound research introduction to many who might not otherwise experience our unique capabilities.

2007 was our eleventh summer of network-wide undergraduate research programming, which we believe makes a significant contribution to the national human resource pool in nanotechnology. The program is a proven turning-point in many an undergraduate career path, with a good percentage of our participants returning to an NNIN site for graduate studies in science and engineering.

We are grateful to the National Science Foundation for their continued funding support, and we seek corporate funds to augment our program. For instance, Intel Foundation kindly supported five of the CNF REU interns, along with three interns each at Stanford and University of Michigan. Intel’s sponsorship means a great deal to our program’s success, and of course, had a major positive impact on those eleven interns! Please contact Dr. Lynn Rathbun, NNIN Program Manager, to discuss your corporate sponsorship.

CNF continues to offer a number of workshops and special events. In April 2007, 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.

The second annual CNF Fall Workshop, “Building Nanostructures Bit By Bit” was held in October 2006. This workshop included discussions on 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. This workshop also provided participants with a rare chance to meet key code developers in the nanoscale simulation. Steve Plimpton of Sandia National Laboratories was on hand to discuss the popular molecular dynamics code, LAMMPS. In addition, Steven Johnson (MIT), developer of MEEP and MPB, provided two lectures focused on nanophotonics.

Planning is currently underway for the third Annual CNF Fall Workshop. Titled “Defining the Interface between Nano-science and Geology,” this event will be held in November 2007, and will explore nanoscale simulation techniques that can help propel the next generation of geologic research. Leading geologists will discuss current simulation needs in earth science. Sessions will cover density functional approaches to model crystal structures under high pressure deep within the Earth. Water interactions with minerals in nanoscale pores and grain boundaries will also be discussed. We will also explore molecular dynamic techniques to elucidate the process of biomineralization. Similar to the past two workshops, afternoon hands-on sessions will allow participants to work directly with these new simulation tools. Details on the upcoming workshop are available on the CNF website.

Use of the CNF computational cluster has increased dramatically as a result of these workshops and the CNF cluster currently provides computing time to over 60 users. In addition, twelve...
publications have resulted from work on the cluster, including one in Nature Physics and two Physical Review Letters.

The CNF has offered the CNF Short Course: Technology & Characterization at the Nanoscale (CNF TCN) for three years now. The short course is offered at the beginning of each summer, for new students and users. Since 2007, the course includes not only lectures and demonstrations in the clean room, but also a hands-on lab activities. 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. (http://www.cnf.cornell.edu/cnf5_courses.html)

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, we have completed the NNIN SEI portal. This new portal is meant to serve as an entry point to the social and ethical issues of nanotechnology. It offers information on upcoming events, publications, research findings by members of the NNIN network, as well as a multimedia archive on past events (both local and out-of-network). More importantly, and a substantial improvement over the previous site, it possesses an intra-net feature that allows all NNIN members to directly add resources to the site. Finally, it also offers users the possibility of subscribing to an email list that will keep them up-to-date with the site’s content.

Continuing with our outreach efforts we have organized and helped sponsor a number of lectures featuring both local and international speakers. These talks cater to both technical and non-technical audiences but, contrary to past years and in an effort to go past the introductory level, these talks are more specialized in scope than past talks.

We were actively involved in the organization of the SEI stream of CNF’s 30th anniversary celebration. Again, here we tried to provide the audience not only with general talks, but also with more focused examinations of specific issues pertaining to nanotechnology. The SEI talks were extremely well attended, and the panel discussion that followed provided the audience with a forum to share and explore their concerns with speakers.

We are currently working with the Center for Nano-technology in Society at the Arizona University State to produce a training package that will be administered to all new NNIN users. This multimedia package is designed to address basic issues, but also to provide more in-depth information for those who are interested. CNF is taking the lead on this project and the training package will be tested here prior to December of this year.

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

On behalf of our staff, we present to you the 2006-2007 CNF Research Accomplishments. As always, we welcome your comments about CNF and its operations, as well as suggestions for improvements.

George Malliaras
Lester B. Knight Director

Donald Tennant
Director of Operations


“All-optical logic based on silicon micro-ring resonators”; Q. Xu, and M. Lipson, Optics Express, Vol. 15, No. 3, 924, 02 Feb. 2007.


“Ballistic deflection transistor and ballistic transistor deflection based logic circuits”; Q. Diduck, patent will be filed by July 24, 2007.


“Biomimetics-Smart Acoustic Microsensor”; R. Miles, Fall Mtg, NYS of the National Academy of Sciences, 104 (26) 10786-10791 (2007).

“Biomimetics-Smart Acoustic Microsensor”; R. Miles, Fall Mtg, NYS of the National Academy of Sciences, 104 (26) 10786-10791 (2007).


“Can We Scanned Probe Microscopy to Measure Local Carrier Mobility?”; S. Yazdianzadeh, March Meeting of the American Physical Society, Denver, Colorado, March 5-9, 2007.


“CMOS-Integrable Semiconductor Nanowires for Specific Label-Free Detection”; M. Reed, AHW 2006, Kohala, HI, 4 December 2006.


“Compact electro-optic modulator on silicon-on-insulator substrates using cavities with ultra-small modal volumes”; B. Schmidt, Q. Xu, J. Shakya, and M. Lipson, 3rd International Conference on Group IV Photonics, Ottawa, ON, Canada, September 2006; LEOS Annual Meeting, Montreal, QC, Canada, October 2006.


“Compaction and fatigue and transport in conjugated polymers”; G.G. Malliaras; APS March Meeting, Denver, CO, Mar. 5-9, 2007; European MRS, Strasbourg, France, May 28-June 1, 2007.


“Evaluating the roles of pili in twitching and long distance movement of Xylella fastidiosa in grape xylem and in the colonization of sharpshooter forug”; L. De La Fuente, T.J. Burr, and H.C. Hoch, Pierce’s Disease Research Symposium, San Diego, CA, 2006.


“Graphite fiber based MEMS”; S. Desai, Student Paper Competition, NTC forum, March 2006, Hilton Head, SC.


“Growth and characterization of Al_{x}Si_{y}N_{z} thin films”; R.J. Brown, and J.R. Shealy, TMS 2007, State College, PA (2007).

“Growth and characterization of Al_{x}Si_{y}N_{z} thin films”; R.J. Brown, and J.R. Shealy, to be published (2007).


“High Throughput Combinatorial Study of Local Stress in Thin Film Composition Spreads”; N.C. Woo, B.G. Ng, and R.B. van Dover, Review of Scientific Instruments, 78, 072208 (July 2007)

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CNF
Research Accomplishments
2006-2007
Nanoscale Biological Physics

CNF Project # 398-91

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Abstract

We worked on four major projects this past year. (1) Evolution and Adaptation in Microfabricated Habitat Patches; (2) Detection of H₂ Production by Photosynthetic Algae; (3) Elongation and Mapping of DNA in Nanochannels; and (4) Time-Resolved Protein Dynamics using Diffusional Mixing.

Summary of Research

(1) Evolution and Adaptation in Microfabricated Habitat Patches. This project aims at testing the foundations of our understanding of evolution and adaptation in the microbiological world. Our premise is that by designing micro-environments using nanofabrication to control resource flow and by limiting the movement of organisms between islands of communities, we can simulate a complex environment, even, if you will, a collection of countries with time. Figure 1 shows a schematic of a microhabitat patch we made at CNF. These experiments have shown the rapid adaptation that bacteria can achieve under stress, as opposed to non-stress conditions [1].

Figure 1: One dimensional array of microhabitat patches.

(2) Detection of H₂ Production by Photosynthetic Algae. This project is connected to efforts to develop high-efficiency hydrogen gas producing photosynthetic organisms to provide biosynthetic fuel. It uses the technology developed in our Evo-Devo project above. Figure 2 shows growth of the photosynthetic single-celled algae Chlamydomonas reinhardtii in our stress-chips. The main push now is on on-chip H₂ gas detection using sensor development.

Figure 2: 77 well coupled microhabitat patch array for Chlamydomonas reinhardtii. Cells are growing in the array.
(3) Elongation and Mapping of DNA in Nanochannels. This project is aimed at mapping and ultimately sequencing single DNA molecules of genomic length (1 million bases or longer) [2]. We are using a combination of e-beam, nanoimprinting, and conformal coating techniques to create sealing nanochannels of widths under 20 nm to elongate genomic length molecules, and we are also working on the development of electronic detection techniques of DNA molecules in the nanochannels. Figure 3 shows a self-sealed nanochannel with electrode array for electronic DNA detection.

(4) Time-Resolved Protein Dynamics using Diffusional Mixing. We are exploiting the zero-mode optical waveguide technology developed by Prof. Craighead at Cornell to do time-resolved protein dynamics [3]. The idea is to combine our diffusional mixer, which can “mix” proteins at any concentration in microsecond time scales [4], with zero-mode waveguides which can sample at the single molecule level dynamics of protein conformations. The zero-mode waveguides act as hydrodynamic shields from the rapid flow and allow us to work with single molecules at high concentrations. Figure 4 shows a zero-mode waveguide experiment using fluorescence correlation spectroscopy.

References


DNA in Nanofluidic Channels

CNF Project # 551-95
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Abstract

We have fabricated and used nanofluidic slits to investigate surface interactions during electrophoresis between deoxyribonucleic acid (DNA) molecules and channel walls. The channels have vertical dimensions of 19 nm and 70 nm, and contain no sieving matrix. Size-dependent mobility is observed for DNA in the 19 nm channels. These results suggest that the notion of free-solution DNA electrophoresis breaks down due to surface interactions in nanoscale environments.

Summary of Research

Nanometer-scale fluidic channels are seeing increasing use in studies of polymer physics. Additionally, small-scale devices are finding more utility in bioanalytical applications such as DNA separation and quantitation. In a recent two-article report by Pennathur and Santiago, results showed that the zeta potential in nanofluidic channels decays over a sufficiently large fraction of the channel width to cause a parabolic velocity profile across the nanochannel for molecules of different valence [1]. Examples of biophysical studies in nanofluidic systems include: DNA confinement [2, 3], DNA compression [4], DNA relaxation [5, 6], and DNA-protein interactions [7, 8].

In this research project, we fabricate devices to test the electrophoretic mobility of DNA molecules in slit-like nanochannels. Length-dependent mobility is observed, even though the nanochannels contain no obstacles or sieving matrix. In the absence of surface friction, these channels would be a free solution environment in which DNA molecules would move with a length-independent mobility [9]. As length-dependent mobility is observed, however, we conclude that molecular interactions with the confining walls are significant, and the notion of free solution electrophoresis breaks down.

Figure 1 shows a schematic layout of the device used to test DNA-surface interactions as well as a proposed configuration of DNA in the loading channel and the nanochannel. DNA are “squished” into a pancake-like state within the 19 nm channels and must continuously interact with the surfaces.

Figure 2 shows the result of various separation experiments performed using DNA of lengths between 2 kb and 10 kb. DNA lengths are noted next to each curve. Note that the separation takes about 5 minutes, which is much faster than a typical gel electrophoresis experiment.

Figure 3 shows a control experiment in which 2 kb and 10 kb DNA molecules were driven through a 70 nm deep nanochannel. No separation of the 2 kb and 10 kb
DNA is observed, indicating that the channel height is a significant factor in the separation of DNA molecules in nanochannels. Note that the sharp spikes in the 100 V curves are indicative of transiently stuck DNA molecules that came unstuck during the course of the experiment and are not the primary DNA of interest in the experiment.

References

Electrospun Deoxyribonucleic Acid Fibers

CNF Project # 599-96
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Abstract
Using an electrospinning process, we have deposited nanofibers of deoxyribonucleic acid (DNA) over prepatterned features, allowing future measurement and manipulation of the fibers. Scanning electron microscopy (SEM) imaging (Figure 1) and atomic force microscopy (AFM) imaging (Figure 2) of the fibers indicate that the fiber diameters are sub 50-nm. Because the DNA is fluorescently tagged, the fibers are visible when observed with a fluorescence microscope (Figure 3). Using an AFM mounted on a fluorescence microscope, we have measured mechanical properties of a single suspended DNA fiber by depressing it with an AFM probe (Figure 4).

Summary of Research
Electrospinning is a technique to produce micro- and nanofibers from a wide variety of materials using an electrically forced fluid jet. In a typical electrospinning system, a droplet of polymer dissolved in solvent is placed on a sharp conducting tip, such as a needle, which is held at a high voltage with respect to a grounded collecting substrate. The droplet will form a cone from which a fluid jet is expelled. This jet will solidify in flight due to solvent evaporation and form a fiber, which is deposited on the grounded surface. The fluid jet can exhibit a whipping instability, resulting in deposition of a non-woven mat of randomly oriented fibers. Several techniques have been developed to controllably deposit fibers [1, 2], and there have been many studies demonstrating devices based on single fibers [3-7] as well as lithography using single fiber masks [8].

DNA is a polymer that has received a great deal of attention due to its ability to code the information necessary for life. Not only is DNA studied in order to better understand both the genomic language and the polymeric behavior of the molecule in living systems, but recently, DNA has also been used as a building block in artificially designed systems. Advances in programming DNA to assume desired shapes [9] and perform specific functions show great promise for harnessing the ability of this polymer. As such artificially designed structures become more prevalent, there is a greater need to understand the properties of DNA at the single molecule, several molecule, and bulk levels.

Like most polymers, DNA can be electrospun to make nanofibers [10]. We have electrospun fluorescently labeled DNA to produce nanofibers as small as 26 nm in diameter (Figures 1-3). These fibers were deposited over prepatterned features such as electrodes and trenches, allowing for future study and manipulation of the fibers. As an example of such an experiment, we have measured the Young’s modulus of a single DNA nanofiber using an atomic force microscope to depress a suspended region [11]. From the force-displacement data (Figure 4), we calculate a Young’s modulus of 15 ± 2 GPa.

References


Figure 1: SEM image of single suspended DNA nanofiber.

Figure 2: AFM image of single DNA nanofiber.

Figure 3: Fluorescence microscopy image of suspended DNA nanofiber.

Figure 4: Force-displacement plot from a single fiber measured using an AFM probe.
Zero-Mode-Waveguides Used in Cell and Surface Interactions

CNF Project # 599-96

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Abstract

Zero-mode-waveguides (ZMWs) are subwavelength apertures on metal films. They are fabricated at the CNF with a lift-off process using electron-beam lithography (Figure 1). We further process these structures for biological studies by topping the surface with polyethylene glycol, and selectively coating the inside of the ZMWs with 2,4-dinitrophenyl (DNP) functionalized polymer. Using fluorescence correlation spectroscopy (FCS), we show selective binding of Immunoglobulin G (IgG) to functionalized ZMWs.

Summary of Research

Due to their optical properties, ZMWs have been used for single molecule fluorescence correlation spectroscopy at micromolar concentration and microsecond temporal resolution [1]. Because ZMWs confine fluorescence excitation to zeptoliter volumes, they have been used to observe cell membrane events with high spatial resolution [2]. In this study, we have focused on selectively functionalizing ZMWs.

We have fabricated ZMWs in metal films capped with a thin layer of SiO₂. The surface surrounding the ZMW openings was first passivated through a polyethylene glycol silane coating. Then, the ZMWs were selectively functionalized by attaching polymer brushes to the glass bottom surface of the waveguides (Figure 2). Such polymer brushes can be derivatized to contain functional groups of interest.

In this study, we used 2,4-Dinitrophenol (DNP) moieties anchored to the polymer brushes. We show the selective functionalization of the ZMWs by incubating with fluorescently-labeled antibodies that bind to the DNP antigens. Through FCS, we show that the fluorescent antibodies bind to and remain attached to the DNP groups, whereas they diffuse freely in the absence of the target. Since it has been shown that cells can actively investigate nano-scale apertures [3], we propose that this approach can be used to deliver targeted stimulation to immune cells, and trigger membrane-associated signaling events which can then be studied in ZMWs with high temporal and spatial resolution.
We tested our devices with 1 μM dUTP (2'-deoxyuridine 5’-triphosphate) as control, and 0.5 μM IgG for specific binding, both labeled with Alexa-488 fluorophore. Using FCS, we look for specific interactions between ligands and binding agents.

As shown in Figure 3, first we note that protein IgG has a longer residence time in the excitation volume of the waveguides, indicating its slower mobility, compared to that of dUTP. Secondly we see a long time constant for the IgG in DNP-functionalized waveguides, comparing to that of non-DNP-functionalized waveguides, suggesting specific interaction between DNP ligands and IgG.

References


Patterned Biofunctional Polymer Brushes to Guide Neuron Growth

CNF Project # 640-97
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Abstract

Poly(D-lysine) (PDL) was attached to patterned poly(acrylic acid) brushes on a silicon or glass surface, which was used to guide neuron growth. 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) (PAA) 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. Here, we immobilized PDL onto PAA brushes and the surrounding PEG helped to restrict cells to grow only on PDL regions. The PDL/PEG hybrid surface was used to guide hippocampus neuron growth.

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 such patterned surfaces have been used to study cell-surface interactions [3]. Most of the patterned substrates are modified with self-assembled monolayers. Recently, polymer brushes have attracted a great deal of interest for 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 a background on silicon wafers. First, PEG was vapor-deposited onto a silicon wafer which had been cleaned using an oxygen plasma cleaner. Subsequently a photoresist was spincoated onto the wafer and patterned using photolithography. The unprotected 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-hydroxysuccinimide (NHS)
and then reacted with amino groups on target biological molecules [5]. Here, PDL was immobilized onto PAA brushes and then the PDL/PEG hybrid surface was used to guide hippocampus neurons to grow. AFM was used to examine the patterned surface. Fluorescence microscopy was used to study the neuron growth process. The neuron processes were well defined in the PDL patterned regions.

References


Retinal Implant Project

CNF Project # 657-97

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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 of Research

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.

To date, the CNF-fabricated components of this system have been various test structures and tools used in the research effort, the flexible circuit on which the remaining components of our prosthesis are assembled, and the stimulating electrode arrays themselves. Silicon (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 on to 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 number of major scientific challenges, however, still remain in producing visible perceptions at very low

Figure 1: Intra-ocular components of chronically implantable prosthesis assembled on flexible circuit.
threshold currents 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. Toward this end, as shown in Figure 2, 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. Toward this end, we have successfully used these devices in vitro to make single unit recordings from retinal ganglion cells.

A second effort we have undertaken is to reduce the threshold current necessary for visual perception by developing an electrode interface that is substantially elevated above the planar surface of the flexible polymer-based electrode array and penetrates into the retina. Placement of the planar electrode array in the sub-retinal space will locate the elevated electrode surface in the central region of the retina nearer to the ganglion cells being targeted for stimulation. The effort has thus far been approached with an SU-8 based high-aspect ratio post structure for which we have done initial feasibility fabrication process development as shown in Figure 3. The results look promising but the final fully integrated fabrication process is still under development.

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

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References

Abstract

We are constructing a new generation of rapid mixers to study the reactions involving biological macromolecules. Prototypes will be built and tested to address concerns about mixing efficiency and flow homogeneity.

Summary of Research

This project focuses on the continued development of microfluidic mixers for use with a variety of experimental probes of macromolecular interactions. In the past we have used both x-ray scattering and fluorescence microscopy to study protein and ribonucleic acid (RNA) folding in addition to protein conformational dynamics [1-3]. Our past experimental work employed CNF-fabricated mixers that relied on hydrodynamic focusing to achieve rapid mixing. Several generations of focusing mixers were built, characterized and used for experiments with biomolecules.

In these mixers, the large macromolecules were focused into an ~ micron-wide jet that flowed down the center of a wider channel. This jet was flanked on either side by buffer containing a small molecule; the rapid diffusion of these small molecules across the focused jet triggered the conformational change of interest. Since the mixing time scales with the squared width of the jet, the mixer operates most efficiently when thin jets are employed. Thin jets are highly compatible with laser microscopy, where micron sized focal or detection volumes are easily achieved [2]; however, use in conjunction with probes, such as x-rays, is challenging because the small size of the jet limits signal strength. If we increase the width of the jet, the mixing time increases dramatically.

This year, we have initiated a project to construct a three channel or ‘T’ mixer that operates by turbulent mixing, based on designs from other groups [4]. Larger probe beams will easily interface with these mixers, without the loss of signal to noise. Due to concerns about the uniformity of mixing, and, more specifically, the transition from laminar to full turbulent flow regimes, we are constructing prototype mixers with varying dimensions for characterization. Mixers will be fabricated and tested, to ensure that flow conditions are optimized for experiments.

To date, three port, or ‘T’ mixers have been successfully fabricated by imprinting zeonor [5] from a fabricated silicon master. One such device is shown in Figure 1. The devices must be well-sealed to avoid leakage at the high pressures involved in creating turbulent flow within the channels. This turbulent flow is required to achieve rapid and uniform mixing of solutions. Mixer characterization is in progress.

References

Figure 1: A photograph of the ‘T’ mixer being fabricated.
Microfluidic Cell Culture Analog Devices to Mimic Animal Exposures to Toxins and Drugs

CNF Project # 731-98
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Abstract
Our group has developed microfluidic in vitro devices to mimic the response of humans or animals to drugs, toxins, or nanoparticles. Each device, or cell culture analog (CCA), contains an array of pseudo tissues that are interconnected by microfluidic channels [1]. The recirculation of blood surrogate through the microchannels allows us to study tissue-tissue interactions, such as the breakdown of a parent compound in the liver and subsequent transport and reaction in the lung. We then combine these in vitro device experiments with physiologically-based pharmacokinetic model simulations to predict toxin and drug dynamics in humans.

Summary of Results
GI Tract Models: Our group has developed an in vitro cell culture model of the gastrointestinal tract that includes digestion, physiologically realistic cell populations, and a mucus layer. The current applications of this model include predicting iron bioavailability, studying the effects of oral exposure to nanoparticles, and measuring the metabolism and toxicity of ingested chemicals or drugs. Co-cultures of absorptive and mucus-producing cells were incorporated into an existing in vitro digestion/cell culture model used to assess iron bioavailability. The addition of goblet-type cells to this model provides more accurate iron bioavailability predictions, and may allow for more direct in vivo-in vitro correlations. Carboxylated polystyrene 50 nm and 200 nm particles were found to interfere with cellular iron transport, indicating that oral exposure to nanoparticles can have an effect on the normal physiological functions of intestinal epithelial cells. We have also developed a microscale cell culture analog (μCCA) of the GI tract that, together with a multi-chamber “body” μCCA, has been used to re-create the toxic effects of acetaminophen. Preliminary results show that the GI tract μCCA acts as a barrier to drugs entering the body μCCA, mimicking in vivo function in this regard.

Chemotherapy and Cancer: We completed work on a system to test potential synergy of doxorubicin, a chemotherapeutic agent, with multidrug resistance suppressing compounds, cyclosporin and nicardipine. This combination, in the in vitro system, repressed the multidrug resistant cancer cell line effectively without increased toxicity to other tissue. Techniques to measure real time fluorescence in such devices to monitor gene expression or cell viability have been developed [2]. We have built a CCA with 3D cultures of multiple cell types in a single microfluidic device (see Figure 1). Colon cancer was chosen as the model system, and the fabricated device consists of separate chambers representing liver, colon cancer, and bone marrow. In each chamber, cell lines representing each organ were embedded in a 3-D matrix of Matrigel, and media was supplied to each chamber by perfusion. The cells in the device were viable and proliferated for 3 days,

Figure 1: A microfabricated CCA to test for colon cancer treatments.
after which they started to die due to nutrient depletion and waste accumulation in the recirculating medium.

We tested the effect of a cancer drug, Tegafur, with our device. Tegafur is an oral pro-drug of 5-FU, which is a widely used cancer drug for colon cancer. Tegafur itself is not cytotoxic and it needs to be converted to 5-FU in the liver to become active; with a conventional \textit{in vitro} experiment such as 96-well plate experiment, one would not be able to observe the cytotoxic effect of Tegafur. In a 3-day experiment with our device, the cytotoxic effect of Tegafur was evident. To verify that the conversion of the drug in the “liver” was responsible for the cell death, the same experiment was run with the absence of liver cells. Without the presence of liver cells, Tegafur did not show any cytotoxic effect on tumor cells.

Cancer Metasis: Using a xenon difluoride technique, we have fabricated in silicon an array of defined semicircular channels that mimic the microcirculation (Figure 2). These channels provide the basis for studies on cancer cell metastasis by studying interaction of suspended cancer cells with endothelial cells lining the microvasculature [3].

Endocrine Disruptors: The CCA device was designed for a long-term low-dose toxicology study of environmental endocrine disrupting chemicals. One MCF7 breast cancer cell line and one Ishikawa endometrial cell line were constructed with estrogen stimulated green fluorescence protein (GFP) expression. GFP intensity increases in both cell lines when exposed to estrogen or estrogenic chemicals (Figure 3). Synergistic effects were tested among several different estrogenic pesticides. No obvious synergic effects were found. We have characterized the effects of different CCA surface treatments, including collagen, fibronectine, poly-D-lysine coating, and silicone oxide and silicon nitride deposition, on GFP responses of the constructed cell lines. Poly-D-lysine was found to cause aggregations in MCF7 cells which lead to false GFP signal, while collagen was found to diminish this effect. In microfluidic devices, hydrophobic chemicals may absorb onto hydrophobic surfaces which greatly decreases the bioavailability of such chemicals. Cyclodextrin-encapsulation of chemicals, surface protein coating and multi-ionic layer coating have been investigated to minimize biomolecular adsorption, with multi-ionic layer coating showing promising results.

Biosensors: We have worked on biosensors using surface plasmon resonance to measure virus concentration [4, 5].

Hydrogel Systems: We have worked with Dr. Mingming Wu on a microfluidic device to study chemotaxis [6]. With Dr. William Shain’s group, we have worked on hydrogel cultures as models to study tissue impedance around microfabricated neural probes [7].

References

Submicrometer Fluidic Channels for Studying Cancer Gene Expression

CNF Project # 762-99

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Abstract

Gene expression is a dynamic process involving messenger ribonucleic acid (mRNA) quantities that can vary from tens to millions. In this work, single molecule quantification [1, 2] of a sample’s genetic content is performed in a fluidic channel with submicrometer (500 nm by 250 nm) cross-sectional dimensions. Molecular beacon (MB) probes are utilized to increase robustness and selectivity in detection via a selective change in conformation and fluorescence intensity in the presence of the complement DNA or mRNA sequence [3, 4]. The MBs used target genetic content associated with regulation and detoxification of reactive oxygen molecules that can lead to increased breast cancer risk. MB probes were designed and provided through collaboration with Prof. Weihong Tan at University of Florida.

Summary of Research

Fluidic channels are constructed in a fused silica substrate using conventional microfabrication techniques—a single layer of photolithography followed by reactive ion etch. Fluid reservoirs access the channels by through-wafer ports and the final device is assembled with a direct wafer bond technique. The process allows for rapid prototyping of many fluid channel arrays on a 100 mm substrate.

To quantify a sample’s genetic content, the single-stranded-DNA (ssDNA) or mRNA is hybridized to a MB and driven electrokinetically through the transparent fluidic channel. Using a focused laser spot to excite fluorescence in the channel cross-section, a subfemtoliter-sized focal volume is formed that contains only one DNA/MB hybrid molecule at a time (Figure 1). This combination of flow control and single molecule confinement extends the limit of detection to an arbitrarily low concentration, restricted only by the tradeoff in data collection time and the experimenter’s requirement for statistical significance in describing the sample population. In this work, detection is performed at the 100 pM level and molecules were counted at a rate of approximately 800 per minute using a driving potential of 100 volts across the fluid channel.

Fluorescence changes in ssDNA/MB hybrids, at both the 100 nM and at the 100 pM concentration regime, were studied using a bulk solution and a single molecule spectroscopy (SMS) measurement, respectively (Figures 2, 3). Both measurements exhibit a 2-3x fluorescence intensity change upon hybridization—demonstrating that MB fluorescence changes can be observed at the single molecule level and are in agreement with ensemble measurements.

The utility of SMS to study hybridization is further demonstrated when the amount of excess target ssDNA is varied. By comparison of ssDNA and MB mixtures at both 10:1 and 1:1 ratios, this measurement technique illustrates a shift in favorable binding conditions measured through a reduced fluorescence enhancement (Figure 4).

This result supports SMS as a tool for investigating binding kinetics and molecular probe specificity.

References

Figure 1: Submicrometer fluid channel and formation of the inspection volume.

Figure 2: Bulk measurement of MBs and ssDNA/MB hybrids.

Figure 3: SMS measurement of MBs and ssDNA/MB hybrids.

Figure 4: Variation in excess target ssDNA impact hybrid population’s fluorescence.
Nanofluidic Channels for Biological Manipulation and Analysis

CNF Project # 762-99
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Abstract

We have used the interface between a nanochannel and a microchannel as a tool for applying controlled forces on a fluorescently labeled deoxyribonucleic acid (DNA) molecule, and observing the molecule in real time. A DNA strand, with a radius of gyration larger than the nanochannel width, that straddles such an interface, is subject to an essentially constant entropic force, which can be balanced against other forces such as the electrophoretic force from an applied electric field. By controlling the applied field we can position the molecule as desired and observe the conformation of the molecule as it stretches, relaxes, and recoils from the nanochannel.

Summary of Research

Fluid-filled channels with dimensions on the order of several tens to several hundreds of nanometers can be used as tools to study fluorescently labeled DNA molecules on an individual basis [1]. A DNA molecule floating in free solution assumes a spherical conformation with a radius of gyration in the range of one-half micron to several microns, depending on the DNA contour length. DNA molecules confined to nanochannels however, are observed to stretch out in one dimension (along the axis of the channel) as depicted in Figure 1. A DNA strand in such an extended state may be interrogated optically, yielding information about the molecule’s length and conformation [2]. In particular, the dynamics of single molecules as they

Figure 1: Schematic of the nanochannel array device. (A) Cross section of device consisting of two bonded fused silica wafers (a, b) with the upper one containing the structure. The microchannel was contacted from the top of the device and fluid reservoirs (c) were attached. Electrical connections to the channel were made by platinum electrodes (d). (B) Close-up of the nanochannel array in the upper wafer. DNA molecules have been drawn in the loading zone (a), as they enter a nanochannel (b), and in an elongated equilibrium conformation in a nanochannel (c).

Figure 2: Time traces of fluorescently labeled DNA molecules confined to nanofluidic channels. Each column of pixels in this image represents the fluorescence intensity along the axis of the channel in one movie frame. Many of these vertical lines are placed side by side to produce the image which shows the position, length, and contour of the DNA molecule in the channel over the course of the entire movie clip. (A) Straightened molecule. First the molecule is driven downward (a) and then upward (b) by 21 V/cm pulses. (B) Molecule with the upper end folded. This molecule is also driven downwards (c) and then upwards (d). The response was the same as in panel A. No unfolding was observed.
contract in the channel or recoil from the channel were studied. In this case, the term “recoil” describes a process of entropically-induced self-extraction from a channel. Further, a distinction was made between molecules that enter the channel straightened out, and those that enter with a front end that happens to be folded over on itself. The part that is looped over on itself is roughly twice as bright as the unlooped portion of the molecule. This is demonstrated by the time trace graphs in Figure 2.

The nanochannel devices, shown in Figure 1, were patterned on a mirror-polished fused silica wafer with a thickness of 500 mm using a combination of electron beam and optical lithography. Initially, a layer of gold, 25 nm thick, was evaporated onto poly(methyl-methacrylate) (PMMA) electron-beam resist to help draw current during the electron-beam process. Negative patterns of the nanochannel regions were exposed using a JBX-9300FS electron beam lithography system. After removal of gold and PMMA development, the patterns were transferred to a chrome mask by evaporation and lift off. Microchannel patterns were then added to the mask using optical lithography and the same lift-off process. Both micro- and nanochannels were etched simultaneously using a Plasmalab 80Plus REI with a CHF$_3$/O$_2$ mixture at 50 Watts for 20 min. Access holes were created by alumina powder blasting from the backside of the wafer. Finally a 170 mm fused silica cover wafer was touch bonded and annealed at 1050°C to the device wafer, enclosing the channels. Nanoports were sealed to the access holes forming buffer reservoirs. Several electron micrographs of the nanostructures before bonding are shown in Figure 3.

Finally, we have modified the microscope setup in order to allow for higher throughput DNA detection. As opposed to illuminating of a large field of view of the chip, and detecting light from that field of view with a charge coupled device (CCD) camera, we now focus two or more laser spots on a channel, as depicted in Figure 4. When a DNA strand passes through a spot, its associated fluorophores are excited and emit a fluorescent signal. Emitted light is collected by one of several aligned optical fibers and detected by avalanche photodiodes. This arrangement allows for molecules to be driven through nanochannels and detected at high speeds. Information regarding velocity, fragment length, and conformation may be collected for thousands of molecules in the course of 1 minute.

References


Affinity Separation in Hybrid Polymeric Microfluidic Devices Coupled with Mass Spectrometry

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Abstract
The combination of microfabricated microfluidic devices and mass spectrometry (MS) provides current technologies with significant advantages such as high throughput analysis and fast analysis time [1]. We report the demonstration of affinity separation of proteins in hybrid polymeric microfluidic devices. The microchips were made of cyclic olefin copolymer and obtained through hot embossing and thermal bonding. An SU-8 electrospray tip was patterned and used to interface the microfluidic system with a mass spectrometer. Polymer beads were successfully polymerized, packed in situ and used as a separation matrix when derivatized by Cibacron Blue 3GA. The performance of the microchip was evaluated by MS analysis of the protein sample mixture.

Summary
Photolithography was used to create SU-8 triangular electrospray tips. These tips were sandwiched between two cyclic olefin copolymer chips by using thermal and press bonding. Before bonding, microfluidic channels were embossed in a polymer substrate using a silicon master. After coupling two components, based on a photografting method in situ UV-polymerization was carried out to create an affinity column in the channel. Polymerized beads were then treated with ethylenediamine and derivatized by Cibacron Blue 3GA for 9 hours at 60°C.

For demonstration purpose, the sample mixture of proteins, lysozyme and cytochrome c, were introduced to the device. A microfluidic channel, 150 µm wide and 150 µm deep, was obtained by an embossing method. The SU-8 was spun on the surface of borofloat wafer. After simple lithography, the borofloat wafer was released by HF etching. This SU-8 triangular tip was aligned with the channel and designed to protrude from the end. This triangular structure yielded the formation of a stable electrospay by guiding a Taylor cone along the electric field. For affinity chromatography, the double T-junction was employed to load protein sample mixtures in the main channel. In order to minimize the dead volume from the interconnection between the external pressure source and the microfluidic device, silica capillary tubes were aligned with the embossed channels and delivered proper pressure.

In situ polymerization was achieved by using a photografting method. Monomer and porogenic solvents were introduced to microfluidic channel via silica capillary. A photomask was used and exposed under UV light in order to control the location of the polymer bed. A 5 mm long polymer bed was successfully located at desired location. After the formation of the polymer bed, for on-chip separation using affinity chromatography, this bed was derivatized by Cibacron Blue 3GA [2].

Lysozyme and cytochrome c were selected as sample mixtures. We were able to demonstrate affinity capture of lysozyme in the polymer bed. Mass spectra shows that only cytochrome c has passed through the polymer bed region. An investigation concerning reverse phase liquid chromatography using the same microfluidic device is under way.

References
Figure 1: Total ion current after the affinity capture of lysozyme from the sample mixture. Only Cytochrome C was observed in the peak.

Figure 2: (A, top) Mass Spectrum of bovine Cytochrome C molecular weight of 12231 Da. This protein is introduced to the mass spectrometer by direct infusion using the SU-8 electrospray tip in the circle. (B, bottom)
Controlling Microarray Spot Morphology Using Polymer Liftoff Arrays

CNF Project # 762-99

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Abstract

Microarrays are popular in biology today for parallel screening of thousands of genes and proteins, however microarray data are known to be noisy and imprecise [1]. To address this issue, we have developed a novel polymer liftoff substrate that improves the uniformity of a microarray printed spot, as well as reduces the background signal, hence achieving a better reproducibility in the results as compared to standard microarray slides [2]. In addition, we have also observed the drying behavior of complex solutions (printing buffers containing DNA) on our polymer substrates, postulating the underlying dynamics that may control microarray spot morphology.

Our polymer arrays are compatible with existing microarrayer technologies, and experiments are underway to use these arrays to study tissue samples.

Summary of Research

Data obtained from DNA microarray-based experiments consists of measurements of thousands of individual genes. As such, the confidence in the data depends critically on the printed spot morphology, amongst other experimental factors. However, the dynamics underlying the drying of complex solutions (printing buffers containing DNA), which influence the spot morphology, are not well understood. To control and improve the uniformity of a printed DNA spot, we have developed a novel polymer liftoff substrate that combines a hydrophobic polymer patterned on a hydrophilic surface, using standard photolithography techniques [3].

Briefly, the layer of polymer was conformally vapor-coated onto glass substrates, then the polymer was subsequently patterned using oxygen plasma reactive ion etching while using a photoresist mask. The process defined the exposed areas of the underlying glass substrate. Thereafter, these polymer microarrays were spotted with probe oligonucleotides in a conventional microarrayer, and then the polymer was peeled away to leave only the probes printed on the surface (Figure 1). These substrates were then hybridized and processed in the same way as a conventional microarray slide.

We have observed a smaller standard deviation in the fluorescence pixel intensities measured from our polymer arrays as compared to conventional microarrays, across a range of concentrations (Figure 2). We postulated that the hybrid hydrophobic-hydrophilic nature of our substrate creates an inward flow of DNA material as the printed spot is drying, hence evenly depositing DNA in each spot (Figure 3). This contrasts to the outward convective flow in a printed spot on a conventional microarray slide, which corresponds to the commonly observed “coffee-ring” pattern formed by a droplet upon drying on a hydrophilic surface [4].
Our polymer arrays offer the advantages of improved uniformity and higher reproducibility over conventional microarrays slides. We have shown these benefits are a result of the constrained deposition of DNA inside the patterned openings via tailoring of the substrate surface properties. These polymer arrays are compatible with existing conventional microarrayer technology. We envision that the results of this work can be applied to other biological applications such various biomolecular arrays (e.g. cells, proteins, RNA), as well as self-assembly and concentration of dilute analytes.

References


Figure 2: Standard deviation associated with measurements of pixel intensities across arrays for successive dilutions. Inset: Standard deviation for individual dilutions represented as a percentage of the mean intensity.

Figure 3: Drying behavior of the printing buffer is different on polymer substrates than on control slides – capillary flows are (a) outward from the spot center for control slides, but (b) inward for polymer substrates. Last panel on each row show the combined time frames and flow direction. Fluorescent beads are used to track the flow.
Micromachined Flow Probe

CNF Project # 807-99
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Abstract

Transgenic mouse models are important tools for physiological research, but existing physiological monitoring tools—specifically devices for measuring blood flow—are often too bulky for acute/chronic mouse studies. To meet this need, we successfully fabricated 1200 micron-high ultrasonic blood flow measurement probe bodies from SU-8 photoepoxy. Transonic Systems is now selling probes manufactured with the CNF-developed SU-8 process. Scientists use these probes for basic research on transgenic mice, and pharmaceutical companies use them in developing new treatments for human diseases. In testimony to Congress, the National Institutes of Health cited this project as an NIH SBIR/STTR Commercialization Success Story.

Summary of Research

Medical researchers are increasingly using genetically altered mice as models for studying drug effects and disease treatments. Such studies often require measurements of arterial blood flow to various organs, such as the kidney. However, in mice these arteries are quite small (300-500 µm in diameter), and the required probe dimensions fall into a gray area between the capabilities of deep reactive ion silicon etching (< 200 µm) and traditional metal machining techniques.

A technology that bridges these extremes is the Microchem Corporation’s SU-8 photoepoxy [1, 2]. SU-8 is a biocompatible negative photoepoxy designed to create films hundreds of microns thick with a single spin. SUNY Albany’s Dr. Bai Xu and the CNF staff helped us extend the standard spin, bake, and exposure recipes [3] so that we could create structures up to 1000 µm thick in a single spin, as well as create dual-layer structures exceeding 1200 µm in height. In contrast, most structures created at CNF are well under 10 µm high.

The fabrication sequence only requires a spinner, a hotplate, an exposure tool, and some wet chemicals. The SU-8 photoresist is spun onto a 3” silicon wafer at low speed, and then the wafer undergoes a hotplate bake to remove solvent. The wafer is exposed through a chrome-glass mask to polymerize the SU-8 pattern. The spin/bake/expose process is then repeated to produce the second layer, and the wafer is placed in a solvent to remove unpolymerized SU-8. The probe bodies are then released by dissolving the silicon wafer.

Figure 1 shows a typical SU-8 probe body. This particular device is 1000 µm high x 3200 µm wide x 3500 µm long. The body is a precision mechanical fixture for the components of an ultrasonic transit-time flow measurement system [4, 5]. Figure 2 compares the tip of a ballpoint pen to the assembled probe (right) and an older Transonic probe manufactured with traditional machining/molding techniques (left).
Probes were then sent to Dr. Michael Callahan (Wake Forest University), who devised a surgical technique for implanting a probe onto a mouse’s renal (kidney) artery. Figure 3 shows the mouse two months post-surgery. Dr. Callahan recorded the renal blood flow traces shown in Figure 4 by simply plugging a lightweight cable onto the connector behind the mouse’s right shoulder and recording blood flow (ml/minute) as the mouse moved around its cage. This marks the first time that anyone has successfully recorded renal blood flow in conscious mouse.

Transonic Systems is now commercially selling blood flow measurement probes whose bodies are manufactured with the Nanofab-developed SU-8 process. These probes are being used in basic research on transgenic mice, as well as by pharmaceutical companies developing new disease treatments. (See the list of Recent Biomedical Research that cites use of these devices).

Our current Nanofab research has developed a much simpler process for releasing the probe bodies from the wafer.

This work was funded by the National Heart, Lung, and Blood Institute of the National Institutes of Health under SBIR (Small Business Innovative Research) Grant #5 R44 HL 55082-03, “Tools & Techniques for Measuring Flow & Pressure in Mice.” In testimony to Congress, the NIH (National Institutes of Health) cited this project as an NIH SBIR/STTR Commercialization Success Story [6].

Recent Biomedical Research that cites use of these devices:


References


Post-CMOS Fabrication of an Electrochemical Biosensor

CNF Project # 848-00
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Abstract

Patch clamp and amperometric recordings are highly sensitive techniques measuring picoampere currents that reveal a wealth of detail on cellular and molecular function. One limitation of these approaches is that each cell requires an individual expensive amplifier for low noise recording. We developed a scalable potentiostat circuit that is capable of measuring currents with sub-millisecond time and picoampere current resolution.

In this report, we describe the post processing of a test chip that was fabricated in a 0.5 µm, 5-V complementary metal-oxide semiconductor (CMOS) process through a metal oxide semiconductor implementation system (MOSIS). Each amplifier on the chip occupied a layout area of 26 µm x 24 µm and contained eight transistors and a 50-fF integrating capacitor. The rms current noise at 2 kHz bandwidth is 110 fA which is comparable to the noise typically achieved with patch clamp amplifiers [1]. The post processing was done using the focused ion beam technique to coat platinum, a polarizable electrode, on the aluminum pads.

Summary of Research

In this report, we detail the post processing steps of the CMOS test die and report experimental results in an electrochemical environment. Post processing involves deposition of a polarizable electrode on the input electrode of the CMOS chip to measure amperometric signals.

The focused ion beam (FIB) is a direct write instrument that is often used to correct CMOS integrated circuits. We used this technique to remove native oxide on the aluminum electrode and then deposit platinum as shown in Figure 1. Using the picospritzer, we released pico literes of buffer with various concentrations of dopamine on the electrode. The Ag/AgCl ground electrode was placed in the pipette.

We have recorded amperometric currents in response to various concentrations of Dopamine as shown in Figure 2. While this method was useful in successfully depositing platinum over a small electrode area, it was not feasible to use the FIB to deposit platinum over an array of electrodes due to two reasons. The first reason was that deposition of platinum using the FIB did not contain platinum to the specified geometry. There was a haze of platinum deposited around the region of interest (Figure 1). This caused shorting of electrodes spaced close to each other. The second reason was that the beam constantly mills away at the surface, removing material which made positioning the beam at different locations without damaging the sample difficult.

We are in the process of developing a reliable method using lithography to deposit an array of platinum electrodes on the CMOS die. The difficulty with lithography is the size of the die which is 2.4 mm x 2.4 mm. This die is too small to be placed on any of the processing tools at the CNF. We use a carrier wafer on which the die sits and the subsequent steps are carried out. Electrochemical and cellular experiments and the reliability of the processing method are to be yet to be tested.

References

Figure 1: Platinum deposited on aluminum electrodes using the FIB.

Figure 2: Electrochemical measurement of dopamine on chip.
Abstract

Neurons release neurotransmitters in a process called exocytosis, wherein packets of transmitter molecules are released from the cells upon stimulation. Adrenal chromaffin cells, which also undergo exocytosis, constitute a model system for neuronal release. Quantal release of adrenaline from chromaffin cells can be measured using the electrochemical technique of amperometry, historically employing a carbon fiber electrode [1]. To gain information about the exocytotic mechanism, we have developed a method of electrochemical imaging employing transparent microfabricated devices based on amperometry. We have successfully measured electrochemical events using transparent electrodes simultaneously with fluorescence imaging, yielding more information about the exocytotic process.

Summary of Research

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. Exocytosis can be observed by amperometry, an electrochemical detection method. In amperometry, transmitter molecules released during an exocytotic event are oxidized by a nearby electrode, and the oxidation current is recorded. Historically, carbon fiber electrodes have been employed for this purpose [1].

To gain information about the mechanism of transmitter release, it is desirable to perform simultaneous fluorescence imaging and electrochemical detection of individual release events. We previously demonstrated that amperometric signals from chromaffin cells can be measured using opaque platinum electrodes patterned on a glass coverslip while simultaneously observing fluorescence from the cell between the electrodes. And, that the platinum electrodes behave similarly to carbon fibers [2]. However, to minimize diffusional broadening, the electrode needs to be in close contact with the plasma membrane of the cell of interest. This is particularly important if one wants to study exocytosis from cells such as neurons using amperometry, and particularly difficult if one wants to observe fluorescent labeling in the cells simultaneously.

To overcome this problem, we have developed transparent electrode arrays for simultaneous amperometry and fluorescence imaging through the electrode. Transparent electrochemical detector arrays were fabricated on glass using standard photolithography techniques, and insulated using photoresist. Arrays were fabricated from either indium tin oxide (ITO) [3], or very thin (~ 6 nm) gold (Figure 1). Amperometric signals from bovine chromaffin
cells could be detected with both materials at low noise, and fluorescence changes monitored through the electrodes. Figures 2 and 3 show examples of an electrochemical signal and a fluorescence image from ITO electrode arrays. Amperometric foot signals [4], indicating the initial stages in exocytosis, were detected with both types of electrodes. For arrays with similar geometry, mean charge and half width of the amperometric spikes were 0.89 ± 0.44 pC, 34 ± 9 ms (SEM, n = 4 cells) for transparent gold electrodes and 0.38 ± 0.05 pC, 32 ± 2 ms (SEM, n = 6 cells) for ITO. The difference in quantal size has a p value of 0.18. The results suggest that amperometric detection of transmitter release with transparent gold electrodes is comparable to that using carbon fibers while ITO may be less efficient.

In the future, we hope to record exocytotic events and fluorescence information directly from neurons. To that end, we are developing electrode arrays suitable for growing neurons across, as shown in Figure 4.

References

Simple Platform for BeadChip™ Bio-Assay

CNF Project # 854-00
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Abstract

An on-chip bioassay plate was fabricated based on a previously developed BeadChip™ format and a simple fluidic system.

Summary

Previously, we have developed a BeadChip™ platform for bioassay and detection based on color-coded microparticles (“bead”) assembled into a silicon chip. To add convenience to the assay reaction process on the BeadChip™ platform, a hydrophilic/hydrophobic pattern was created around each bead array. Such a pattern regulates the movement of the sample solution on the chip surface, thus facilitating the assay process. A format resembling a tradition 96-well titter-plate was fabricated on a wafer. Each well encompasses a BeadChip™ array. Assay for 96 samples can be carried out simultaneously on this plate.

References

A Microfabricated PCR-Based Biosensor

CNF Project # 884-00

Principal Investigator: Dr. Carl A. Batt

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Abstract

A portable, fully-automated, polymerase chain reaction (PCR)-based detection system has been developed for the rapid detection of bacterial pathogens and forensic deoxyribonucleic acid (DNA) analysis [1].

Experimental Research

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) [2]. 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 single nucleotide polymorphisms, 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 the fluid in three dimensions using two patterned layers thereby completely surrounding the central stream with sheath flow (Figure 1). In addition, we have elegantly positioned our optical excitation and detection systems into the particle counter by lithographically integrating optical fibers with the micro-fluidics flow-cell (Figure 2).

Figure 1: Hydrodynamics flow focusing in 3-D with two-layer microfluidics. A central stream containing red food coloring is surrounded on all sides by sheath fluid. The channel width and height are each 200 µm.

Figure 2: A 5 µm fluorescent microsphere passes through the excitation volume of the particle counter. The left two fibers are coupled to excitation sources and the right two fibers are coupled to photodetectors. The channel height and width are each 125 µm.
Integration of fiber optics eliminates the need for cumbersome optical realignment with repeated use of the same device by enabling lock-and-load compatibility of the microchip with the optical detection system. Using this system, we are able to observe fluorescent DNA hybridized to 5 µm diameter beads, demonstrating the potential for bead-based DNA analysis (Figure 3).

**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 Magnetic Bead-Based Microfluidic Mixer as a Sample Preparation Module for Portable PCR-Based Biosensing

CNF Project # 884-00
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Abstract

For the purposes of rapid detection of bacterial pathogens and forensic deoxyribonucleic acid (DNA) analysis, a portable, fully-automated, polymerase chain reaction (PCR)-based detection system has been developed in our lab. 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. To further broaden the utility of this system, we are developing a sample preparation module, which would isolate and purify cells of interest. Current work has focused on the development of a magnetic bead-based microfluidic sample preparation system for the isolation and purification of target cells from a raw sample using antibody-coated magnetic beads.

Summary of Research

A portable, fully-automated PCR-based detection system has been developed in our lab for the rapid detection of bacterial pathogens [1-3] and forensic DNA analysis. To further broaden the utility of this system, we have developed a microfluidic sample preparation module that removes cells of interest from a raw sample by mixing with antibody-coated magnetic beads, as shown in Figure 1.

Fabrication of the microfluidic mixers employs a two-step SU-8 process to generate the polydimethylsiloxane (PDMS) mold. The PDMS-on-PDMS mixer chip (Figure 2) consists of serpentine channels with herringbone structures for passive mixing [4, 5], and the two layers of PDMS are manually aligned and adhered using oxygen plasma and 60°C baking temperatures. Bubble traps and post-mixing stray-bead-capture chambers are included, and are used in combination with permanent magnets for cell capture.

Figure 1: Microfluidic mixer schematic.

Figure 2: Digital photograph of the Microfluidics Desktop automated PCR system.
Bead leakage was studied at various flow rates with different methods of magnetic field application. Higher flow rates resulted in more bead leakage and weaker flexible magnets allowed for more even bead distribution throughout the mixer than the rare earth magnets. Passivation of the PDMS is required to prevent proteins and cells from nonspecifically adhering to the polymer walls. Experiments with different methods of PDMS passivation (5% Pluronic, 5% Bovine serum albumin (BSA), and no passivation) were conducted, and it was found that incubation with 5% BSA was most effective. The 5% Pluronic passivation technique requires incubation inside the PDMS device for at least 24 hrs prior to use, which introduces difficulties such as bubble formation, as opposed to the 5% BSA passivation method, which requires only 10 minutes of incubation prior to the actual experiment.

Cell samples and antibody-coated magnetic beads are simultaneously pumped into the microfluidic device for passive mixing. The external magnetic field traps the magnetic beads and captured cells within the PDMS channels, and a phosphate buffer saline (PBS) wash is pumped through the device to separate the captured cells from the raw sample. The cells are subsequently lysed open with a flow-through of 4.0 M guanidine thiocyanate (GuSCN) lysis buffer, and the cell contents are pumped into the DNA purification microchip. Bacterial cells have been selectively isolated from both pure bacteria culture samples and contaminated raw chicken samples and lysed to release DNA. The DNA purification microchip employs silica-coated microstructures to selectively bind, wash, and elute nucleic acids in preparation for real-time PCR. The Microfluidics Desktop platform (shown in Figure 3) with integrated microprocessor, pumps, valves, thermocycler and fluorescence detection modules, is used to isolate and lyse bacterial cells, purify bacterial DNA, and detect bacterial DNA using real-time PCR amplification. Between 100 and 1000 Salmonella typhi cells could be detected using this system with good repeatability and an average turnaround time of 120 minutes.

References

**Bacterial Colonization and Communication in a Microfabricated Fluidic Channel**

**CNF Project # 935-01**

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**Abstract**

Bacterial plant pathogens *Xylella fastidiosa* and *Xanthomonas campestris* pv. *campestris* are responsible for important economic losses in the industry of grapes and cabbage, respectively. Both bacterial species are confined to living in the water (sap)-conducting xylem vessels of plants. We are studying the biology of these pathogens in microfluidic chambers used as ‘artificial xylem vessels’. Using this approach we were able to understand *X. fastidiosa* movement against the sap flow, and explain the role of two types of pili in this bacterium, especially regarding movement and biofilm formation. We are currently studying signaling processes involved in *X. campestris* pv. *campestris* flagellar movement, important for entry into the host plant xylem.

**Summary of Research**

*Xylella fastidiosa* is a plant pathogenic bacterium that causes important losses in grapevines and other high value crops. The bacteria exclusively inhabit the water (sap)-conducting xylem vessels of the plant hosts. It is believed that *X. fastidiosa* causes disease by developing bacterial aggregates and biofilms that block xylem sap flow. Because temporal and spatial activities of the bacteria can not be observed microscopically within the plant, microfluidic devices have been developed to mimic features of plant xylem (Figure 1). Using such devices, we discovered that *X. fastidiosa* migrates upstream via hair-like structures (type IV pili) [1, 2] that are repeatedly extended and retracted from the bacterial cell in a movement known as twitching. Such movement explains, in part, the spread of...
bacteria in the host plant against the flow of sap [1]. In addition to type IV pili, X. fastidiosa possess shorter type I pili positioned at the same cell pole. Using microfluidic devices, we measured the adhesion force of both types of pili, and found that the shorter type I pili are primarily responsible for cell attachment to surfaces [3]. Mutants without type IV pili (pilB, viz., type I pili only) exerted the greatest adhesion force to surfaces, while mutants without type I pili (fimA) had the weakest attachment to the surface (Figure 2). The presence of the type I pili slow the speed of movement of the bacteria, as was demonstrated by our measurements of the speed of twitching movement of wild-type cells compared to pili-defective mutants. Cells without the type I pili ‘brake’ (fimA) move six times faster than wild type cells. A mutant defective in a type IV pilus tip protein (pilY1) moves three times slower than the wild type (Figure 3). Our findings indicate that X. fastidiosa uses both types of pili to colonize the xylem environment: type IV pili are necessary for movement, and type I pili are helpful in surface attachment, therefore influencing biofilm development [4].

**References**


**Figure 3. Comparative paths of twitching movement within microfluidic devices of Xylella WT, pilY1 (type IV pili tip protein) and fimA mutants.**

**Figure 4. Fluorescent image of GFP-expressing Xanthomonas in microfluidic channels.**

**Xanthomonas campestris pv. campestris** (Xcc) is another xylem-inhabiting bacterium which causes black rot disease of cabbage, an economically important crop for New York State. Unlike X. fastidiosa, Xcc rely on motility via flagellar propulsion for entry into the plant xylem. However, little is known as to when the flagellar genes are deactivated and biofilm formation begins, allowing the bacteria to plug the xylem. The addition of a green fluorescent protein has allowed us to visualize activation and deactivation of gene promoters involved in colonization and virulence. This promoter activity is being compared to the motile and attached states of the bacterium while in microfluidic chambers.

Through the use of microfluidic devices, we are gaining information on the biology and the infection process of xylem-inhabiting bacterial plant pathogens, which may lead to the development of novel control methods against these pathogens.
Microscale Analysis of Cerebrospinal Fluid Proteins

CNF Project # 942-01

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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. To achieve the aim of improving diagnosis, we are developing two technologies. The first technology involves devices for the separation and electrospray ionization of CSF proteins. The other technology involves chemistry for the attachment and detection of specific biomarkers.

Summary of Research

It is important to use separations as a front end to mass spectrometry (MS) in the industrial proteomics community [1, 2]. The use of such separations permits improved analysis of cerebrospinal fluid (CSF) proteins and facilitates the discovery and measurement of protein biomarkers. We recently demonstrated the important role the CSF proteins play in the early diagnosis of Alzheimer’s disease [3].

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 prefractionation (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. However, a key challenge not addressed by any of our earlier technologies was the desire to have microfabricated tips rather than capillary glass tips. To address this issue, we have developed SU-8 based approaches to combined separations and electrospray ionization. Using this new technology, we have separated devices from cyclic olefin copolymer and SU-8, and demonstrated the analysis of bovine cytochrome c using an LCQ Deca ion trap mass spectrometer as shown in Figure 1.

References


Figure 1: Mass spectrum of 5 micromolar bovine cytochrome c using SU-8 triangular tip.
Patterned Surfaces to Investigate Spatially Regulated Mechanisms in Immune Cell Signaling

CNF Project # 996-01
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Abstract

Micro- and nano-fabricated surfaces have been widely used for applications in cell and tissue engineering. However, the full potential of these technologies has not been explored, particularly in the area of molecular cell biology. Employing these technologies, we are investigating fundamental mechanisms in immune cell signaling, specifically IgE receptor (FcεRI) signaling involved in allergic responses on mast cells. We are interested in learning the spatial regulation mechanisms for intracellular signaling events and the role of the actin cytoskeleton in these processes.

Summary

Recently, we established the use of patterned surfaces as a tool for visualizing spatial distribution of signaling molecules, as well as providing new insights in the structural and functional relevance of membrane compartmentalization [1, 2]. We have used standard photolithography techniques and the polymer lift-off method to fabricate surfaces containing patterned lipid bilayers with haptens that serve as antigens [3]. Antigens on the lipid bilayers bind and cross-link FcεRI-bound IgE on the surface of mast cells, thus activating signaling events in these cells (Figure 1). By spatially clustering receptors on the surface of mast cells, we are able to control and observe the local environment in which signaling molecules undergo a series of biochemical events. Previously, we showed that early signaling components such as Lyn kinase selectively co-redistribute with patterned receptors [2]. Currently we are studying the dynamics of the actin cytoskeleton following FcεRI mediated activation and we have identified possible new adapter proteins that may be involved.

We found that F-actin and other actin binding proteins such as vinculin (Figure 2) and paxillin are recruited to the clustered receptor sited and that this local recruitment may be mediated by interactions with Lyn kinase. This approach combined with more standard biochemical analysis is yielding new insight into the role of the actin cytoskeleton in FcεRI receptor signaling.

References

**Figure 1:** Cartoon representation of the interaction between receptors on the cell surface and the patterned lipid bilayers.

**Figure 2:** Visualization of the redistribution adapter proteins upon stimulation with fluorescence microscopy.
MEMS-Based Muscle Interfaces for Insect Cyborg Control

CNF Project # 1121-03
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Abstract
We, for the first time, demonstrated a reliable hybrid tissue-electronics interface in insects, by using silicon probes and inserting them at an early pupae stage so that the tissue grows around the probes for a highly natural implant in the insect. This work paves the way for future engineering approaches to utilize the bio-electronic interfaces and to create insect cyborgs. A successful emergence yield rate of 90% was obtained with the implanted systems. Preliminary flight control results indicate left and right wing control of *Manduca sexta* and full-flight control is under investigation.

Summary of Research
Here, we report on a novel hybrid biology pathway for autonomous centimeter-scale robots that forms intimate electronic-tissue interfaces by placing electronics in the pupae stage of insect metamorphosis. When micro-air-vehicles (MAVs) or tiny fliers are considered, the power source required to fly them within the constraint of generating lift, powering flight control sensors and actuators, and collision avoidance has limited the mission time and autonomy. A great idea has been to directly use nature designed and optimized flyers, namely, insects as MAVs. Insects are self-powered, are cm-scaled and operate with highly efficient flight muscle actuators. Electronic systems can be implanted in insects to study and control the insect flight by recording from and actuating the sensory, neural and muscular system. However, it is a challenge to implant electronic systems to intervene in the insect’s flight mechanism without disturbing the insect for an efficient flight. Any artificially attached platform and performed surgery on the “adult” insect is not reliable as the inserted devices on this stage can shift, create mass-balance disturbance and cause performance effecting tissue damage.

In our previous works [1, 2], we had demonstrated for the first time an efficient method to implant structures to tobacco hawkmoth *Manduca sexta*. With its 1-2 gram carrying capacity, miles of flight distances, wingspan of 10 cm and lifetime of 2-3 weeks, the *Manduca sexta* moth makes a wide range of applications for such systems possible. The method involves inserting structures to the pupae at early stages of metamorphosis such that the body adapts the structures during the development and inserted structures emerge out as a part of the body (Figure 1).

We optimized our insertion methodology to have an emergence success of 90% with our implantations. This hybrid structure enables a platform where CMOS devices and MEMS structures can be used as sensors and actuators not only for insect flight control but also for biological and environmental sensing. Our microsystem (Figure 2) consists of three parts: power, probe and control layers. The power layer comprises of two coin batteries and a slide-switch positioned on a printed circuit board (PCB). Each battery has an energy capacity of 8 mAh and weighs 120 mg. Conductive adhesive was used to attach the
batteries to the platform. The control layer is an 8 x 8 mm$^2$ PCB holding the microcontroller (Atmel Tiny13V) and an LED. A microcontroller was electrically connected to the PCB via flip-chip bonding. Wire-bonding was used to connect the PCB to the probe layer. The microfabricated silicon probe is sandwiched between these two layers. The overall system has dimensions of 8 x 7 mm$^2$ and total mass of 500 milligrams.

The MEMS-based silicon microprobe part consists of four tips (each 200 µm wide and 250 µm thick). The separation and the length of the tips were designed to match the main flight muscles that are located in the dorsal-thorax of the Manduca sexta. The dorsoventral and dorsolongitudinal muscle groups in this location move the wings by changing the conformation of the thorax and supplies the mechanical power for up- and downstrokes. Platinum (electrode metal) was evaporated and patterned on a 4” silicon wafer with a layer of 900 nm low pressure chemical vapor deposition (LPCVD) nitride. PECVD nitride was deposited on the metal and patterned to provide passivation. The metal lines running along the length of the probe tip, that actuate the muscle cells were only exposed close to the end of the tip (100 x 100 µm$^2$) for muscle specific excitation. The desired probe thickness was achieved by deep reactive ion etching (DRIE). A backside-only KOH etching was done to release the device.

The silicon-probe based microsystem platforms were inserted to the pupae seven days before the emergence. This is when a thin thoracic skin is formed under the cuticle of the pupae. When there is no skin, bodily fluids can leak from the insertion holes on the cuticle. In contrast, when the thoracic-skin is thicker, the probes buckle and break. The adaptation of probes by the muscle was highly maximized as the muscle grew around the probes. The emerged platform can be seen on Figure 3i where it is held with tweezers to show the wing opening of the moth. The x-ray image of the dotted part on Figure 3A shows one of the inserted probes. On Figure 3B, image processing of CT images shows components of high absorbance indicating tissue growth around the probe. Moths with inserted probes emerged out with a success rate of 90%. Upstrokes and downstrokes of each wing were obtained with electrical actuation.

References


Ultrasonic Microprobes with Integrated Sensors for Biomedical Applications

CNF Project # 1122-03
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Abstract

We report on a microfabricated silicon probe integrated with an ultrasonic actuator and polysilicon strain gauges for Microdissection TEsticular Sperm Extraction (TESE) surgery. An insertion experiment was performed on rat testis tissue and by sensing the strain we were able to ascertain the size of the sperm-carrying microtubules in the sample. This information is important in Microdissection TESE to distinguish tubules with and without fertile sperm, eliminating large incision currently required for optical spermatazoa localization. Experimental data on tissue rigidity measurements indicate that the micromechanical assay can also be used for minimally-invasive testicular cancer detection.

Summary of Research

In medical procedures such as percutaneous liver biopsy and endoscopic surgery, force measurement during the tissue-tool interaction can differentiate tissue by mechanical properties [1]. We report on a microfabricated silicon probe integrated with ultrasonic actuator and polysilicon strain gauges for Microdissection TEsticular Sperm Extraction (TESE) surgery. TESE is a highly invasive surgical procedure in which tubules carrying fertile spermatozoa are surgically extracted from the testicle of an infertile male, by visual examination of the tubules of the testicle under a microscope [2]. Tubules with low spermatogenesis have thinner walls and smaller diameter, as compared to healthy tubules. Our hypothesis is that by monitoring the force experienced by a microfabricated force probe inserted with 100-micron-scale incision, the surgeon can map the diameter and stiffness of tubule walls encountered during the incision. This could eliminate the need for a large incision and post-operative care, microscope visualization, excision of large volumes of testicular tissue, and could reduce the risks of causing permanent damage to the testis.

The fabrication process of our microprobe is similar to the ultrasonic microprobes reported earlier [3], however in this work, polysilicon strain gauges were integrated for force measurement, with differential force measurement across probes. Four polysilicon resistors are connected to form a Wheatstone bridge arrangement with two sense resistors for force measurement on each of the two microprobes (Figure 1). In this work, the silicon microprobes are 5 mm long, 100 µm long wide and 140 µm thick projecting outwards at the tip of the horn. The tip of the microprobe has 2 sharp blade-like edges – a V-shaped edge on the top formed by the LPCVD nitride film and one at the bottom formed by silicon, due to the anisotropic backside-only KOH etch (Figure 2). The two sense polysilicon resistors are positioned on the microprobe near its clamped edge, so their resistance changes with the compressive/tensile strain experienced when the blade-like edges of microprobes pierce the tubule walls. The Wheatstone bridge, with an amplification of 100, is formed on the two probes to realize a differential force measurement system, eliminating common mode forces on the two probes, as it is unlikely that the two probes would be cutting tubules simultaneously.

When the tubules are encountered by the blades on the microprobe, the strain experienced by the polysilicon resistors is due to a combination of pure axial compression and the bending of the cantilever. Depending on whether the tubules are encountered by the top (-0.977 V/N) or bottom (0.777 V/N) blade, the bending moment causes a compressive or tensile strain respectively. The
integrated ultrasonic actuator effectively stiffens the tissue [3] and reduces the deformation and applied force during probe insertion.

The microprobes are inserted into the rat testis tissue to a depth of 2 mm at 100 µm/s. The bulk portion of the microprobe is intended to be thicker in order to be stiff enough to be used for penetrating the outer testicular tissue and eventually reaching the seminiferous tubules. Once inside the tissue, the blades on the microprobe puncture the tubules and the puncture artifact in the force signal is observed to be a negative spike (compression) for the top nitride blade and a positive spike (tension) for the bottom silicon blade (Figure 3). The average time between two consecutive puncture artifacts multiplied by the speed (100 µm/s) can be taken as the mean diameter of the tubules, since the tubules are densely packed wall-to-wall in the tissue. From the FFT of the positive and negative spike data, the effective tubule diameter was calculated to be 41.16 ± 1.58 µm (Figure 4). This is close to the expected tubule diameter of rat testis.

The mean puncture force for the tubule wall as seen by the top blade on the microprobe was observed to be 101.51 mN. The data was also analyzed by amplitude binning, which indicates that the punctures were probably of three kinds: a) puncturing through the walls along the diameter (110-160 mN); b) along any chord of the tubule’s cross-section (60-110 mN); or c) separating attached tubules (0-60 mN). Thus we can quantify the size and the stiffness of the tubules encountered during an insertion experiment. At present we are integrating sample collecting channels in the probes that can be used to extract sperm from the ruptured vessels.

Since the two microprobes on the device are separated by a distance of 500 µm, the probability that both the microprobes will be cutting tubules at the same time is small. By monitoring the ratio of the offsets in the Wheatstone bridge of the strain gauges in the two microprobes when both are not cutting, the offset of a probe when cutting a tubule can be eliminated if the other probe is not encountering any tubules. This method can also help remove other effects such as drift, loading, temperature etc.

References

Implantable Flexible Microfluidic Devices for Convection Enhanced Neural Drug Delivery

CNF Project # 1195-04
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Abstract
Convection enhanced delivery (CED) can improve the spatial distribution of drugs delivered directly to the brain. In CED, drugs are infused through a needle or catheter, and can penetrate deeper into the tissue than drugs delivered by pure diffusion from an implant or other reservoir [1]. However, currently there are several challenges associated with this technique, such as tissue damage, backflow, and occlusion of the needle tip. To overcome these issues, we have created flexible implantable microfluidic probes that are suitable for chronic implantation. These probes consist of a flexible parylene channel on a rigid biodegradable polymer scaffold poly(lactic-co-glycolic acid) (PLGA). The scaffold is designed to allow tissue penetration and then erode over time, leaving only the flexible channel in the tissue.

Summary of Research
To make the flexible parylene probes, a 4″ silicon wafer is first slightly roughened in a CF₂ plasma and a layer of parylene C is deposited to form the base of the device. Next, a 10 µm high film of photoresist is spun and patterned to define the inside of the channels. This photoresist is encapsulated in a second layer of parylene C which forms the top of the device. A 200 nm aluminum film is evaporated and patterned into the final shape of the device through photolithography and wet etching. The wafer is then etched in an oxygen plasma to define the device body and open the channels. The aluminum etch mask is then stripped and the channels are cleared of photoresist in an acetone bath. A photograph of the finished devices on the wafer is shown in Figure 1.

In order to make the degradable scaffolds, a hot embossing process is used. Photolithography and deep silicon etching are used to selectively etch a silicon wafer to a depth of 200 mm to form a “master.” This silicon master is then silanized, and used to cast a PDMS mold. When the mold has cured, it is peeled from the wafer and used for the embossing procedure. A thin film of poly(lactic-co-glycolic acid) (PLGA) is sandwiched between the PDMS mold and another plain piece of PDMS. This sandwich is then heated to 180°C and pressed at 500 psi to form the scaffolds. The PLGA is cooled to below its glass transition temperature while it is still under pressure. Once cooled, the scaffolds can be removed from the mold. An SEM of the scaffold tip is shown in Figure 2.

In vitro testing of the device in 0.6 wt% agarose gel brain phantoms has shown that the device can deliver solutions at relevant flow rates with few of the problems of traditional needles, and recent in vivo tests in a mouse model show promising early results.

References
Figure 1: Photograph of finished flexible microfluidic devices on wafer.

Figure 2: SEM showing tip of biodegradable scaffold.
A Hydrogel-Based Microfluidic Device for the Studies of Directed Cell Migration

CNF Project # 1209-04

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Abstract

We have developed a hydrogel-based microfluidic device that is capable of generating a steady and long-term linear chemical concentration gradient with no through flow in a microfluidic channel. The device consists of three parallel channels in a hydrogel matrix and the chemical gradient is established in the center channel via diffusion of chemicals from the side channels. By flowing the two side channels with different chemicals, one can establish and maintain two independent gradients of different chemicals in the center channel. Chemotactic responses of *E. coli* to these “competing concentration gradients” provide insights into comparative roles of the receptors in the cells’ chemotactic behavior.

Summary of Research

One of the most fascinating and important aspects of cell biology is cell migration, and such an event is often mediated by the chemical gradients that are present in the environment. To study cell migration, we have developed a microfluidic device that is capable of generating steady and long-term linear chemical concentration gradients with no through flow in a microfluidic channel [1, 2]. The operation principle of the device is shown in Figure 1.

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**Figure 1.** Operating principle of the microfluidic device. The photograph below the schematic shows an actual image of the three channels when a fluorescent dye is flowing through one of the side channels.

**Figure 2.** Device fabrication starts with an Si master shown here. The features shown are created by etching Si wafers.
Three parallel microfluidic channels are patterned on a thin piece of agarose gel from a silicon master (Figure 2) produced at Cornell NanoScale Facility.

Fluid flowing in one outer channel (source channel) has a constant chemical concentration, while the fluid flowing in the other outer channel (sink channel) usually is devoid of the chemical and removes it from the system. The chemicals diffuse across the channels, and the steady state of the system is a linear chemical concentration gradient in the center channel. The gradient in the center channel is potentially able to persist infinitely provided that the flows in the side channels are maintained at a constant rate. By flowing two different chemicals through the two different side channels opposing gradients of these chemicals (Figure 3) can be established within the device. Chemotactic responses of cells to these “competing concentration gradients” provide insights into comparative roles of the receptors in the cells’ chemotactic behavior.

This three-channel configuration, diffusion based device has inherent advantages such as the chemical stimuli can be applied independently of mechanical stimuli and the gradient can be maintained indefinitely. The degree of chemotaxis can be easily quantified using this device in conjunction with fluorescence imaging techniques or other imaging analysis tools. This device will also be useful in controlling the chemical and mechanical environment during the formation of tissue engineered constructs.

References


Mimicry of Biological Adhesion Through Fabrication of Fibrillar Surfaces

CNF Project # 1225-04
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Abstract

The remarkably good ability for adhesion in geckos is because of the fibrillar structure found on their feet. This project aims to fabricate artificial mimics of these structures to make dry adhesives. The adhesive properties in these natural interfaces are due to their highly compliant geometry, so that they can make very good contact with all kinds of surfaces and hence weak surface interactions like van der Waals forces can be sufficient for good adhesion. We have made adhesive surfaces using the polymer poly(dimethylsiloxane) (PDMS). Our samples have thin pillars (fibrils) standing on the PDMS base and are topped by a thin roof of PDMS. We made different samples with varying fibril to fibril spacing and pillar/fibril heights. An indentation test was used on the samples to measure their adhesive properties such as work of adhesion, pull-off force and compliance.

Sample Fabrication

Samples with thin pillars/fibrils are made by molding polydimethylsiloxane (PDMS, Sylgard 184, Dow Corning) in silicon molds. Silicon (Si) molds are made using standard photolithography and deep ion etch techniques. The depth of the holes is estimated by the duration of etch, and that decides the height of the pillars in the samples. After the holes of desired depth and with desired pattern are made on the Si wafer, a self-assembled monolayer (SAM) of the molecule n-hexadecyltrichlorosilane is deposited on the wafer to reduce its surface energy. The polymer is poured in the Si mold and sandwiched between a glass slide (having SAM on it) and the mold. It is then cured in an oven at 80°C for 2 hours and then kept in dry ice for around 6 hours. In dry ice, the PDMS shrinks more than silicon, making it easier to remove it from the mold. The final step of affixing a thin terminal plate of PDMS on the pillars is accomplished by spin-coating a SAM-coated Si wafer with PDMS, and then placing the samples on this wafer with pillars in contact with liquid polymer. The assembly is then cured at 80°C for an hour in the oven. Glass coverslips are attached to the back of the samples while they are still on the wafer using O₂ plasma to activate adhesion between the two. Once the samples have the additional backing of a coverslip, they are carefully pulled off the wafer. Figure 1 shows a typical sample.

Indentation Experiments

Indentation experiments were carried out in a custom apparatus built on an inverted optical microscope. Typical experimental force and displacement data from a fibrillar surface and a flat control surface are shown in Figure 2. In either case, in the first cycle the sample was indented to the same depth but retracted to different depths, then cycled to the maximum depth ten times, and finally retracted completely out of contact. Note the markedly different behavior of a fibrillar surface and flat control surface. First, the fibrillar surface is much more compliant than the flat surface. Second, the fibrillar surface requires a greater pull-off force. Third, the fibrillar surface requires a greater amount of work to separate the indenter from the sample. Fourth, the fibrillar sample shows greater hysteresis in an indentation cycle. Contact micrographs are shown in Figure 2 for when the indenter is at a depth of 0 µm (a,e), 15 µm (b,d), and 30 µm (c) for both indentation and retraction. Figure 2 shows that the evolution of contact of the flat control surface is approximately symmetric about maximum indentation. This is not the case for the fibrillar samples. In fact, the contact area on the fibrillar surface remains pinned when the indenter retracts from 30 µm to 15 µm. This fact leads to energy dissipation and enhanced adhesion.
This work has been reported in the following journal publications:


Figure 1: Scanning electron micrograph of a synthetic fibrillar array with a terminal thin film. Fibril height is about 67 µm and spacing is 62 µm. Each fibril is square in cross-section with sides nominally 14 µm wide. The terminal film is about 4 µm thick.

Figure 2: Typical force vs. indenter depth of a fibrillar surface and a flat surface. The micrographs show the contact area corresponding to various points on the graph.
Microfabricated Tools for Macromolecular X-Ray Crystallography

CNF Project # 1232-04

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Abstract

Mitegen, LLC, develops, manufactures and sells a variety of microfabricated tools for handling small, fragile samples, especially those used in macromolecular crystallography and molecular structure determination. In the last year we have developed a variety of new tools for common sample manipulations and processes that improve product durability.

Summary of Research

Mitegen, LLC, was founded in 2004 to commercialize microfabricated tools for use in protein, virus and small molecule structure determination by x-ray crystallography. The first of these tools was developed with NIH support by the PI, a Cornell undergraduate and two Cornell graduate students in the PI’s Physics Department research group in 2002-2003 [1]. Subsequent product development and production has involved extensive use of CNF facilities and has heavily drawn upon the expertise of CNF staff. Mitegen’s core products — MicroMounts™ and MicroMeshes™ — have a unique combination of flexibility, rigidity and extremely low x-ray background scattering, and are now used by academic, government and pharmaceutical company laboratories in more than 30 countries. An international collaboration of scientists recently used our MicroMesh™ mounts at the Swiss Light Source to determine the structure of a viral protein with crystals that averaged only 5 µm in size — the smallest crystals ever successfully used in a de novo macromolecular structure determination [2].

Our CNF-related product development in the last year has focused on three areas. The first effort, led by Mitegen employee Ben Apker and CNF staff scientist Daron Westly, has been to develop processes based upon new polyimides to improve the mechanical strength and durability of our products, and also to allow much thicker polyimide films (up to 20 µm) to be patterned. We will begin shipping products based on these new processes in August 2007.

Figure 1: A 300 µm wide MicroSieve™.

Figure 2: MicroGripper™ mount holding a 300 µm protein crystal.
The second effort, by Ben Apker and by Cornell EPE summer interns Kent Loh and Ankit Patel, has been to design and produce new sub-millimeter-size tools for common sample manipulation tasks. Since protein crystals and other biological samples are very fragile, the tools must be soft and flexible so that incidental contact does not cause damage. Tools developed include MicroChisels™ (for removing samples adhered to substrates and separating samples), MicroLassos™ (for pulling samples that have adhered to substrates), MicroSieves™ (for transferring samples between solutions and holding them during soaks), MicroSaws™ (for cutting gels, dense lipid phases, and protein skins that develop over crystallization drops), MicroTips™ (for fine position of small samples), MicroRulers™ (for measurement of sample dimensions), and MicroProtractors™ (for measuring facet angles of crystals). We are also developing variants of the measuring tools that contain an x-ray phosphor, to allow easy measurement of x-ray beam dimensions and the position of the beam relative to the sample.

Our final effort, by Apker, Loh and Patel, has been to develop new tools that can actively grip fragile samples and hold them rigidly but delicately. One design uses a series of thin, flexible fingers, which grab the crystal when the mount is pushed down over it. These MicroGrippers™ are important, for example, for x-ray data collection at room temperature, because existing sample mounting methods allow sample slippage and motion.

References
Fabrication of Inorganic-Organic Interfaces

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Abstract

A molecular level understanding of interfaces between dissimilar materials is becoming more important in semiconductor manufacturing. Here we discuss the formation of structures which contain an organic-inorganic interface, with an emphasis on the effect of varying the chemical nature and microstructure of the organic layer. By selecting an appropriate chemical functionality, gas-phase organometallic precursors can be made to bind specifically and non-invasively to the endgroups on self-assembled monolayers. When transition metal nitrides are deposited on these organic layers by atomic layer deposition (ALD), growth rate and morphology of the resulting film are affected by the chemical functionality of the organic.

Summary of Research

Interfaces have always governed the performance of solid state devices. From a simple diode to complex interconnects, inorganic-inorganic interfaces have defined semiconductor technology to date. As semiconductor manufacturing moves to future technology nodes, devices will shrink to dimensions approaching the molecular regime. At these small length scales, designs are envisioned which incorporate the use of organic molecules, both as active [1] and passive [2] components. Organics are attractive because their structure and chemical reactivity can be customized using the tools of synthetic chemistry. Furthermore, they can be designed such that they form self-assembling structures which are organized on a molecular level. This project focuses on two stages of the formation of an organic-inorganic structure. The first is the growth and characterization of the organic layer; the second is the subsequent deposition of an inorganic film. We have mainly used well established “wet chemical” techniques to deposit the organic layer, while atomic layer deposition (ALD) in ultra-high vacuum has been used to deposit the inorganic layer.

We begin with a well-characterized substrate, either SiO$_2$ grown to give a high number of surface -OH groups, or evaporated gold. Self-assembled monolayers (SAMs) are then formed on the substrate from solution. RSiCl$_3$ or alkoxy groups are used to anchor the SAM to SiO$_2$ via the formation of Si-O-Si bonds, while R-(thiophene) is used the in case of gold. These SAMs have one of a number of endgroup functionalities, including -CH$_3$, -NH$_2$, (-NH$_2$)$_2$, and -OH. In addition to straight-chain SAMs, we have also fabricated branched organic structures with -NH$_2$ and -OH endgroups.

To obtain a branched -NH$_2$ functionalized SAM, first a monolayer of (3-aminopropyl)ethoxydimethylsilane was attached to the SiO$_2$ surface (“generation 0”). The surface was then treated with an acrylate to convert amine termination to a branched ester termination. Finally, the samples were immersed in a diamine solution to re-obtain -NH$_2$ terminal functionalities (“generation 1”), in (ideally) twice the concentration of the first monolayer.

Once the organic layers are formed, inorganic films are deposited using transition metal coordination complexes and NH$_3$. We have examined the reactions of the gas-phase organometallic precursors Ti[N(CH$_3$)$_2$]$_4$ and Ta[N(CH$_3$)$_2$]$_5$ with the SAMs described above [3]. Ellipsometry, atomic force microscopy (AFM), and x-ray photoelectron spectroscopy (XPS) have been useful techniques to examine the nature of SAMs, as well as to quantify the kinetics of adsorption of the complexes on these layers. Figure 1 shows the XPS signal from the Ta(4f) level for -NH$_2$ terminated layers, demonstrating that increasing the degree of branching (increasing the surface concentration of -NH$_2$) increases in the uptake of Ta[N(CH$_3$)$_2$]$_5$. Similar results were obtained for a branched -OH terminated film. Angle-resolved XPS shows that the deposition of the organometallic is confined to the endgroup for SAMs with
reactive -NH₂ endgroups, while in the case of unreactive -CH₃ terminated SÅMs, the organometallic tends to deposit at the SAM/SiO₂ interface.

These experiments in the monolayer regime are used to guide subsequent work, in which TiN and TaN layers are grown by ALD using the above mentioned organometallics in combination with NH₃ [4]. A custom-designed UHV chamber in which the organometallic precursor is delivered to the substrate by a collimated molecular beam is used to carry out ALD. One advantage to using a collimated beam is that terraced films can be grown without a vacuum break, simplifying the examination of the film at varying stages of growth. Another advantage is that the incident kinetic energy and angle of the precursor can be controlled. ALD experiments show that both the growth rate (Figure 2) and roughness of the nitride layers correlates with the endgroup of the SAM. Using a SAM with a reactive endgroup (-OH or -NH₂) leads to a higher growth rate, and more uniform, smoother films as compared to unreactive (-CH₃ terminated) SAMs. High resolution STEM images acquired by Prof. David Muller and Peter Ercius (Applied and Engineering Physics, Cornell) show that a conformal film is deposited on a NH₂ terminated SAM (Figure 3). Corresponding elemental EELS measurements indicate a peak in the carbon signal at the TiN/SAM/SiO₂ interface, showing that some remnant of the SAM exists after deposition.

References

Fabrication of Polymer Microfluidic Devices for Particle Separation via Insulative Dielectrophoresis

CNF Project # 1360-05
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Abstract

We report continuous-flow, dielectrophoretic (DEP) cell manipulation with an application to the rapid screening of mutated Mycobacterium smegmatis populations. Innovative aspects of the device include; (i) the use of novel, DC-offset, AC electric fields, (ii) the use of high width to depth aspect ratio constrictions (250:1), and (iii) application to post-mutagenesis screening M. smegmatis based on cell membrane characteristics.

Results

A two-stage silicon master fabrication procedure followed by hot embossing of micro-channels in polymer substrates allows for the rapid and facile fabrication of devices with high width to depth aspect ratios (250:1). After hot embossing, a chemical bonding procedure is used to enclose the embossed channel in polymer [1, 2]. Regularity of the aspect ratio across the channel was confirmed via profilometry after hot embossing, and confocal visualization after enclosing the channel.

A novel device design modulates electric fields in three dimensions using coherently patterned, curvilinear constrictions in the depth of a microfluidic channel (Figure 1). Curved constrictions induce dielectrophoretic trapping normal to the constriction and transport tangent to the ridge, achieving a continuous particle separation transverse to the direction of flow. Linear and non-linear electrokinetic phenomena are independently modulated using a DC-offset, AC electric field.

Particle separation takes 5 mm of channel length and accomplishes sorting in seconds, after which particles can be transported via linear electrokinetic effects to separate output channels. The small spatial scales and rapid time scales of this sorting technique, coupled with the ability to act on a large number of particles simultaneously, yield exceptionally high throughput compared to other continuous flow particle sorting techniques such as FACS and flow cytometry.

We have demonstrated separation of polystyrene particles with 20% variation in DEP mobility (Figure 2 & 3) and continuous control of a M. smegmatis particle stream position transverse to the direction of flow (Figure 4). Time-average fluorescence images indicate preferential deflection of high-DEP mobility particles (Figure 3) versus low-DEP mobility particles (Figure 2). Figure 4 shows a running integral of image intensity at the outlet (dashed lines in Figures 2 & 3) of similar images obtained using Mycobacterium smegmatis as the analyte.

Figure 1: Microchannels fabricated in Zeonor polymer with curvilinear constrictions in channel depth.
Well-characterized particles exhibit predictable behavior, traveling tangential to a curvilinear constriction in depth until linear electrokinetic mobilities dominate and particles pass the constriction at a point transverse to flow characterized by their particular mobility ratio and the operating conditions of the experiment. *M. smegmatis* show the same transverse deflection observed with microspheres and predicted by our model. Successes in sorting particles based on DEP mobility, dielectrophoretic manipulation of *Mycobacterium*, and validation through a model-based framework positively indicate the potential to use this device for high-throughput screening random mutations in *Mycobacterium*.

**References**


A Novel Method for Accurate Patterning and Positioning of Biological Cells

CNF Project # 1370-05
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Abstract
The ability to anchor cells in predefined patterns on a surface has become very important for the development of cell-based sensors, tissue-engineering applications, and the understanding of basic cell functions. Currently, the most widely used technique to generate micrometer or sub-micrometer-sized patterns for various biological applications is microcontact printing (µCP). However, the fidelity of the final pattern may be compromised by deformation of the poly (dimethylsiloxane) (PDMS) stamps used during printing. A novel technique for accurately patterning and positioning biological cells is presented, which can overcome this obstacle.

Introduction to Research
We have developed a new technique to control the placement and growth of cultured cells on patterned silicon oxide [1]. First, we coated a hydrophobic self-assembled monolayer (SAM) derived from 1-hexadecanethiol on a patterned gold surface to prevent cell growth, and then we deposited a hydrophilic SAM derived from 3-trimethoxysilyl propylidethylenetramine (DETA) on the exposed silicon oxide surface to promote cell growth. The fabrication process to make the substrate for subsequent cell patterning is illustrated in Figure 1.

First, 500 Å of plasma enhanced chemical vapor deposition (PECVD) silicon oxide film was deposited on a three inch silicon wafer at 250°C. Then, a thin Cr/Au film was patterned on the surface. Next, Shipley 1818 photoresist was spin-coated at 3000 rpm to get a 2.2 µm thick film. After soft baking at 90°C for 60 s, a contact aligner was used to expose the photoresist with a dose of 76.8 mJ/cm². This step was followed by baking in ammonia for 90 min in an image reversal oven, where the whole substrate was flood-exposed for 60 s. After soft baking at 90°C for 60 s, a contact aligner was used to expose the photoresist with a dose of 76.8 mJ/cm². This step was followed by baking in ammonia for 90 min in an image reversal oven, where the whole substrate was flood-exposed for 60 s. After developing in MF321 developer for 90 s, oxygen plasma was used to remove the photoresist scum residue. Then, 50 Å of chromium was deposited on the surface followed by 500 Å of gold by an e-beam evaporator. Finally, the lift off process was performed using a Microposit Remover 1165. The Cr/Au patterns on top of the silicon oxide surface are shown in Figures 2 and 3.

Figure 1: Preparation of the substrate for cell patterning.
Biological Applications

Summary

We have developed a convenient and robust method to position and pattern cultured cells with single cell resolution, which is easy to align with pre-existing structures on the substrate. An additional advantage of this method is that cytophobic/cytophilic patterns can be easily visualized prior to cell culturing. The best resolution we have obtained to date is two microns. It has been established that the single cells we have utilized here – immortalized mouse hypothalamic cells – can be successfully anchored on 20 µm wide square cytophilic islands, and 2 µm wide pathways are suitable for the neurite growth. With this technique, design and fabrication of complex biosensor systems will become possible.

References

A Multi-Electrode Array System for Patterned Neuronal Network

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Abstract

Presently, multi-electrode array (MEA) have been widely used as a non-invasive in vitro recording method to study excitable cells and tissues, such as peripheral neurons, stem cells, or sliced brain tissue. The limitation for a conventional MEA system is the accuracy of the response of an individual-cell-based neuronal network. Here, we have developed a novel MEA system with the capability to culture neuron cells in predefined patterns on top of electrodes which can record the signals of excited neuron cells.

Introduction to Research

The fabrication process is shown on Figure 1. A thin film of Cr/Au/Cr electrodes and their interconnects are patterned on top of a fused silica wafer by metal lift-off with image reversal processing. First, Shipley 1818 photoresist was spin-coated at 3000 rpm to get a 2.2 µm thick film. After soft baking at 90°C for 60 s, a contact aligner was used to expose the photoresist with a dose of 80.4 mJ/cm². This step was followed by baking in ammonia for 90 min in an image reversal oven, where the whole substrate was flood-exposed for 60 s. After developing in MF321 developer for 90 s, oxygen plasma was used to remove the photoresist scum residue. Then, 100 Å of chromium was deposited on the surface followed by 1000 Å of gold and 200 Å chromium by an e-beam evaporator. Finally, the lift off process was performed using a Microposit Remover 1165.

Then, one micron of plasma enhanced chemical vapor deposition (PECVD) oxide is deposited on top at 240°C and the openings in it are etched using dry etching technique. Next, the main distinguishing step with respect to a conventional MEA system was performed: a thin layer of Cr/Au was deposited and patterned on top of the PECVD oxide layer by image reversal processing. The function of this layer is to facilitate the anchoring of the neurons on the pre-defined positions and the guidance of neurites in between the neurons. Prior to culturing neurons on top of these MEAs, proper treatment with SAMs (using a previously described method [1]) has to be performed to make the gold surfaces hydrophobic and unsuitable for neuronal attachment and growth. Finally, an additional 3000 Å of gold was patterned on the contact pads by metal lift-off with image reversal processing for the subsequent wire bonding process.
A single MEA die that has been fixed and wire-bonded on a printed circuit board is shown in Figure 2, and an optical image of the fabricated MEA is shown in Figure 3. The underlying gold electrodes are light gray, whereas the top gold patterns (for neuronal guidance) are pale gray. Dark gray regions are exposed PECVD oxide, which will be treated using hydrophilic SAMs for neuronal anchoring and outgrowth. An SEM image of the 3.1 µm wide opening in PECVD oxide deposited on top of the gold electrode is shown in Figure 4. The circle and interconnect lines are visible as the topographical features, where dark grey is the exposed PECVD oxide and light grey is the thin gold deposited on top of it. Characterization of the MEA device by culturing hippocampal neurons is underway.

**Summary**

We have developed a novel MEA system with single cell resolution patterning that makes it possible to investigate the activities of individual cells in a patterned neuronal network.

**References**

Cellular Responses to Micro- and Nanofabricated Surfaces

CNF Project # 1372-05
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Abstract

Micro- and nano-fabrication provide opportunities to design devices that interact at the same scale as cells and molecular cell products that direct cell function. These opportunities can be harnessed not only to develop new strategies for understanding basic cell functions, drug actions, cell responses to stress, but also to build improved devices for long-term residence and function. These later devices will provide new methods for clinical application including drug delivery, chemical sensing, and recording and stimulating electrical responses. We are using these opportunities to develop improved methods for promoting long-term device-brain interactions. To accomplish our goals we are using in vitro systems as test beds as a step towards developing devices for animal studies and future clinical applications.

Summary of Research

We have used CNF to design and fabricate devices for two distinct applications. The first project is developing new methods to control the placement and distribution of biochemical signals on cell culture growth surfaces. This research program uses micrometer thick hydrogel coatings of surfaces as a foundation for cross-linking proteins. Since multiple biological signals are required we have developed these methods to permit a number of proteins. Synthetic hydrogels are ideal for biomedical applications in that they present an optically transparent, flexible, and non-toxic material. Hydrogel coatings were photo-polymerized in the presence of streptavidin-acrylamide, resulting in functionalized planar gel surfaces. Biotin-labeled biomolecules will find with near-covalent affinity to the hydrogel streptavidin. Soft protein lithography using microcontact printing is used to control the patterning of molecules onto test surfaces. Microcontact printing uses the relief pattern on the surface of an elastomeric stamp to deposit molecules into a specific pattern onto surfaces. 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 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.

Elastomeric stamps were produced using a series of silicon masters produced 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 polydimethylsiloxane stamps were produced by casting.
Biological Applications

The relief patterns were designed for a number of biological experiments. For instance patterns were made with repeating 15 µm discs and 2 µm-wide connecting lines (Figure 1). The geometry of these was made to correlate with the spacing and density of microelectrodes on planar arrays used for recording and stimulating neurons in primary cell cultures (Figure 2).

Studies in the past year have focused on studying; (i) the effects of neuron number and electrical stimulation on development of nerve cell networks [4] and (ii) the utility of thick hydrogel coatings for producing complex cell structures that can mimic the blood-brain [5] (Figure 3) or intestinal mucosa.

Summary

We have developed a novel hydrogel constructs that can be patterned with multiple biologically relevant molecules at micrometer scale resolution to direct nerve and endothelial cell growth and attachment. Results from this study indicate; (i) hydrogel surfaces can be patterned with multiple proteins to direct neural cell growth and attachment and that hydrogel systems can be fabricated to permit development of three-dimensional structures that mimic normal brain tissue.

References

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

CNF Project # 1377-05
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Abstract
Recent advances in the experimental sciences and coupled data analyses have paved the way for investigating systems of increasingly 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 manifest 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 of 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 of Research
During the past year, we have completed the construction of a 10 x 10 planer array of patch-clamp electrodes at a 100-micron 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 frontside deep reactive ion etch. After these steps, the silicon wafer possessed a 10 x 10 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 10 x 10 array of 25 µm in diameter trenches were etched to meet the front-side set, yielding one hundred apertures within the wafer. At this point, the silicon wafer possessed a 10 x 10 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 10 x 10 array of 25 µm in 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, as shown in scheme 1, the above-described interface is coupled to a PDMS-based microfluidic system and an electrochemically passivated platinum multi-electrode array. The microfluidic module is composed of two flow and two control layers stacked in an alternating fashion: 1) incoming flow, 2) lower-level control, 3) outgoing flow, and 4) upper-level control layers [6]. In the lower- and upper-level flow layers, the solution channels are 100 µm wide and 15 µm high except the central region where the 100 flow channels are individually directed toward the 10 x 10 array of electrode termini. Here, the channel widths gradually reduced down to 30 µm.

Each incoming flow channel in the lower level is aligned to a corresponding outgoing flow channel in the upper flow layer and vertical connecting channels are drilled with a laser prototyping instrument. Once the fluidic channels in both layers are filled with an intracellular solution, each circular metal pad in the electrode array is electrically connected to its corresponding top patch hole. Meanwhile, the lower- and upper-level control layers, with channel width and height 100 µm and 20 µm, respectively, contain integrated hydraulic valves enabling the electrical isolation
of individual flow channels in the lower (incoming)- and upper (outgoing)-level flow layers, respectively. By pressurizing the control channels (~ 10 psi) and thereby activating their hydraulic valves, the electrical resistance between neighboring electrodes can be increased to above 50 gigaohms. For the high fidelity of electrophysiological recording, a high seal resistance between the cellular plasma membrane and patch hole substrate is desired. In our completed device structure, when the flow channels are employed to apply negative pressure to aid in membrane sealing, the seal resistances we measure are typically > 500 MOhm for 90% of 100 electrode-channel-patch-hole assembly.

Thus far, initial experiments have shown the viability of the implemented multielectrode 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 with patterned arrays of rat hippocampal neurons.

References


Surface Plasmon Resonance Immunosensor for Quantification and Interaction Study of Outer Membrane Vesicles from E. Coli

CNF Project # 1379-05
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Abstract

Surface plasmon resonance (SPR) biosensors based on an antibody-immobilization were studied for quantification and interaction of outer membrane vesicles (OMVs) from E. coli. A homemade wavelength interrogation method SPR sensor was developed for real-time reaction analysis on gold surface. A monoclonal antibody was immobilized on the SPR sensing surface by streptavidin-biotin binding onto a mixed -COOH and -OH terminated self-assembled monolayer (SAM) of alkanethiols on a gold layer. SPR wavelength shift was measured during the binding of OMVs which were specific to the antibody immobilized on the SPR sensing surface. SPR wavelength shift was depend on the amounts of OMVs, and immobilized OMVs were visualized by green fluorescent protein (GFP) which is fused to cytolysin A (ClyA) and exposed to the surface of OMVs. The standard curve for OMVs was generated. A sandwich assay with anti-GFP antibody was demonstrated to evaluate the possibility of the SPR biosensor for monitoring the interaction of OMVs and the specific binding of the antibody to the immobilized OMVs. We believe that the reported SPR biosensor will find applications not only as a real-time analytical tool for bacteria-driven OMVs, but also a potential useful tool for the investigation on the mechanism of OMVs, providing further manipulation such as a therapeutic application.

Summary

The secretion via OMVs enables bacteria to secrete a diverse group of proteins and lipids in small proteoliposomes with an average diameter of 50-200 nm into the extracellular environment. In the case of pathogens, OMVs have been endowed for the transport of active virulence factors including adhesins, toxins and immunomodulatory compounds that permit vesicles to bind and invade host cells, induce cytotoxicity and modulate the host immune response. This potent role of OMVs opens another mechanism of gene transfer to encode virulence factors and antibiotic resistance among bacteria.

Surface plasmon resonance (SPR), an optical phenomenon occurring at the interface of glass and noble metal, is being widely researched for the detection of various biochemical reactions with advantages of sensitivity, rapidity, specificity and portability. SPR sensors are suitable for the reaction analysis near the surface. Another benefit of SPR sensors is that changes in resonance angle or wavelength can be monitored in real time unlike other analysis methods which can only analyze at the end point of reaction.

In this report, we exploit the SPR biosensor for detection and quantification of OMVs from E. coli. Biotinylated anti-E. coli antibody was injected over the streptavidin surface. PBS solution was used for running buffer to wash the unbound biotinylated anti-E. coli antibody from the streptavidin surface. Samples with different amounts of OMVs (from 5 ug to 40 ug) were injected over a biotinylated anti-E. coli antibody surface. There was no change in SPR wavelength after OMV injection, which indicates that OMVs bind to the antibody irreversibly. The standard curve for
OMVs was generated from the above results. The binding experiment of a sandwich assay with anti-GFP antibody shows the utility of the SPR biosensor in the interaction study by validating that ClyA-fused GFP is exposed to the surface of OMVs. To further extend our capability to target a broader origin of OMVs, different antibodies on the gold chip could be similarly employed. By virtue of their small size, adhesive properties, endotoxic component, and ability to carry and deliver toxic components into host cells, OMVs are intriguing targets for the therapeutic use as a delivery vehicle of DNA and protein. The SPR biosensor, with the advantages of sensitivity, rapidity, specificity and portability in real-time monitoring, can provide a facile tool to investigate and manipulate the prospective applications of OMVs.

References

**Microfluidic High Speed Thermal Stimulator**

CNF Project # 1404-05  
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**Abstract**

We have developed a microfluidic thermal chip that provides rapid temperature changes in the solution combined with accurate temperature control. The thermal chip was designed to facilitate the patch-clamp to study temperature dependent activities of ion channels. The device consists of a fluid channel for perfusing solution connected to an accessible reservoir for making patch-clamp measurements on individual cells. The thermal chip was constructed using SU-8 materials on glass wafer to minimize the heat loss to the substrate and channel walls. The chip is capable of changing the temperature ranging from a bath temperature of 200°C to 800°C at an optimum heating rate of 0.50°C/ms. To demonstrate the ability of the thermal chip we have conducted on-chip patch clamp recordings of temperature sensitive ion channels (TRPV1) transfected into HEK293 cells. The heat-stimulated currents were observed using whole-cell and cell-attached patch configurations. The results demonstrated that the thermal chip could be used as a thermal clamp for many thermosensitive ion channel studies.

**Summary**

Cell function and cellular activities can be influenced by many external stimuli including extracellular osmolarity, chemical, mechanical stress, voltage, as well as temperature. An accurately controlled stimulation to cells provides a means to study cellular processes and signaling mechanisms. In the past several years, thermo-nociception research has focused on imparting acute thermal stimuli to cells and studying the gating properties of ion channels in response to changes in temperature [1]. This requires a sharp gradient in temperature to be applied to a cell in order to accurately measure the temperature-gated channels response [2, 3]. Microfluidic devices offer the advantage of precise thermal input and because of the small volume, fast temperature changes [4].

The thermal chip consists of a fluid channel connected to a reservoir accessible for making patch-clamp measurements on individual cells. A thin film platinum heater was used to generate rapid temperature change. A separate thin film platinum sensor was placed in the flow channel near to the opening area. The heat generated from the heater is carried downstream to the testing region by the passing flow, and the temperature change at the cell location can be monitored using the resistive sensor.

The prototype thermal chip was constructed on the glass wafer. Three masks were designed and made for electrodes, fluid channels, and windows for conductive region. Using a lift-off technique, 2000 Å thick thin film platinum electrodes were deposited on the glass wafer by e-beam
deposition. The line width of the heater electrodes is 12 µm, and sensor electrodes 10 µm, which leads to resistance at room temperature of 102 Ohm for heater and 154 Ohm for sensor, respectively. In order to reduce the amount of interference between the heater and the patch clamp, a one micron oxide layer was deposited on the entire wafer using the low pressure chemical vapor deposition (LPCVD) method. The fluidic channel was constructed using SU-8 photoresist following standard protocol. The chip properties were characterized as a function of power ranging from 7.2 mW to 256 mW at various flow rates. As expected, a higher heater power produced both a greater temperature rise and a faster rate of rise (larger $dT/dt$). In addition, feedback may be used to improve the rise-time. Flow rate is the primary parameter that governs the temperature rise of the water. The chip was characterized for various flow rates ranging from 6.1 µL/min to 34 µL/min. Lower flow rates resulted in larger increases in fluid temperature due to the longer times that fluid elements remain in contact with the heater.

To demonstrate the performance of the thermal chip for patch-clamp experiments we examined TRPV1 transfected HEK293 cells. Using the chip, we observed the effect of temperature on the patch current in a cell-attached patch at different voltages. At -50 mV, increasing the temperature from 200°C to 480°C increased the inward current by ~ 4.5 pA. The patch-clamp studies showed that increasing temperature increased the conductance of the patch and of the open channel and the time constant of the patch’s response to temperature is much slower than the chip rise time permitting kinetic studies. Using the same approach, the thermal chip can be used to build temperature controllable reactors for various biochemical assays and chemical reactions.

**References**


Microchamber Studies of Cell Assisted Cell Growth in Microbial Colonies

CNF Project # 1457-06
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Abstract
CNF microfluidics are used to explore the means and manner by which cell colonies encourage growth within themselves. We are especially interested in uncovering how cells indicate their presence to one another and the pattern of growth that result.

Summary of Research
A central problem in biology is the manner in which collections of cells come to operate in a cooperative manner. Using microfluidic devices made with the help of the CNF we have been exploring over the past year the manner in which members of amoeba colonies signal each other’s presence in order to encourage growth. As indicated in Figure 1, we used PDMS on glass flow chambers in order to decide whether cells were passing molecular messages in solution. We were able to operate flow cells at such high flow rates that we should have been able to greatly perturb the diffusive transport of messages between cells by flushing signal molecule downstream. As reported in Ref. 1, we found that vigorous growth was not attenuated. We concluded that intercellular signaling was being performed in some other manner. With this view, we went on to develop a successful theory based on contact interactions between cells.

Recently, we have been using this same microchamber design to study the spatial-temporal patterns of growth in these social cellular systems. Figure 2 shows a snapshot of cells in a sparse colony. We have been applying novel measures of spatial distributions as a function of time in order to quantify growth pattern dynamics.

We are hopeful that our studies will assist in useful ways—for example in the problem of tissue regeneration and the control of tumors.

The master for soft lithography was produced at the CNF in the summer of 2005 by NBTC REU student Gustavo Abandon and NBTC graduate student Benjamin Smith.

Subsequent fabrication and research were performed by graduate students Albert Bae, Elijah Bogart (University of Utah) and undergraduates Wui Ip (Binghamton University), NBTC REU student in summer 2006 and Nathan Franck (University of Vermont and Cornell University).
References


Figure 2: Snapshot of amoeba cell colony growing in microchamber (field is about 200 μm across).
Fabrication of Branched, Multiple-Diameter Networks of Semicylindrical Channels in Silicon that can be used for Three-Dimensional Culture of Human Endothelial Cells

CNF Project # 1465-06
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Abstract

The majority of microfluidic devices employ networks of channels with rectangular cross-sections. At the microvascular scale of 30 to 300 µm in diameter, however, the distribution of fluid mechanical stresses and the induced shape of cultured cells will be quite different in a rectangular channel from the near-circular cross-sections seen in vivo. In this work, xenon difluoride isotropic etching of silicon was optimized for production of channels with semicircular cross-sections. This process was then used to produce a network of microvessel-scale semicylindrical channels, the diameter of which was decreased with each level of branching. Additionally, it was demonstrated that human endothelial cells will adhere to all surfaces of these channels, indicating that such chips may be useful in the future as in vitro models of the microvasculature.

Motivation

The goal of this project is to construct a network of microvessels on a chip the size of a microscope slide that can be used to study the interactions of circulating tumor cells with the microvascular endothelium in real time. Several researchers have made microvascular devices [1-4], but all such devices have employed square or trapezoidal cross-sections rather than the near-circular cross-section of physiological microvessels. Square channels impose widely varying fluid shear stresses on cells in different channel positions [2, 5] that can induce significant variation in cell physiology [6, 7]. It has also been suggested that the actual curvature of the cells has an effect on their physiology [8], and that the geometry of cell-cell interactions plays an essential role in the formation of metastatic tumors [9]. For these reasons, the first year of work on this project has focused on fabricating a network of channels with semicircular cross-sections on a microvascular scale (30-100 µm in diameter) and demonstrating that human endothelial cells will grow in these channels.

Description of Research

The initial year of this project has produced the first semicylindrical channels etched using xenon difluoride gas, a plasma-free gas-phase etchant that is compatible with all standard photoresists. This work has included four major phases.

First, the Xetch process was characterized to determine the effect of etch cycle time and number of cycles on channel diameter and circularity of channel cross-sections. We found that longer cycle times produced deeper, narrower channels than shorter cycle times, with 20 to 30 second cycles producing the most circularly symmetrical channels, a typical example of which is shown in Figure 1. The channel diameter seemed to exhibit a plateau effect with respect to increasing cycle time, but increased in a
nearly linear fashion with respect to number of cycles. We decided to use a fixed cycle time of 20 seconds, and to vary the number of cycles to determine channel diameter.

Second, a procedure was defined for re-coating the wafer with photoresist between etching steps that would effectively cover the previously etched topography. A wide range of resist thicknesses and spin speeds were tried, but no combination of parameters would consistently coat the previously etched channels. We finally determined that pre-coating the entire wafer with twice the recommended volume of a thin photoresist, initiating a test spin to evenly distribute, and ramping slowly to 1000 rpm produced an even coat that completely bridged the previous topography.

Third, a four-level branched network of microvessels was designed and produced, with each branch level decreasing in diameter to maintain the same fluid shear stress on the vessel wall. The device design, shown in Figure 2, requires six etching steps: one base layer to define alignment marks, four xenon difluoride etches to obtain the four different channel diameters, and a final deep Bosch etch to define fluidic connections. Etching times were determined from the process characterization performed previously, though the resulting features were approximately 10% larger than expected, indicating that etching is significantly pattern-dependent. An electron microscope image of several branched structures from a resulting device is shown in Figure 3.

Fourth, we have demonstrated the ability to culture Human Umbilical Vein Endothelial Cells (HUVEC’s) within our microvascular channels. Endothelial cells, the cells that line all blood vessels, require very specific surface conditions for adhesion and survival; hence, completed devices were coated with poly(D-lysine) and either fibronectin or type I collagen prior to seeding with HUVEC’s. Figure 4 shows a cross-section computed from a series of confocal microscope images that demonstrates HUVEC’s (live cells are white) adhering to all surfaces of the device.

References

Nano-Field Effect Transistor Device  
for Ultra-Sensitive Detection of Bio-Molecules

CNF Project # 1469-06  
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Abstract

A set of nano field effect transistors (nano-FET) were designed and fabricated with doped semiconductor nanowires (30-500 nm) on silicon/silicon dioxide wafers to optimize the best feature size. Synthetic peptide nucleic acids (PNA) as capture probes were immobilized on a polymer sensing surface. The interaction of antibody-antigen was converted into electronic signals through a novel molecular signal transduction process. The signal molecules captured on the sensing surface, which changes the electrical status of nano-FET and generates a detectable signal. Electronic impedance spectra was used to measure the signal. Enterotoxin B of *Staphylococcus aureus* was used as target molecule in the detection model. Detection sensitivity was achieved at 1-10 fM levels. Further investigations are necessary to improve the quality and yield of nano-devices. The final goal of this project is to develop portable nano-electronic detector for near real time bio-agent detection in the community and clinical environment.

Summary of Research

The recent advances in nanotechnologies have provided us to achieve a new sensing philosophy using nanowires-based transistors that merge chemical as well as biological events for silicon based devices (1-3). These nano-devices play a key role with regard to their sensitive behavior upon a surface bio-event. New research in the field of nanowires has provided evidence that nanowires-based methods (4) may be the next great leap in clinical tools. Recently, there has been an increasing demand for detection of specific nucleic acids and bio-threat agents. Most existent technologies for bio-molecular detection are optical detection (5), or depend on polymerase chain reaction (PCR) amplification. Labeling procedures and expensive equipment are required in these detection processes. The development of nanowire field effect transistor based biosensors opens a door for label free and ultra-sensitive bio-molecular detection.

The nano field effect transistor (FET) devices were designed and fabricated at the Cornell NanoScale Facility (CNF), Ithaca NY. Low doped nanowires (30-150 nm) were patterned using e-beam lithography on a silicon/SiO₂ substrate. The nanowire ends were then connected with patterned Ti/Au (15 nm/100 nm) layer leaving a 100 nm gap between two electrodes. The outline of the fabrication processes and scanning electron images of a nanowire are depicted in Figures 1 and 2. The inset of Figure 2 clearly shows the ~ 100 nm gap between two gold pads connected with ~ 30 nm nanowires.

![Figure 1: Flow diagram of fabrication step of nanowire based device.](image-url)
Electrochemical impedance spectroscopy was used to characterize nano-FET performance. The sensing surface of the nano-FET was modified using a self assembled monolayer on the bare metallic surface and polymer film by using short and long carbon chain organic sulfur and silicon compounds with carboxylic and epoxy tails. These –COOH groups serve as bonding sites for covalently linking to specific capture peptide nucleic acid (PNA) probes or antibodies. The remaining area of the surface was blocked to avoid non-specific binding. The modified surfaces were then verified with DAPI, a fluorescent dye, which binds specifically to nucleic acids and PNA. Oligonucleotide A20, as the signal molecule, was used to characterize the detection sensitivity of the nano-FET. The EIS bode plot of the detection of signal molecule with various concentrations showed a gradual decrease of impedance correspondence to a gradual increase electronic charge on the sensing surface of nano-FET (Figure 3). The detection sensitivity is \(10^{-16}\) to \(10^{-18}\) mole.

In the real sample detection, *Staphylococcus aureus* enterotoxin B was captured by a pair of anti-toxin B antibodies, and signal molecules were generated in the off-chip process. Signal molecules, hybridized with PNA on the sensing surface, generated a detectable electronic signal which reports the presence of toxin B. The EIS bode plot of enterotoxin B detection is shown in Figure 4. The detection sensitivity in this particular experiment was \(1.7 \times 10^{-16}\) mole. Optimizing detection conditions is under way. The data presented here demonstrated the electronic detection of toxin B using electrical impedance spectroscopic measurements at lower frequencies. Our results indicate a great promise of nano-FET device used in bio-molecular detection. Further investigation will focus on the improvement of nano transistor performance and yield. More bacterial toxin detection will be demonstrated.

We gratefully acknowledge the assistance of Rob Ilic and Michael Skvarla from Cornell NanoScale Facility at Ithaca, NY. This work was supported in part by the USDA grant CSREES 3447916054, NASA grant NNG06GB45G and HUD grant B02SPID0181.

**References**


Nanoscale Optofluidic Devices for Biomolecular Analysis

CNF Project # 1472-06
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Abstract

The recent interest in the development of nucleic acid biosensors and high-throughput screening techniques has been in part driven by the need to rapidly diagnose emerging viral threats. Currently the key challenges are to develop label free biosensors with low mass sensitivity and high specificity without sacrificing the extreme parallelism of the microarray format. In this project we will present our work towards the development of nanoscale optofluidic sensor arrays (NOSA), which represent a potential solution to this problem.

Summary of Research

Figure 1 shows an overview of the NOSA array. In the first stage of operation or Functionalization stage (Figure 1a), oligonucleotide probe immobilization is conducted in the nanowells by aligning the PDMS fluidics opposite the direction of the photonic resonators. Using this technique each resonator has an identical array of probes. Following conjugation the fluidics are removed and the screening stage is initiated (Figure 1b). During the screening stage the delivery fluidics are aligned and sealed and one sample is introduced per resonator. A successful binding reaction in any of the sensor sites causes the local refractive index to increase along with the effective optical length of the resonant cavity. Since all the wells have a different size, a change in any one or combination of the wells results in a unique shift in the resonant peak.

We are currently developing two forms of the NOSA resonator which will be discussed here. The first form is the inline resonator design which is shown in Figure 2. Figure 2a shows the inline NOSA resonator consists of a unique cavity design which comprises reaction wells of increasing size. When a change in refractive index in one of the wells is observed, the effective optical length...
of the cavity is increased along with the wavelength of the resonant modes. Because the size of each reaction well is twice that of the previous well, the amount that the peak shifts can be uniquely related to the combination of wells in which a binding reaction has occurred. In Figure 2b we illustrate the NOSA concept and determine the ultimate sensitivity of the device a series of finite difference time domain (FDTD) simulations were conducted. Here we show the strongest resonant peak from the multimode cavity. Figure 2c shows the shift in resonant peak due to a change in refractive index of 0.001 in select nano-wells. As can be seen, a change in refractive index in any of the wells increases the optical length of the cavity and thus the wavelength of the resonant peak. Since the nanowell sizes are selected such that each is double the size of its predecessor a refractive index change in any combination of the wells causes a shift to a unique location.

The second approach utilizes a 1D resonant cavity on a 1D waveguide with a unique differential size functionalization approach. Figure 3 shows a 4 by 5 array of these devices comprising 20 independent functionalization sites. This approach allows binding events at one or at a combination of the many sensing sites which causes a unique shift in the output resonator spectrum. The advantage of this approach is that it can very easily be multiplexed to perform massively parallel detections while still allowing for potentially sub-femtogram level detections.
Microfluidic Networks for Studying Thrombosis

CNF Project # 1476-06
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Abstract

Since blood is a moving biological fluid in vivo, it is important to study blood clot formation in vitro under physiological flow conditions. To achieve this objective, we have developed methods that meet the following criteria in an attempt to mimic in vivo fluid dynamics, molecular transport, and biochemistry: 1) flow channels with physiologically relevant length scales, 2) the ability to pattern surface molecules with spatial control, and 3) the ability to introduce soluble molecules with spatial and temporal control.

Summary of Research

Microfluidic channels are an ideal environment to study blood under flow because they can mimic the size, geometry, and fluid dynamics of arterioles, venules and capillaries. In this work, microfluidic devices were fabricated using standard soft-lithography methods [1]. Molding masters consist of KMPR photoresist on a silicon substrate. Our devices use a vacuum assisted bonding method to reversibly seal polydimethylsiloxane (PDMS) devices to glass and polymer substrates [2]. Figure 1 shows a representative microfluidic channel with a cross-sectional area of 100 µm x 100 µm surrounded by a vacuum chamber with an array of cylindrical posts. The posts act as a structural support so that the PDMS does not buckle during application of vacuum.

The recruitment of platelets to injured vessels is facilitated by exposure of surface bound proteins such as collagen, von Willebrand factor, and tissue factor. We adapted a microfluidic method [3] for patterning these proteins onto the surface of glass substrates with high spatial precision (~ 10 µm) within microfluidic channels. The micropatterned proteins mimic a small vascular injury where subendothelial collagen is exposed to flowing blood. Murine whole blood was introduced into microfluidic channels at arterial wall shear rates (1000 1/sec) and platelets were observed to adhere and aggregate only on patches of patterned collagen (Figure 2). Adhesion, rolling, and aggregation of individual platelets have been observed using high-speed fluorescence microscopy.

Figure 1: PDMS microfluidic channels and pillars.

Figure 2: DIC image of platelet adhesion and aggregation on micropatterned collagen.
Following adhesion to a vascular injury, soluble procoagulant factors are released from platelets (ADP, thromboxane A2) and generated on the surface of platelets (thrombin). The role of the flux of these procoagulant factors in thrombosis and clot stability is unknown. We have used a “membrane sandwich” multilayer technique [4] in conjunction with vacuum assisted bonding to introduce procoagulant factors at a controlled flux into flowing blood. In the device, one set of channels was situated perpendicular to a second set of channels and separated by a membrane which acts as a microfilter between the channels (Figure 3). The flux of the factors between the channels was controlled by concentration gradient, pressure gradient, and pore size. Figure 4 shows a fibrin gel formed by flowing thrombin through the bottom channels and human fibrinogen (3 mg/mL) in the top channel at a wall shear rate of 50 1/sec.

**References**


An Experimental Technique to Characterize Nano/Microscale Particles and Biomolecules

CNF Project # 1484-06

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Abstract

The purpose of this project is to develop an experimental technique which is based on Coulter counter particle sensing. The primary targets that we characterize are nano/microscale particles and biomolecules. A Coulter counter typically consists of two reservoirs separated by a membrane and connected by a single pore through that membrane. When an individual particle passes from one reservoir to the other, a current pulse is generated. The Coulter counter can measure the particle size and monitor its movement. Thus, we are pursuing how to optimize the Coulter counter in the nano/microfluidic platform and to apply it to biological and clinical areas.

Summary

This project presents an experimental technique to characterize nano/microscale particles and biomolecules, which is based on the Coulter technique of particle sensing [W.H. Coulter, U.S. Patent No. 2,656,508 (20 Oct. 1953)]. Coulter counters typically consist of two reservoirs separated by a membrane and connected by a single pore through that membrane. When an individual particle passes from one reservoir to another, a current pulse is generated. The Coulter counter can measure the size of each particle whose dimension are on the order of the pore dimension and monitor its movement. Thus, we are pursuing how to optimize the Coulter counter in the nano/microfluidic platform and to apply it to biological and clinical areas.

The nano/micromachined Coulter counter has been fabricated at the CNF to demonstrate this experimental technique. Fluidic components including reservoirs and pores are fabricated on Si wafers by wet/dry etching and sensing components are done on Pyrex™ glass wafers by lift-off and e-beam evaporation. Then both wafers are precisely bonded together by an aligner and an anodic bonder. The individual Coulter counter is connected with a world-to-chip fluidic interface to flow the target solution and Axopatch 200B to characterize the target.

A major technological challenge would be how to make the Coulter counter cost-effectively with nano/microfluidic components and how to reduce its noise level to characterize target. In addition, from a practical standpoint, a reliable world-to-chip fluidic interface is needed for easy coupling between macroscale in the real world and nano/microscale in this Coulter counter.
**Figure 1:** Experimental tool to characterize nano/microscale particles and biomolecules.

**Figure 2:** Experimental data showing current blockage.
Proteomic Technologies for the Analysis of the Disease Modifying Effects of IVIG Immuno Therapy

CNF Project # 1498-06
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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. Our goal is to develop a magnetic bead-based nanotechnology that can be used as a tool in the analysis of an array of cerebrospinal fluid protein biomarkers for Alzheimer’s disease. This technology would ultimately be used to build a GMR sensor that can capture, separate, and process these protein biomarkers from minute levels of complex biofluid with a high level of sensitivity and specificity.

Summary of Research

It is important to use separations as a front end to mass spectrometry (MS) in the industrial proteomics community [1, 2]. The use of such separations permits improved analysis of cerebrospinal fluid (CSF) proteins and facilitates the discovery and measurement of protein biomarkers. We recently demonstrated the important role the CSF proteins play in the early diagnosis of Alzheimer’s disease [3].

Our initial goal was to test and develop various chemistries for the reproducible attachment of proteins and antibodies to a silicon nitride surface. We tested two different modifications strategies based on epoxide and aldehyde linkages. We observed that the epoxide approach to protein attachment resulted in a more reliable and robust attachment that was suitable for protein-protein interaction studies using micron-scale magnetic beads.

We demonstrated the quantitative range of this technology in the study of transferrin, human IgG, and protein G in studies of pure proteins/antibodies as well as in mixtures of proteins. We also tested the effect of bead size and found that 2.8 µm beads (Figure 1) resulted in less consistent results than 1 µm beads (Figure 2) as shown for the case of Protein G detection of human IgG. The smaller bead size more reproducibly and quantitatively bound to the substrate.

References

Figure 1: 2.8 µm bead based detection.

Figure 2: 1 µm bead based detection.
Phenolic Based Molecular Glass Resists for Next Generation Lithography

CNF Project # 386-90
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Abstract
The idea of using small molecules instead of polymers for next generation lithography has enabled improved resolution and line edge roughness (LER). Rather than using polymeric materials, we are focusing on a new class of materials known as molecular glasses. These are low molecular weight organic materials that demonstrate high glass transition temperatures despite their modest size. Unlike polymeric resists, these molecules have the added advantages of distinct size and uniformity. We have synthesized a series of molecular resists containing rigid aromatic backbones and phenolic moieties. An increase in glass transition temperature is observed with increasing size and rigidity. Glass transition temperatures (Tgs) between 80-130°C have been observed for resists with molecular weights within the range of 500-900g/mol. These phenolic based resists also show the high sensitivity and sub-50 nm contrast required of candidates for next generation lithography.

Introduction
As the semiconductor industry moves to the 32 nm node and below, obtaining smaller feature sizes with reduced fluctuations on the resist pattern known as line edge roughness (LER) is a main focus [1]. As extreme ultra violet (EUV) lithography gains credibility as the next generation lithographic technology LER, sensitivity and outgassing remain crucial factors. The patterning target for the 32 nm node in 2009 is reported to be LER < 2 nm and sensitivity 2-5 mJ/cm$^2$. Hence, novel resist architecture and design strategies need to be introduced to successfully meet these requirements.

A recent advance in resist design has been the introduction a new type of molecular glass (MG) photoresist [2]. These materials combine the beneficial aspects of small molecules along with the favorable aspects of polymers. Like most organic molecules, molecular glasses have a well defined structure and purity. But unlike most small molecules they have a low tendency towards crystallization. These molecules are trapped in a kinetically stable amorphous state. Like polymers, they too demonstrate glass transitions (Tgs) significantly above room temperature.

This report sheds light on the challenges of designing a molecular glass photoresist for EUV lithography. When choosing a robust glass forming core, the molecular architecture is a very important parameter. This defines molecular flexibility as well as the packing ability of various molecular systems. Branched and star shaped molecules are well known glass forming molecules due to their topology. This paper introduces a new family of phenolic based bulky molecular systems with branched architecture. The phenolic component provides rigidity, etch resistance and base solubility due to the presence of the hydroxyl groups that can be modified with a solubility switching functionality.

Experimental Section
The synthesis of polyphenols was performed by the condensation of phenol with a ketone or aldehyde in the presence of hydrochloric and acetic acid [3]. By varying the aromatic core, several compounds were synthesized with increasing mass and phenolic content. The compounds were obtained in relatively moderate yields after column chromatography. These compounds were protected with tert-butoxycarbonyl (t-BOC) to varying degrees (50%-100%) by a standard base catalyzed reaction in the presence of 4-dimethyl amino pyridine (DMAP).

Results and Discussion
Our initial efforts on patterning of MGs were based on commercially available phenolic compounds such as alpha, alpha’,alpha’-tris(4-hydroxyphenyl)-1-ethyl-4-isopropylbenzene (CR 1) [4]. Patterns of 70 nm lines were obtained with EUV lithography. When compared with the calix[4]resorcinarene resists that have produced 30 nm resolution, the sub 100°C glass transition was identified as a limiting factor for these phenolic resists to attain sub 50 nm features [5]. Hence the structures (CR2-6) are based on a systematic increase of size and phenolic content around a planar benzene core.
Chemistry

Figure 1: The phenolic MG resist structures.

Table 1: Thermal properties of t-BOC protected phenolic MGs.

<table>
<thead>
<tr>
<th>MG Compound</th>
<th>% of t-BOC Protection</th>
<th>Phenolic Function</th>
<th>Mw (g/mol)</th>
<th>Tg (°C)</th>
<th>CO Ratio</th>
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<tr>
<td>CR1-66</td>
<td>66</td>
<td>2</td>
<td>624.8</td>
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<tr>
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<td>2</td>
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<tr>
<td>CR4-50</td>
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<td>2</td>
<td>778.9</td>
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</table>

Lithographic Evaluation

Phenolic MG resists were evaluated through EUV exposure at Albany Sematech RTC and Lawrence Berkeley National Laboratory (LBNL). The contrast curve was obtained for CR 6-50.

The EUV microexposure tool at LBNL is capable of fabricating fine features below 30 nm. For the compounds tested, the films were baked (PEB) at 75, 80, 85 and 90°C for 30s. The best result was obtained using a post-exposure bake of 80°C, developed in 0.26N TMAH solution. Although the PEB temperature was around the Tg of this resist material for CR3-50 sub 50 nm resolution was achieved. The LER was calculated using SuMMIT image analysis software.

References


Figure 2: Contrast curve of CR6-50 obtained at Albany Sematech RTC. PEB = 80°C, 30s, Developed in 0.26N TMAH. Sensitivity = ~ 10 mJcm⁻².

Figure 3: SEM images of EUV exposed resist CR2-50, dose 18.5 mJcm⁻², LER (3σ) 7.3 nm.
New Photoresist Platforms Developable in Super-Critical CO$_2$

CNF Project # 386-90
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Abstract

For more than a decade, the idea of using supercritical fluids in semiconductor processing has been actively explored by many researchers. Super-critical carbon dioxide (scCO$_2$) in particular is well-known as an environmentally benign solvent, but it also has many potential performance advantages. With its low critical temperature, zero surface tension, and non-polar inert character, scCO$_2$ shows great potential for its ability to process sensitive materials with patterned features on a very small length scale.

However, barring excessive fluorination, most materials traditionally used by industry show poor solubility in this solvent. This is where molecular glass resists show their unique nature. Because of their small size, molecular glasses have the potential for scCO$_2$ solubility while still showing equal performance to polymer photoresists [1]. Also, because of their small, discrete structures, these materials can show lower line-edge roughness compared to traditional polymer resists.

We demonstrate some recent successes in developing photoresist features in the sub-65 nm range with only scCO$_2$ as the developer solvent [2]. Additionally, we evaluate the effect of molecular structure on photoresist dissolution rate and show the consequences of protecting groups, glass transition, and pi-pi stacking on dissolution.

Experimental

The setup and use of the scCO$_2$ dissolution rate apparatus is described in a previous paper [3]. Briefly, a 1 cm by 1 cm piece of a silicon wafer coated with molecular glass film is placed into a 25 ml observation chamber, which is aligned with a laser and fitted with a quartz glass observation window for the laser beams to pass through. At the start of the experiment, CO$_2$ is quickly introduced into the observation chamber at the specific temperature and pressure. The reflected intensity of the laser on the film (at a measured incident angle of 3° from normal) is continuously logged until complete dissolution, when the reflected intensity from the bare silicon constitutes a steady maximum. For simplicity, the time from when the CO$_2$ is introduced until the time when the film is completely dissolved is taken to be the dissolution time. Comparison with the original film thickness allows one to calculate the dissolution rate with an estimated ± 3% observational error.

Patterning was performed by including 5 wt% (with respect to molecular glass) of a photoacid generator, triphenylsulfonium perfluoro-1-butanesulfonate, into the molecular glass solution before spin-casting. When exposed to ultraviolet or electron-beam radiation, the photoacid generator generates a proton that subsequently catalyzes the cleavage of tboc groups from the molecular glass. This renders the molecule relatively polar and therefore relatively insoluble in scCO$_2$. High-resolution patterning was performed with a Leica VB6-HR operating at 100 kV, followed by a post-expose bake at 90°C for 30 seconds to activate the deprotection. Development was performed in the same scCO$_2$ chamber used for DRM measurements.

Results

In general, the solubility of all molecular glasses should be dependent on their molecular weight. This effect may be seen in their respective dissolution rates, which are plotted on one master plot in Figure 1. For simplicity, the required pressure at 40°C for each molecular glass to reach a dissolution rate of 400 nm/min is plotted against molecular weight.
In general, for the majority of the data there is an expected linear dependence of dissolution rate on molecular weight. However, there are three outlying points in this case where dissolution is inhibited. Further inspection of the thermal properties indicates this may also be loosely correlated to material glass transition temperature (T_g). This observation suggests that these particular films still exist above their T_g when exposed to scCO_2, unlike the lower T_g materials that are plasticized to a T_g below the scCO_2 temperature. These correlations suggest that in situ T_g is a very important factor in predicting film dissolution rate in scCO_2.

Due to excessive plasticization of these particular molecular glass resist films under scCO_2, using them to form stable, high-fidelity patterns remains a challenge. As a result of their un-crosslinked nature, flow and pattern distortion happens quickly near the T_g. Therefore, only molecular glasses with very high T_g’s can be patterned and developed reliably with scCO_2. Of course, this limitation can be mitigated if the molecular glass is crosslinked upon patterning, as Shiraishi et al. have shown [4]. From the molecular glasses reported in this paper, hexa(hydroxyphenyl)benzene-tboc has been reliably patterned and developed without the assistance of crosslinking. The resulting pattern is shown in Figure 2. Success with this molecular glass is due to the high T_g of the material, which increases upon deprotection and leads to a patterned material that resists plasticization.

**Figure 1:** Correlation of dissolution rates with molecular weight. In general, most points follow a trend, with outliers due to specifics of molecular architecture.

**Figure 2:** Results of e-beam patterning of hxph-mp-tboc followed by development in scCO_2 at 40ºC and 300 bar. 150 nm lines and spaces are shown.

**References**


**Architectural Studies in High Resolution, Versatile Resists**

**CNF Project # 386-90**

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**Abstract**

Multifunctional atom transfer radical polymerization (ATRP) initiators based on sugars were utilized to grow short tert-butyl methacrylate (tBMA) arms. The kinetics of this system was studied using nuclear magnetic resonance (NMR) and gel permeation chromatography (GPC), and shown to be living. The polymerization was further engineered to obtain samples of controlled degrees of polymerization such that the polymer component only accounted for 55-85% of the total molecular weight of the hybrid molecule. A sucrose-based system was examined using thermal gravimetric analysis (TGA) and differential scanning calorimetry (DSC), demonstrating a clear architectural effect on glass transition temperature ($T_g$). Finally a sample engineered for high $T_g$ but low molecular weight was evaluated using e-beam lithography and scanning electron microscopy (SEM).

**Summary of Research**

Understanding the role of architecture on photolithographic performance will be crucial in designing new photoresists capable of meeting the requirements of the semiconductor industry for next generation lithography. Block copolymers have been successfully used as additives to enhance resolution [1] and as photoresists themselves to create patterns with sublithographic features [2]. Molecular glass photoresists [3] have also been examined as a viable alternative to conventional polymers [4]. This study lays the groundwork for creating hybrid photoresists containing molecular glass cores and polymeric arms. Hybrid photoresists can be used to study architectural effects on lithographic performance by varying the number of polymer arms ($f$) and the degree of polymerization of those arms ($n$). In the limit of $n = \infty$ the hybrid is a polymer, while in the limit of $n = 0$ the hybrid is a molecular glass. Polymer and molecular glass photoresists can be viewed, not as separate entities, but rather two ends of an architectural spectrum, as seen in Figure 1. Therefore a systematic study of architecture, its effect on properties and ultimately lithographic performance is possible.

Star poly(tBMA) has been grown from fully converted glucose and sucrose multifunctional ATRP initiators (with $f_{max} = \{5,8\}$ respectively) to study polyelectrolytes [5]. In this study, molecules with arms with a degree of polymerization an order of magnitude lower than previously reported were required to observe the hybrid photoresist in the limit as $n$ approaches zero. Kinetics studies were performed in order to obtain samples of the required degrees of polymerization. These were characterized using NMR to determine conversion and GPC to determine molecular weight and polydispersity. Molecular weight versus conversion plots (not shown) were linear—revealing that the systems were living. However the polydispersity was high and polymer arms with $n < 10$ were unobtainable due to rapid initial conversion. The sucrose system was engineered by modifying the reaction temperature, solvent volume and Cu(I):Cu(II) ratio. This prevented the rapid initial conversion of a monomer resulting in controlled conversion immediately upon initiation as demonstrated in Figure 2. GPC data indicated that the system was still living and samples had a polydispersity index between 1.05 and 1.10.

A series of hybrid resists were synthesized for $1 < n < 10$ utilizing the sucrose based initiating system. TGA and DSC were used to characterize the samples effectively mapping the effect of architecture on $T_g$ as the hybrid resist approaches the molecular glass regime as seen in

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Figure 1: Schematic of architectural spectrum. From left to right: segmented block copolymer resist, glass-coil block copolymer resist, glass-brush resist, molecular glass resist.
Figure 3. This result is significant as it demonstrates that high molecular weights can be achieved with very short polymer arms.

One of the higher T_g samples was selected for lithographic testing. The photoresist with five weight percent photoacid generator was spin coated onto a primed wafer, exposed to e-beam, developed and viewed with a SEM in Figure 4. Resolutions as low as 70 nm were achieved with 1:3 line spacing before the poor adhesion and insensitivity due to the resist being fully protected with tert-butyl groups became limiting. Further work is required to examine a partially protected resist in order to determine its ultimate resolution.

References


Patterning Organic Materials with Super-Critical CO$_2$ Soluble Photoresist

CNF Project # 775-99
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Abstract

The fabrication of functional devices from organic materials usually includes at least one patterning step, whether it be organic light-emitting diodes (OLEDs) patterned into pixels for displays, or organic thin-film transistors (OTFTs) which require patterning at the gate, dielectric, electrode and channel material levels. Traditional patterning techniques used on inorganic materials such as metals and silicon do not carry over well to organic materials because of the harsh chemicals used in developing and removing photoresist. In this work, we show that photolithographic patterning of these sensitive materials can be achieved using a specially prepared photoresist which is both developed and removed with super-critical carbon dioxide (scCO$_2$). Because scCO$_2$ has little effect on most organic semiconductors, this resist can be seen as a “gentle” alternative to traditional photoresists. Here we demonstrate patterning of both OLED materials and conducting polymers into structures suitable for OTFT electrodes. With further refinements in the process, a robust photolithographic patterning system capable of high resolutions and unlimited layering will be possible with a wide variety of chemically sensitive materials.

Summary of Research

Photolithography is widely used in the semiconductor industry to pattern a wide range of materials. Photoresist is spin-coated from an organic solvent onto a substrate. After exposure, development typically occurs with an alkaline developer and the resist is later removed with more solvent or oxygen plasma ashing. All of these steps (particularly the development and removal steps) are damaging to the materials used in organic electronic devices such as OLEDs and OTFTs. Because of this, photolithography has been eschewed by the organic electronics community, at least for the crucial task of patterning the active organic layer (metal contacts that can be put on first are usually done with photolithography). Yet photolithography is currently the best means of achieving high resolution, high throughput patterns with good registration over an arbitrary number of layers.

Previous attempts by our group to pattern organic materials with photolithography used the chemical vapor deposition (CVD) polymer parylene to make a mechanically peelable stencil mask capable of making features a least as small as one micrometer [1]. This technique has been used to good effect to make polymer contact transistors and also to pattern an ion transporting light emitting material between contacts in order to study ion motion in situ (see the report titled: Direct measurement of the electric field distribution in a light-emitting electrochemical cell). The technique is limited for multi-layer applications because of the parylene etch step involved. It is also problematic to peel parylene from small features without damaging them. This leads us to the present solution, which is to make a photoresist which is itself free from liquid developers and solvents.

Recently, a class of resists that are soluble in supercritical carbon dioxide (scCO$_2$) has been developed [2]. Both the development and lift-off steps can be accomplished in scCO$_2$ and the resists are patternable in 248 nm light. The resist used in this work is the random co-polymer P(FDMA-co-MAA). Figure 1 shows the fabrication steps for subtractive

![Figure 1: Schematic diagrams of subtractive and gap filling photolithographic patterning methods.](image-url)
Chemistry

patternning of an organic material as well as a second, “gap filling” method. In the subtractive case, resist is first spun on top of the organic layer (the fluorinated solvent used is agreeable to many organics). The resist is patterned in a manner similar to other negative photoresists, except the development step is done in a high pressure reactor with CO$_2$ at 4000 psi and 40°C. The organic layer can then be etched in the unprotected regions and the remaining resist is removed with further scCO$_2$ after exposure to HMDS. The conducting polymer PEDOT:PSS was patterned in this manner, as is shown in Figure 2. Patterning of this type can be used to make polymer electrodes for OTFTs, among other things.

The gap-filling technique was employed to pattern OLED materials. In this case, the PEDOT layer (used for hole injection into the OLED) isn’t etched. Instead, the openings formed in the resist allow the spin coated OLED material to make electrical contact with the PEDOT and underlying ITO layer. Since current can’t flow through the resist, the OLED only lights up in the patterned region. Figure 3 shows the OLED patterned into circles of three different sizes: 1 mm, 100 µm and 10 µm. With more work, this method should be extendable to three colors for display applications. We are also exploring a liftoff technique with the material which would allow for patterning of an organic layer without exposing it to the resist solvent.

References


Dielectric Fluctuations and the Origins of Noncontact Friction

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Abstract

Dielectric fluctuations underlie a wide variety of physical phenomena, from ion mobility in electrolyte solutions and decoherence in quantum systems to dynamics in glass-forming materials and conformational changes in proteins. We have shown that dielectric fluctuations also lead to noncontact friction. Using high sensitivity, custom fabricated, single crystal silicon cantilevers, we measured energy losses over poly(methyl methacrylate), poly(vinyl acetate), and polystyrene thin films. A new theoretical analysis, relating noncontact friction to the dielectric response of the film, is consistent with our experimental observations. This work constitutes the first direct, mechanical detection of noncontact friction due to dielectric fluctuations.

Summary

As part of our efforts to bring magnetic resonance imaging to the nanoscale [1], we have investigated the noncontact friction experienced when an ultrathin, highly compliant silicon cantilever is brought near the surface of a polymer film. We find, surprisingly, that the main source of friction experienced by such ultrasensitive cantilevers arises from thermal dielectric fluctuations in underlying polymer film [2, 3].

The cantilever used in this study was similar to cantilevers whose fabrication and characterization we have described previously [4]. We have used such cantilevers to detect nuclear magnetic resonance [1, 5] with record sensitivity, to detect electric spin resonance [6], and to characterize the magnetic properties of individual submicron magnetic particles [7].

The cantilevers used in the noncontact friction study can be seen in Figure 1. The cantilever has dimensions of 250 µm by 5 µm by 340 nm, a force constant of 700 µN, a resonance frequency of 7.385 kHz, and a mechanical quality factor in vacuum of approximately 31,000. The tip region of the cantilever was thinned from 340 nm to < 100 nm using a reactive ion etch. The cantilever tip had a radius of approximately 30 nm and was coated with a thin layer of platinum using a shadow mask technique [8].

We used the cantilever to probe noncontact friction at distances of 3 to 200 nm above thin polymer films, as sketched in Figure 2. The cantilever oscillates parallel to the sample, in contrast to the configuration of conventional atomic force microscopy. The sample is a polymer film of thickness 12 nm to 450 nm, spin-cast onto an epitaxial Au(111) substrate. The tip-sample voltage (V iter) was applied to the substrate while the cantilever is grounded. This applied voltage produced a charge on the cantilever tip which interacts with local fluctuating electric fields to produce a time-random force on the cantilever that, in turn, leads to friction.

Figure 1: Ultrathin, highly force compliant cantilevers for detecting dielectric fluctuations in polymer films.
An example of our findings can be seen in Figure 3. Here we plot the friction experienced by the cantilever, as inferred from its ringdown time, as a function of height over a 450 nm thick film made from three different polymers (poly(methyl methacrylate), PMMA; poly(vinyl acetate), PVAc; and polystyrene, PS. It is clear that the friction is dramatically different over the three materials.

Details of the measurement can be found in Reference [2] and a theory for the effect can be found in References [3, 5].

References


Microfluidic Mixing

CNF Project # 1278-04
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Abstract

Microfluidic systems present advantages and challenges relative to larger systems for performing chemical processes. They facilitate manipulation and analysis of small sample volumes and process parallelization within a small footprint. However, they generally suffer from inefficient mixing and low rates of interfacial mass transfer, due to the laminar flow and the long diffusive mixing times compared to the convective time scale of the flow. We study the effect of passive mixing structures on transport in these systems in the context of inter-stream mixing and mass transport from the fluid to a solid boundary.

Summary

Our research focuses on the understanding and optimization of mass transfer from microfluidic flows. This fundamental process is core to the operation of surface-based sensors, biochips, and electrochemical systems. We exploit a set of obliquely oriented grooves patterned on the walls of the microchannel to generate various classes of three-dimensional flows; this strategy is called the staggered herringbone mixer (SHM) [1]. We are using electrochemistry to characterize rates of mass transfer from these flows to reactive boundaries; we are also developing this system with Prof. Héctor Abreuña’s (Chemistry, Cornell University) to increase power and fuel efficiency in a microfluidic fuel cell [2].

For electrochemical applications, we work on glass substrates for insulation and optical access. The process of forming the groove structures starts with a 400 nm film of amorphous silicon (a-Si) deposited by plasma-enhanced chemical vapor deposition (PECVD) onto a 500 µm thick borosilicate glass wafer. Photoresist is spun and patterned to expose regions of the a-Si that are then etched with an SF6/O2 reactive ion etch to expose the glass substrate. The wafer is submerged in concentrated HF to etch the glass to a depth of 50 µm through the windows in the a-Si. The a-Si is then stripped with another SF6/O2 etch, revealing the grooved mixing structures. Due to the isotropic nature of the HF etch, the grooves are half-cylindrical with smooth walls (see Figure 1). To create a conductive and electrocatalytic surface, the structured glass surface is metalized: an adhesion layer of Ti (8 nm) and a seed layer of Au (150 nm) are deposited by electron beam evaporation. A 5 µm layer of electroplated gold is deposited to increase conductivity and improve the stability and durability of the metal surface during electrochemical operation.

To form an electrochemical cell, a silicone gasket is placed between two electrodes; the gaskets are produced by soft lithography in polydimethylsiloxane (PDMS). A master of the desired structure is photolithographically patterned in SU-8 on a silicon wafer. After silanization, the master is covered with PDMS and a flat glass plate is used to contact the SU-8 and produce a PDMS gasket of the desired thickness (~ 100 µm). Through-holes for the inlets and outlet are sandblasted through the glass electrodes and the system is assembled and pressure-sealed in an acrylic jig. The system can be run as a potential cell (electroactive species flow

Figure 1: SEM micrograph of structured electrode before electroplating.
over the electrodes, potential is applied and the resulting current is measured) or as a fuel cell (fuel flows over one electrode and oxidant over the other, producing a potential difference and current through a load). Experiments are currently being carried out in both systems, to elucidate the nature of transport to boundaries in the SHM flow and to quantify the enhancements in a system based on our collaborators’ microfluidic fuel cell [2]. Preliminary results with sodium ferrocyanide in the potential cell demonstrate increased scaling of current with the Peclet number $Pe$ at high flow rate ($Pe = Q/WD$, with volumetric flow rate $Q$, channel width $W$, and diffusivity $D$). Current scales roughly as the square root of $Pe$, rather than the cube root scaling expected from the case of a flat electrode surface [3] (see Figure 2). The fuel cell system shows increased current density and power output, demonstrating the system’s feasibility as a power source for consumer electronics and similar applications.

References


Nanostructure-Tailored Chemiresistor Sensor Arrays

CNF Project # 1345-05
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Abstract

This project focuses on the study of how design parameters influence the performance of sensor arrays constructed from nanostructured thin films and chemiresistor arrays. The chemiresistor sensor array consists of interdigitated microelectrodes fabricated using the CNF. The ability to tune interparticle spatial properties is essential for the design of sensing nanomaterials toward the desired sensitivity and selectivity for the detection of volatile organic compounds. The results have provided important insights into the detailed delineation between the interparticle spacing and the nanostructured sensing properties.

Summary

We have carried work in three closely-related areas of this project. First, we have studied several design parameters influencing the performance of sensor arrays constructed from nanostructured thin films and interdigitated micro-electrodes (IMEs) [1]. The nanostructured thin films on the IME devices were prepared from nonanedithiol (NDT) and mercaptoundecanoic acid (MUA)-linked assemblies of 2 nm sized gold nanoparticles. The sensor array data in response to volatile organic compounds were collected and analyzed using fractional factorial experimental design and analysis of variance for understanding effects of the design parameters on the sensitivity. While the smaller value for the microelectrode space, width, and length generally led to higher response sensitivity, a strong dependence on the nature of the nanostructured thin films was found. The microelectrode space was the most important design parameter for NDT-based thin films. The principal component analysis results for classification performances of the arrays consisting of a set of thin films have demonstrated the possibility of optimizing sensor arrays by appropriate selections of microelectrode parameters and nanostructured sensing films.

Secondly, we have studied the particle size and interparticle spatial effects on the electrical conductivity properties of molecularly mediated thin film assemblies of nanoparticles (Figure 1) [2], which is important for exploring the unique electrical properties in applications such as chemical sensors and biosensors. We investigated such effects for thin film assemblies of gold nanoparticles of highly-monodispersed sizes (2-4 nm) using alkyl dithiols of different chain lengths (0.8-2 nm). The conductivities of the thin films were measured using interdigitated microelectrodes as

Figure 1: An illustration of the molecularly-mediated thin film assembly of nanoparticles between microelectrodes (Cover page in J. Mater. Chem., 17, 457, (2007)).
the platform. Experimental results have shown that the activation energy increases with chain length and decreases with particle size, and the electron tunneling decay term decreases with particle size. The results have revealed that the experimentally determined conductivity and activation energy data for the nanoparticle films quantitatively match the calculations from electrostatic model of granular metals through the electron tunneling mechanism. The strong correlation between the experimental and calculated data was attributed to a combination of the high monodispersity of the nanoparticles and the uniformity and stability of the thin film assemblies. These findings have important implications for the fine-tuning of nanostructured thin films as chemical and biological sensing materials.

Finally, we studied the tuning of the interparticle spatial properties of nanoparticle assemblies for the design of sensing materials toward desired sensitivity and selectivity [3]. Molecularly mediated thin film assemblies of metal nanoparticles with controllable interparticle spatial properties were used as a sensing array. The interparticle spatial properties are controlled by a combination of difunctional alkyl mediators (X-(CH$_2$)$_n$-X) such as alkyl dithiols, dicarboxylate acids, and alkanethiol shells capped on nanoparticles. Alkanethiolate-capped gold and gold-silver alloy nanoparticles (2-3 nm) were studied as model building blocks toward the thin film assemblies, whereas the variation of alkyl chain length manipulates the interparticle spacing. The thin films assembled on an interdigitated microelectrode array platform are characterized for determining their responses to the sorption of volatile organic compounds (VOCs).

The correlation between the response sensitivity and the interparticle spacing properties revealed not only a clear dependence of the sensitivity on alkyl chain length but also the occurrence of a dramatic change of the sensitivity in a region of chain length for the alkyl mediator comparable with that of the capping alkyl chains (Figure 2). This finding reflects a balance between the interparticle chain-chain cohesive interdigitation and the nanostructure-vapor interaction which determines the relative change of the electrical conductivity of the inked nanoparticle thin film in response to vapor sorption. The results have provided important insights into the detailed delineation between the interparticle spacing and the nanostructured sensing properties.

References


Figure 2: The correlation between the response sensitivity and the interparticle spacing properties in response to three different VOCs.
Microfluidic Device for Combinatorial Chemistry

CNF Project # 1360-05
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Abstract

This work presents a microfluidic device that has the unique capability of automating combinatorial processes such as protein refolding and bioconjugate synthesis. Conventionally, these processes are done by hand-pipetting or using robotic systems. The microfluidic approach offers the advantages of automation, cost-effectiveness, compatibility with optical detection, and a million-fold reduction in sample volumes. Successful devices will greatly reduce the cost of realizing biopharmaceuticals.

Introduction

In this work, we develop a microfluidic device designed specifically for protein refolding applications. Protein refolding has been a bottleneck in the production of biopharmaceuticals on a large scale. In order to maximize the production of proteins, bacterial hosts are induced with promoters, resulting in the production of aggregated, over-expressed proteins. The process of refolding, which involves the conversion of these inactive aggregated proteins into their functional native state, is a highly empirical process. Determination of solution conditions to properly refold a protein is a combinatorial process which can be automated using our microfluidic device.

This microfluidic approach also has potential applications in bioconjugate synthesis for gene silencing. Short-interfering RNAs (siRNA) are small nucleotide chains which associate with the corresponding mRNA and suppress the expression of specific disease-causing proteins. Introduction of siRNA into cells needs a delivery vector. The combinatorial process of creating polymeric delivery vectors can be automated using our microfluidic device.

Summary of Research

Lysozyme refolding has been chosen as the model system for evaluating device performance, as its refolding protocols and activity assays have been widely developed [1]. We implement a combinatorial protocol using 16 refolding chaperones. The inactive aggregated lysozyme is solubilized in a strong denaturing medium (8 M guanidinium chloride, 1 mM EDTA, 50 mM Tris HCl and 16 mM dithiothreitol). This solubilized lysozyme along

with the denaturants is diluted in a renaturing buffer (50 mM Tris HCl, 1 mM EDTA, 5 mM glutathione, 2 mM dithiothreitol, 0.85 M guanidinium chloride) and a mixture of three artificial chaperones, chosen combinatorially. The 16 chaperones are used in two different concentrations and this results in a total of 4480 combinations.

Reagent aliquoting and mixing is achieved using microfluidic control [2]. The device consists of two layers of PDMS (poly (dimethylsiloxane)) on a glass slide. The schematic of the channel layout is shown in Figure 1. The layout consists of input channels for protein and reagent solutions, an annular mixer and an output channel for
the refolded protein. High pressure air is passed through control channels to control fluid flow in flow channels. The device is integrated with a control unit which enables automation of the processes (Figure 2).

Peristaltic pumps present on each flow channel allow precise control of the amount of fluid through the channel. The flow rate of the fluid in the flow channel depends on the frequency of actuation of the control channels. The volume injection rate as a function of the actuation frequency was calibrated by monitoring the dye-water interface (Figure 3). Dye-water mixing experiments performed at different frequencies resulted in a maximum flow rate of 1.07 nL/s. This corresponds to an actuation frequency of 25 Hz and a mixing time of 45s.

Preliminary experiments with protein solutions were performed on a three-input device (Figure 4). A solution of the solubilized lysozyme in denaturing medium was metered through one input. Solutions of urea (46.88 mg/mL) and guanidinium chloride (55.96 mg/mL), used as the artificial chaperones, were prepared independently in the renaturing buffer and were metered through the other two inputs. All the flow channels connected to the annular mixer were closed with control valves and the peristaltic pump on the annulus was actuated to mix the solutions. A solution of partially refolded lysozyme was obtained from the output channel.

Future work includes thorough calibration of the flow channels to meter precise fluid volumes into the mixer and scaling up of the device to include more chaperone inputs and combinatorial capabilities.

Conclusion

In conclusion, by integrating the control capabilities with the PDMS device, we can facilitate automation of combinatorial protein refolding protocols. The system is compatible with on-chip detection using terahertz spectroscopy [3], which gives the potential for real-time monitoring of kinetic intermediates.

References

Electroactive Nanowells for Concentration, Electrochemical Sensing and Spectrographic Microfluidic Memory

CNF Project # 1387-05
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Abstract

In this work we describe the development of electroactive micro-/nanowells which exploit highly localized electrokinetic effects in order to actively concentrate, confine, store and reject particles in well defined geometries. In this article we present experimental results demonstrating repeatable trapping and repulsion of polystyrene particles in wells ranging in diameter from 100 nm to 20 µm. Immediate applications include active cell trapping and concentration and unlabeled sensing and the development of a spectrographic microfluidic memory.

Summary of Research

The ability to deliver individual or small numbers of particles into confined geometries is of great importance for a number of biological applications (including high throughput pathogen detection, enhanced sensitivity for surface phase binding assays, parallel single cell trapping, interrogation and storage) and non-biological applications (including storage of information, directed or passive self-assembly processes and organic electronics). Within this broader set of devices, microwell arrays have become a popular tool for trapping and storage of particles as they enable discrete immobilization of cells without the need for surface binding chemistry (as the trapping site geometry is designed to prohibit trapped particles from dislodging easily) in a simple platform compatible with fluorescence microscopy.

Devices and Fabrication

Figure 1a shows a schematic of our “electroactive microwells” outlining the basic architecture. The electric field is applied between the upper and lower indium-tin-oxide (ITO) electrodes, and the wells are defined photolithographically in a polyimide (PI) dielectric. As can be seen, particles are driven, sensed and repelled from the well depending on the polarity. Microwells with sizes ranging from 6 to 25 µm were patterned on a 5 µm thick spin-on photoactive polyimide dielectric on top of a 8 Ω/square surface resistivity indium tin oxide (ITO) coated glass slide. An additional ITO electrode was inserted on top of the wells, separated from the polyimide by two 70 µm spacers, serving to enclose the channel structure and to close the electric circuit. Figure 1b is a finite element simulation of the electric field in the well.

Figure 2 shows our initial trapping experiments using a 0.1 mM phosphate buffer solution at pH 7 containing 1.9 µm diameter polystyrene particles. These particles were carboxyl modified, giving them a negative ζ-potential. It is expected that by applying a positive voltage on the bottom electrode, the PS beads will be driven from the bulk flow into the well, and they will be ejected by reversing the polarity. Figure 2 displays time lapse images of particle trapping and rejection in a 20 µm well for the case of a 5V applied DC bias and an imposed pressure driven flow of 30 µm/s (average velocity). During the attraction phase, Figure 2 (a-d), particles were drawn...
from the bulk solution into the well over 30 seconds. Here particles which were either initially positioned or convected into a region approximately three times the diameter of the well would be captured (we refer to this region as the “attraction basin”). However, particles which approached the well in a plane near the surface were observed to be repulsed from it in an upwards and radial direction, implying that additional electrokinetic effects aside from the expected electrophoresis have significant impact on the overall attraction process. When the polarity is reversed, Figures 2 (e-f), particles are repelled out of plane as they approach the well’s edge. Particles ejected from the wells form ring-like patterns, suggesting a strong repulsion force that points radially outward from the well’s center. The size of the ring shown in Figure 2f roughly coincides with the attraction basin, suggesting that particles decelerate due to both the drop in field intensity away from the well and the contribution of viscous drag.

Spectrographic Microfluidic Memory

Current optical storage devices such as DVDs have their read/write capabilities fundamentally restricted by the diffraction limit of light. We are also developing an optofluidic architecture for storing cocktails of colloidal quantum dots in electroactive nanowell structures. One application of these devices is the development of a fluidic memory approach which could enable the generation, reading and erasing of multiple bit information packages on single light diffraction limited data marks by spectral and intensity multiplexing of quantum dot cocktails (Figure 3). Here we focus on the development of the electroactive nanowell trapping architecture. Briefly, we have shown that by applying an electric potential between a top and bottom indium tin oxide (ITO) electrodes, microparticles suspended in solution can be attracted, stored and rejected from a targeted well structure by electrokinetic actuation. Nanowells 100 nm in diameter and 1 µm deep were fabricated by depositing silicon and a small oxide thin film on top of an ITO cover slip, patterning the wells on electron beam resist followed by a series of dry etching steps that leave the ITO substrate exposed in the well sites (Figure 4). When the quantum dots are electrokinetically transported to their sensing sites, they are then excited by a UV-blue light, and their discrete fluorescent signal is captured by a fiber spectrometer. Data erasure can be selectively performed by reversing the polarity of the field and ejecting the quantum dots from the nanowell data marks.

Figure 2: Time lapse illustrating particle attraction and repulsion of 1.9 µm polystyrene beads in a 20 µm well.

Figure 3: Conceptual overview of nanofluidic device and medium.

Figure 4: SEM of nanoscale electroactive nanowell site with feature size less than 100 nm.
Fabrication of Nanofluidic Filter Device for Surface Enhanced Raman Spectroscopy

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Abstract

We have fabricated a nanofluidic filter device that can reproducibly form nanoparticle-molecule clusters at a specific location for surface enhanced Raman spectroscopy (SERS). This device can provide much higher Raman signals with better reproducibility comparing to convention SERS approach. The device was fabricated on a Pyrex™ 7740 wafer using conventional photolithographic and etching techniques. With a micro-nanochannel pinched structure, SERS-active nanoparticles can be trapped to form the cluster which provides a reliable detection spot for SERS. Adenine was successfully detected with this device and the electromagnetic enhancement factor over conventional SERS technique was calculated to be $\sim 10^8$.

Summary of Research

To improve the reproducibility and sensitivity of current surface enhanced Raman spectroscopy (SERS) technique, we report a novel nanofluidic filter device that can trap nanoparticles and molecules at a specific location to form nanoparticles/molecules clusters with reliable and hot spot site. The nanofluidic filter device has a pinched micro-nanochannel structure, which enable trapping nanoparticles and molecules. Nanoparticles with a dimension larger than the depth of nanochannel can be trapped and aggregated into clusters at the entrance of the nanochannel. These nanoparticle clusters have turned out to contain hot spot area that can produce high SERS signals. Figure 1 shows the schematic diagram of side view of nanofluidic filter device and the mechanism of forming nanoparticle clusters at the entrance of the nanochannel.

The nanofluidic filter device was fabricated on a double-size polished Pyrex wafer with conventional photolithography and etching process. A shallow trench of 40 nm was first patterned and dry etched. This nanochannel is critical for entrapment of gold nanoparticles for SERS applications, as shown in Figure 2. As a hard mask for subsequent wet etching process, a thin oxide layer was deposited in a vapor phase. After the wet etch, the nanostructure was revealed by removing the oxide layer, resulting in the nanofluidic filter device with a pinched micro-nanochannel structure.
etching, 10 nm Cr and 50 nm Au layers were sequentially electron-beam evaporated onto the wafer. Then, a deep trench of 6 μm that is directly connected to the reservoirs as well as nanochannels was patterned and wet etched with concentrated hydrofluoric acid (HF 49%) solution. After removing the remaining Cr and Au layers, the inlet and outlet holes were drilled by the sand-blaster. For the final step, the glass wafer was bonded with a cover Pyrex wafer by clinging each other using de-ionized water.

Gold colloids, SERS-active substrates, with a diameter of 60 nm in aqueous solution, were blended with adenine molecules at a volume ratio of 5:1 and dispensed into the device through the reservoir. By the capillary force, the sample solution was pulled into the channel within a couple of seconds. The gold colloids are trapped at the entrance of the nanochannel and aggregated to form clusters. Then, the SERS signals were monitored as the function of time. From the measured Raman intensity, as shown in Figure 3, the electromagnetic enhancement factor of the nanofluidic filter device was calculated to be ~ 10^8.

In conclusion, we successfully demonstrated a nanofluidic device, which provides much higher Raman signal at a designated hot spot with better reproducibility. This approach can potentially be extended to detection of many other bio-molecules of practical importance, which are difficult to detect in a low concentration using the SERS technique.

References
Gallium Nitride Ballistic Electron Acceleration Negative-Differential-Conductivity Diodes for Terahertz Applications

CNF Project # 370-89
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Abstract
Electronic generation of terahertz (THz) signals using gallium nitride (GaN) is predicted to exhibit high efficiency. Theory by Ridley et al. [1] shows that electrons in an ultra thin (20-120 nm) epilayer of intrinsic GaN can be ballistically accelerated to negative mass states (under the influence of an applied bias voltage of 2.7 volts) with hot enough injection. If a majority of the electrons can be made to populate these states, negative-differential-conductivity (NDC) will be observed at the device terminals.

The heating problem is mainly a direct consequence of high operating current densities in the order of 1-2 MA/cm². These high current densities combined with finite parasitic resistances (i) causing permanent damage to contacts through catastrophic IR heating (ii) and causing a voltage drop across the i-layer diameter due to spreading resistance, which introduces field non-uniformity. If this voltage drop is too high, it will suppress the NDC effect. The fact that hot ballistic electrons dump their kinetic energy into the N⁺ region near the contact also contributes to the heating problem mentioned above.

These problems in our new design are addressed through a thicker intrinsic region which will reduce the current densities by a factor of sixteen while maintaining the ballistic nature of the transport, and our expectations are strongly supported by Wraback’s experiments [2, 3] which clearly show NDC at electric fields even below (electron transfer) threshold voltages at distances up to a quarter micron.

Summary of Research
Both GaN n⁺-i-n⁺ structures and structures with aluminum gallium nitride (AlGaN)/GaN launchers (for 0.38 eV injection) have been fabricated and characterized. However, no negative differential conductivity has been observed yet, mainly due to parasitics and related heating problems (Figure 1).

Figure 1: IV curves for a 5 µm diameter 30 nm i-layer diode (pulsed vs. continuous wave bias regimes).

Figure 2: New geometry vs. old geometry.
One other geometry to overcome the parasitics and heating issues has been designed and planned but not fabricated yet (Figure 2). In this design, the active region is 1 µm in diameter whereas the contact area is 25 times larger. This is accomplished by etching a conic shaped mesa using ion milling techniques. Concentrating the current in 1 µm diameter will help reduce the spreading resistance. In addition, a 25 times greater area will reduce the I2R heating at the contact which results in catastrophic failure. However there are high frequency performance concerns about this design due to the 2 µm mesa height it requires from the processing point of view and the relatively thin skin depths at those frequencies.

GaN epilayers for this research are grown on N⁺ SiC substrates in our own MBE lab. We process 15 mm² samples. First we dry etch the cylindrical mesas using the ICP tool reserved for III-V’s. Then we evaporate the ohmic contacts followed by the oxide deposition. To make contacts to the devices, the oxide is drilled first (with sloped walls for continuous metal coverage), and is followed by an electroplating process which lays out the probe pads in electrical contact with the device.

References


C-Doped Semi-Insulating GaN HFETs on Sapphire Substrates with a High Breakdown Voltage and Low Specific On-Resistance

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Abstract

High breakdown voltage (BV) aluminum gallium nitride/gallium nitride (AlGaN/GaN) heterojunction field effect transistors (HFETs) with a low specific on-resistance ($R_{D_{son}}$) were successfully fabricated using intentionally C-doped semi-insulating GaN buffers with a high resistivity on sapphire substrates. A high BV of ~1600 V and low $R_{D_{son}}$ of 3.9 mΩcm$^2$ was achieved from the fabricated devices with no field plate design. This result is almost touching the 4H-SiC theoretical limit and is a record achievement for GaN HFETs realized on sapphire substrates, to the best of our knowledge. Additionally, the effects of gate-drain spacing ($L_{gd}$) on device characteristics were investigated through device fabrication and characterization.

Summary of Research

Figure 1 illustrates the schematic cross-sectional structure of fabricated AlGaN/GaN HFETs with a C-doped semi-insulating GaN buffer on sapphire substrates. The GaN-based heterostructures were based on GaN grown by metal-organic chemical vapor deposition on c-plane sapphire substrates, given its cheaper price and larger wafer size than SiC substrates. The fabrication began with evaporated Ti/Al/Mo/Au (15/90/45/55 nm) for the source/drain ohmic contacts. The devices were annealed at 800°C for 30 s under N$_2$ ambient in a rapid thermal annealing (RTA) system. Next, the mesa isolation was performed by reactive ion etching with chlorine-based gas mixture (Cl/BCl$_3$/Ar). From on-wafer transfer-length measurements, an ohmic-contact transfer resistance of 0.5 Ω-mm and a sheet resistance of 540 Ω/□ were obtained. Thereafter, a 600 nm thick oxide insulation layer was deposited on the mesa-etched region to eliminate pad-to-pad leakage current paths and then was wet-etched. The Ni/Au (50/300 nm) gates were evaporated and lifted off. Finally, a 60 nm thick SiN was deposited at a substrate temperature of 300°C using plasma-enhanced chemical vapor deposition.

Figure 1: Schematic cross-sectional view of the fabricated AlGaN/GaN HFETs.
Figure 2: On-state DC characteristics of the fabricated AlGaN/GaN HFETs.
surface heat treatment (500°C/10 min) under N\textsubscript{2} ambient in the RTA system was performed for surface stabilization, immediately before the SiN deposition. Fabricated devices with source-gate spacing (L\textsubscript{sg}) of 2 µm, gate length (L\textsubscript{g}) of 2 µm, gate width (W\textsubscript{g}) of 250 x 2 µm and L\textsubscript{gd} of 5, 10 and 16 µm were prepared, and L\textsubscript{s} and L\textsubscript{d} were both varied from 7 to 40 µm to optimize device performance.

Figure 2 presents the on-state I\textsubscript{DS}–V\textsubscript{DS} characteristics of the fabricated devices. The devices showed excellent pinch-off characteristics and maximum drain current density (J\textsubscript{DS, max}) for L\textsubscript{gd} = 5, 10 and 16 µm were 460, 410 and 360 mA/mm under the gate-source voltage (V\textsubscript{GS}) of 1 V, respectively. It was also observed that L\textsubscript{gd} has a strong effect on AR\textsubscript{DS(ON)} here. Here, the device active area was defined by the mesa isolation process. The AR\textsubscript{DS(ON)} increased almost linearly with increasing L\textsubscript{gd}, and the measured AR\textsubscript{DS(ON)} values of L\textsubscript{gd} = 5, 10 and 16 µm were 1.6, 2.4 and 3.9 mΩcm\textsuperscript{2}, respectively.

Figure 3 shows the off-state breakdown characteristics of the fabricated AlGaN/GaN HFETs. Here, all of the breakdown characteristics were observed under V\textsubscript{GS} of −5.5 V and fluorinert FC-40 was applied on the surface of the fabricated devices for BV measurements to avoid problems with arcing and tracking due to environmental conditions [1]. A high BV over 1560 V was achieved on the fabricated HFETs with L\textsubscript{gd} = 16 µm. At this length of L\textsubscript{gd}, the average breakdown field along the channel was only 1 MV/cm, compared with the ideal GaN breakdown strength of 3 MV/cm. This means that the peak electric field is near the gate edge, and gives room for further material and process development. Furthermore, burnout marks were found on the surface of the device active area after destructive BV measurements. For L\textsubscript{gd} = 5 µm, burnout marks were clearly found between gate and drain. On the other hand, burnout marks for L\textsubscript{gd} = 16 µm were in the whole active area due to a relatively high applied bias between drain and source. However, the measured BVs increased clearly with L\textsubscript{gd} and were 400, 770 and 1560 V for L\textsubscript{gd} = 5, 10 and 16 µm respectively. It is suggested that the actual device breakdown was determined by the gate-drain breakdown, yielding BVs increased with L\textsubscript{gd}.

Figure 4 presents the BV–AR\textsubscript{DS(ON)} relations for the fabricated devices with L\textsubscript{gd} of 5, 10 and 16 µm. The trend of BV–AR\textsubscript{DS(ON)} showed a clearly linear relation in the log-log plot, suggesting that the device performance is very predictable with the variation of L\textsubscript{gd}. It was also shown that the results of this work were very reproducible, when compared with our previously published results [2, 3]. In particular, a BV over 1560 V and AR\textsubscript{DS(ON)} of 3.9 mΩcm\textsuperscript{2} was accomplished from the fabricated GaN-based HFETs realized on sapphire substrates and this result is even touching the 4H-SiC theoretical limit.

References

AlGaN/GaN High-Electron-Mobility Transistors on Different Substrates

CNF Project # 370-89
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Abstract

The performance of aluminum gallium nitride/gallium nitride (AlGaN/GaN) high-electron-mobility transistors (HEMTS) on diamond and silicon carbide (SiC) substrates is examined. Additionally, the temperature rise in similar devices on diamond and SiC substrates is reported. Recently, identical AlGaN/GaN HEMTs have been fabricated at Cornell NanoScale Science & Technology Facility (CNF) on diamond, bulk GaN, and SiC substrates.

Introduction

AlGaN/GaN HEMTs are well-suited to high-frequency and high-power applications [1, 2]. SiC is presently the substrate of choice for high-performance GaN HEMTs with a thermal conductivity an order of magnitude greater than that of sapphire. Bulk GaN is of interest as a substrate for the low dislocation densities in the epitaxial layer and an expectation of improved reliability. However, regardless of substrate, thermal limitations on device performance emerge under conditions of high bias and power drive [3]. Electron mobility has been observed to decrease in AlGaN/GaN HEMTs as a function of temperature rise, \( \mu \sim T^{-1.8} \) [4]. This decrease induces an increase in knee voltage which limits the dynamic range of large-signal operation.

Further advancement of GaN HEMTs for high-power applications requires reducing the temperature rise of the devices; the solution reported in this paper involves locating the device structure within close proximity to CVD diamond, which has a thermal conductivity 3-4 times that of SiC [5]. Group4 Labs has developed a method to atomically attach GaN epitaxial layers to polycrystalline diamond [5]. As previously reported [6] and supported by our results, the attachment process leaves the two-dimensional electron gas (2DEG) confinement layer intact.

Comparative experimental study of the HEMT operating temperature rise requires a localized technique. Scanning thermal microscopy involves the replacement of the AFM tip with a microscopic resistive filament, which acts as one leg of a Wheatstone bridge. Operating temperature measurements have been completed as a function of dissipated power density, in W/mm, on diamond and SiC substrates.

Summary of Research

The GaN-on-diamond material was prepared by Group4 Labs [5]. Material on GaN and SiC substrates was grown by General Electric and Northrop Grumman, respectively. The devices were fabricated at the CNF. The HEMTs on diamond substrate were fabricated exclusively via electron beam lithography, necessitated by bowing on the order of 50 \( \mu \)m over 1 cm\(^2\). To ease handling, the GaN-on-diamond wafer was mounted to a 15 mm x 15 mm carrier using Crystalbond 509 adhesive and dismounted before each processing step that exceeded 170°C temperature.

A standard Ti/Al/Mo/Au ohmic recipe was used, including a post-deposition anneal. Mesa isolation was achieved after ohmic contact anneal via an inductively coupled plasma (ICP) Cl\(_2/\)BCl\(_3/\)Ar etch. Finally, the devices were passivated with \( \sim 85 \) nm SiN\(_x\) deposited by plasma enhanced chemical vapor deposition (PECVD) at 375°C.

Transfer length method (TLM) measurements were performed via a four-probe technique using a Keithley 236 source measure unit (SMU). DC characterization was performed using an HP 4142 (Figure 1) and small-signal measurements were completed with an HP 8510 using Cascade on-wafer probes. Large-signal, class B, continuous-wave measurements were made using a Focus load-pull system powered by a traveling wave tube at 10 GHz (Figure 2).

Thermal measurements were performed on the HEMTs using a ThermoMicroscopes AFM-based SThM system with DC drain-source bias applied across one channel of the device. The 5 \( \mu \)m-diameter platinum (or Pt/10%Rh alloy) filament was connected to the Wollaston wire...
The probe tip was positioned in contact with the insulating SiN$_x$ layer atop the channel between the gate and drain. The finite temperature gradient in the SiNx is comparable for the devices on either substrate, as the passivation layers are the same thickness and were deposited at the same temperature. The thermal resistance of the devices on SiC was observed to be $\sim 12$°C/(W/mm), whereas using diamond substrate it was observed to be $\sim 6$°C/(W/mm) (Figure 3).

Ongoing work involves the characterization of identical devices on diamond, bulk GaN, and SiC substrates which have been processed at CNF.

References


Flat Panel Displays

CNF Project # 470-93
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Abstract

This project is concerned with glass substrates for flat panel display and electronics-on-glass applications. Photolithography and electron beam lithography were used to fabricate simulated particles on substrates down to 0.2 microns, which were then used to characterize metrology tools and to observe the effect of particles on film deposition processes. Exploratory work was also conducted in microfluidics for cell culture.

Summary of Research

This project is concerned with glass substrates for flat panel display and electronics-on-glass applications. As the design rules for devices in these technologies grow smaller, the sensitivity to particles of deposition and lithography processes on the substrates increases. Over the past year we have used the facilities of the Cornell NanoScale Science & Technology Facility to fabricate simulated particles on our substrates. Contact photolithography was used to replicate SiO$_2$ cylinders with diameters greater than two micrometers, and electron beam exposure of spin-on-glass in the JEOL 9300 was used for patterns with diameters down to 0.2 µm. These were then used to characterize metrology tools and to observe the effect of particles on film deposition processes.

Automated detection and measurement of particles on glass was found to be substantially more difficult than similar measurements on silicon. Developing methods for characterization of particles on glass substrates is an important topic, as coating studies show that the area of effect on film thickness and adhesion can be much larger than the initial particle size.

Future plans for this project include studying wet cleaning processes for glass substrates, followed by deposition of SiO$_2$ and Si$_3$N$_4$ thin films by both plasma enhanced chemical vapor deposition (PECVD) and low pressure chemical vapor deposition (LPCVD). These processes would help us to understand the coating influences on glass substrates with different compositions, as well as particle sensitivity. We will use this knowledge to continue developing novel thin film coatings on display substrates. It is believed that the processes we are developing at the CNF will enable the demonstration of new product concepts, and this technology will then be transferred to production.

Exploratory work was also conducted in microfluidics to study cells under a dynamic culture microenvironment in vitro. These approaches are being explored to study the effect of conditions on cell culture response and to better understand how cells respond to their microenvironment, in support of Corning Life Sciences. Microfluidic devices were fabricated from highly gas-permeable polydimethylsiloxane material by means of soft lithography. Standard photolithography methods were used to fabricate features in 10-500 micrometer range.
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Figure 1: 5 µm simulated particle fabricated in SiO₂ on a display glass substrate.

Figure 2: 5 µm feature coated with 500 nm of silicon resulting in local delamination.
Tera-Bit Metal Nanocrystal Nonvolatile Memories and Interfaces

CNF Project # 715-98
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Abstract

To realize nonvolatile information storage at the tera-bit scale, we have explored several promising approaches such as metal nanocrystal memory and molecular memory with mono-dispersed fullerenes. Besides its potential high bit-density, metal nanocrystal memory has the unique opportunity to achieve low program/erase voltage with robust reliability, which is otherwise not possible in the current Flash or SONOS technology. Moreover, our study of the fullerene-embedded memory also shed light on a feasible molecular interface with complementary metal oxide semiconductor (CMOS) circuits through a floating gate implementation.

Summary

A fast and reliable nonvolatile memory is an essential component of an information processing system and our research group is pursuing several promising approaches in this area. Previously we have demonstrated the feasibility of metal nanocrystal memory to lower the program/erase (P/E) voltage, improve cell density, and reduce P/E time while keeping retention time and endurance to the specifications used currently [1]. We have now built trustworthy models to understand the design space [2-3], using experimental data obtained with both MOS and carbon nanotube (CNT) channels [4] to calibrate the transport parameters such as effective mass and tunneling cross sections. With optimization strategies in both nanocrystal arrays and the heterogeneous gate stack, a metal nanocrystal memory design with 1.0 V memory window, 13 µs programming, 2.5 µs erasing and over 10-year retention time has been demonstrated at ± 4 V operation (Figure 1), which highlights the potential of nanocrystal memories as the next-generation nonvolatile memory.

In the course of this work we have also found that opposite polarities of charge storage happened in Au metal NC memories with different control oxides (Figure 2). The effective NC work function is found not only a bulk property of the NC, but also governed by the interface

Figure 1: The calculated program, erase, and retention characteristics of a scaled NC memory design operated at ± 4 V.

Figure 2: CV measurements of Au metal NC capacitors with Al₂O₃ control oxide and evaporated/PECVD SiO₂ control oxide.
with surrounding dielectric, as a result of the Fermi-level pinning [5]. This fundamental interface property should be taken into account in the selection of NC and dielectric materials for the NC memory optimization.

Driven by the ever increasing need for high density digital memory at low cost, we also have demonstrated the possibility of using carbon molecules in the form of fullerenes (C_{60}), also known as “bucky balls,” as an electrostatic non-volatile memory storage device [6]. We can employ C_{60} nonvolatile memory cells to experimentally program four C_{60} molecular orbital states, C_{60}^0, C_{60}^{-1}, C_{60}^{-2}, and C_{60}^{-3} (Figure 3). This was possible because the mono-disperse nature and molecular size of C_{60} results in a very sharp Coulomb staircase that can be observed at room temperature. A physical model based on the molecular orbit structure, the 3-D electrostatic charging energy, and the Fermi-level pinning theory yields good quantitative agreement with experiments. This result not only leads to a better understanding of the C_{60} molecular orbital structure and corresponding chemical redox levels, but could also potentially pave the way for realizing reliable multi-level molecular memories. Furthermore, this is the first demonstration that the CMOS circuit system can directly control and sense the redox states of nano-scale molecules.

References


Electrochemical Detection of Protein Interactions by Integrated Neuromorphic CMOS Sensors

CNF Project # 752-98
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Abstract

Electronic detection for protein microarrays holds high promise for autonomous sensor networks. Electrochemical platforms based on field effect transistors (FET) generally require large Ag/AgCl or Pt reference electrodes to properly set the bias points. Chemoreceptive neuron metal oxide semiconductor (CνMOS) with an extended floating-gate structure provides an integrated approach for protein detection without strict need for analyte reference electrodes. The readout FET biasing is set through the control gate that is capacitively coupled to the extended floating gate. Current-source active biasing can be readily achieved by an internal feedback circuit. With a covalent linking approach, CνMOS can detect the capture of streptavidin by biotinylated BSA in real time with a resolution of approximately 0.025 µg/cm² or 2Å changes in protein layer thickness. Both spectroscopic schemes between 1 kHz and 100 kHz and frequency tuning for high sensitivity can be performed. Through capacitive sensing, this approach eliminates DC charge transfer problems at the sensor interface, which is a common problem for existing electrochemical detection schemes.

Summary of Research

FET sensors have long been used in biosensing [1] and electrochemical detection [2]. Traditionally in the ion-sensitive FET (ISFET) structure, sensing is performed directly on the gate oxide or additional coatings, while a reference electrode such as Ag/AgCl or Pt induces the field effect through the analyte. The detection of protein binding using various tethering schemes has been reported with an ISFET-style sensor in impedance spectroscopy configuration [3]. Such efforts, though successful, prevent utilizing the full potential of CMOS devices in protein microarrays since scaling advantages of CMOS are overshadowed by the need for a relatively large, highly-stable reference electrode, whose presence is often invasive to the analyte for in vivo sensing as well.

Results

An impedance spectroscopic arrangement allows CνMOS (Figure 1) to operate without strictly requiring a reference electrode. CνMOS has fabrication similar to FLASH memory cells. A final passivation oxide etch exposes polysilicon sensing gate to microfluidics. Protein attachment was performed directly on silicon dioxide after treatment with (3-glycidoxypropyl)trimethoxysilane (3-GPS). This attachment scheme provides substantial simplicity over APTS, gold-thiol, biotin/avidin tethering techniques. Biotinylated-BSA was covalently bound to the 3-GPS.

Streptavidin acted as the capture analyte. Models of CνMOS predict increasing drain current with decreasing
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sensing gate capacitance [4] (Figure 2). To characterize the films, silicon wafers were used as witness substrates in the covalent attachment procedure and film quality was assessed by an ellipsometer. An AC voltage was provided to the control gate input and to the reference input of a lock-in amplifier. The drain current is transduced to a voltage with a transimpedance amplifier (TIA) and fed to both a lock-in amplifier and oscilloscope for analysis. When the streptavidin binds to the biotinylated BSA, the effective thickness of the protein layer grows and the analyte capacitance decreases, which can be monitored in real-time at a single frequency of high sensitivity surface (Figure 3). The final protein thickness equals 18Å, which corresponds to 69% surface coverage. A calibration curve can be constructed to relate captured protein mass from CvMOS output and control samples monitored by ellipsometer (Figure 4). Protein thickness can be converted to surface coverage of the layer of streptavidin. We can conclude that CvMOS can monitor 0.025µg/cm² or 2Å change in protein layer thickness with a dynamic resolution on the order of 50s.

References

Interdigitated Electrodes for Organic Solar Cells

CNF Project # 775-99
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Abstract

A new device structure is proposed for organic solar cells, whereby multiple organic semiconducting layers are deposited in between inter-digitated electrodes. It aims to have the multiple advantages of efficient light absorption, and charge carrier generation and collection. Inter-digitated electrodes have been fabricated using a wet-etch undercut method as well as a two-step lithography process. The first yielded small electrode gaps of ~ 200 nm between rough electrodes, while the second produced a gap of ~ 450 nm with much smoother electrodes.

Summary

Organic solar cells have attracted much attention in recent years due to their significant cost advantages and ease of processing and fabrication as compared to their inorganic counterparts (such as silicon cells). A major drawback of organic solar cells is their low efficiency, and a lot of work has gone into getting better efficiencies from these cells.

To achieve high efficiency, it is necessary to incorporate a heterojunction, which is an interface between a donor and an acceptor organic semiconductor material. Excitons created by photon absorption in either material diffuse to the heterojunction, which are then dissociated into electrons and holes due to the offset in the highest occupied molecular orbital/lowest unoccupied molecular orbital (HOMO-LUMO) levels of the two materials. Good light absorption, efficient exciton dissociation and charge carrier collection are all important for a highly efficient cell. The two most common device structures are the bilayer heterojunction and the bulk heterojunction, neither of which boasts all three of the above mentioned properties.

We propose a new “planar” device structure that incorporates all the above features. Such a structure consist of interdigitated electrodes of two metals with different work functions, with multiple donor and acceptor organic semiconducting layers deposited in between. Figure 1 shows a schematic of the cross section of such a device. Individual layers can be made as thin as necessary to facilitate exciton diffusion, while multiple layers can be deposited to increase light absorption. Finally, the charge collection will be driven by the electric field arising from the difference in metal work functions, and the carriers will have a connected pathway to the electrodes.

Two different methods have been employed to fabricate the interdigitated electrodes. In the first method, the first metal is patterned by lithography and dry etching (ion milling), followed by a wet etch undercut step. The second metal is then deposited, and unwanted metal is removed by liftoff.
This process is good for producing very narrow gaps (~200 nm), but the electrodes have very rough edges (Figure 2). The second method is simply a two-step lithography process whereby the two electrodes are patterned one after the other by liftoff. Much smoother electrodes are obtained (Figure 3), although the gap is significantly larger (~450 nm). Future work will involve the deposition of organic layers in between the electrodes, as well as solar cell testing and characterization.
Growth Study of Pentacene Thin Films and Transistor Fabrication

CNF Project # 775-99
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Abstract

We report the first-ever investigation in situ and in real-time of the structural evolution of organic small molecule thin films of pentacene during deposition by grazing incidence wide angle x-ray scattering. Small molecule films were deposited in a custom-built vacuum sublimation chamber which was fitted on the D1 beam line. These measurements have provided both in-plane and out-of-plane structural information about the growing polycrystalline films from the moment the growth begins. We report a number of interesting features in these measurements and find that the structure of pentacene films is essentially unaffected by the choice of the dielectric substrate. We also show morphological data from ex situ atomic force microscopy measurements and we measure transistor characteristics for these films.

Summary

A thermal silicon oxide layer was grown on a highly doped silicon wafer. The oxide layer served as the gate insulator, while the highly doped silicon acted as the gate electrode. The substrates were cleaned prior to deposition in an ultrasonic bath with deionized water, dried with filtered nitrogen and given a UV/ozone treatment. The samples where placed inside a custom-made deposition chamber with in situ x-ray scattering capability. A 220° Be window is fitted on the vacuum chamber which was mounted at the D1 station of the Cornell High Energy Synchrotron Source (CHESS). The pentacene was vacuum-sublimed from a Knudsen type cell. The thickness and growth rate were monitored using quartz crystal microbalance (QCM). After deposition, atomic force microscopy (AFM) was conducted ex situ in tapping mode using a DI 3100 Dimension microscope.

In this work, we investigated in situ and in real-time the structural evolution of organic small molecule thin films of pentacene during deposition by grazing incidence wide angle x-ray scattering (GIWAXS) [1, 2]. Pentacene films were grown on silicon oxide (SiO$_2$), Shipley 1805, hydrogen silsesquioxane (HSQ) and polymethyl methacrylate (PMMA). All of the above photoresists were spun onto the SiO$_2$. Analysis of the GIWAXS snapshots shows that the pentacene structure remains unaffected. In situ GIWAXS measurements are shown in Figure 1. Snapshots from an early and late growth stages are presented.

Figure 1: In situ GIWAXS measurements of a pentacene film grown on SiO$_2$ (a), (b) and Shipley 1805 (c), (d) Snapshots at different thickness are presented.
We fabricated top contact organic thin film transistors (OTFTs) with these pentacene films [3]. The top contacts were fabricated by gold evaporation using a shadow mask. The dielectric appears to influence the performance of the OTFTs. Figure 2 presents the current density vs. voltage characteristics of devices grown on SiO$_2$ and Shipley 1805. The hole field effect mobility of pentacene in the case of the plain oxide was 0.6 cm$^2$/Vs. On the other hand, the mobility of devices with Shipley was 0.01 cm$^2$/Vs. Shipley 1805 provided a suitable surface to successfully grow pentacene films that can be used to fabricate OTFTs, but their device performance was inferior to devices using silicon oxide as dielectric. A comparison of the AFMs acquired from films grown on these dielectrics is shown in Figure 3.

**References**


Direct Measurement of the Electric Field Distribution in a Light-Emitting Electrochemical Cell

CNF Project # 775-99
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Abstract
The interplay between ionic and electronic charge carriers in mixed conductors offers rich physics and unique device potential. One example of such a device is the light-emitting electrochemical cell (LEEC), in which the redistribution of ions assists the injection of electrons and holes from metal electrodes, leading to efficient light emission. We report the fabrication of planar LEECs and probe their operation using electric force microscopy. We show that obtaining the appropriate boundary conditions is essential for capturing the underlying device physics. To achieve this, we developed a patterning scheme that avoids overlap between the mixed conductor layer and the metal electrodes, allowing an accurate in situ measurement of the electric field distribution inside an LEEC. The measurements show that accumulation and depletion of mobile ions near the electrodes creates high interfacial electric fields which enhance the injection of electronic carriers.

Summary of Research
Electric force microscopy (EFM) allows direct microscopic determination of local potential [1]. The voltage applied to a metal coated cantilever is swept, producing a frequency shift which is parabolic about the local potential. Unfortunately, the sandwich-type configuration which is used in LEECs to minimize the thickness of the iTMC layer is not amenable to facile probing. To enable a direct measurement of the electric field distribution in LEECs with EFM, planar devices were fabricated in which a \([\text{Ru(bpy)}_3]^{2+}\text{(PF}_6^-)_2\) film was spin coated onto an insulating substrate with pre-patterned Au electrodes.

Figure 1 shows the distribution of the potential (A) in these devices. The thin solid trace, which indicates the first measurement after the application of a 5V bias, shows that the potential drops linearly as a function of distance between the two electrodes. The corresponding electric field is approximately constant, indicating that the \([\text{Ru(bpy)}_3]^{2+}\text{(PF}_6^-)_2\) film acts as a resistor. After one hour of continuous application of bias, during which steady-state is reached, the electric field (thick solid trace) shows a small enhancement near the cathode, accompanied by a slight decrease throughout the rest of the \([\text{Ru(bpy)}_3]^{2+}\text{(PF}_6^-)_2\) film.

Based on this electric field distribution it is not possible to unambiguously discriminate between the two models of LEEC operation, and hence it is not possible to pin down the underlying device physics. A closer observation of the data, however, reveals considerable changes of the potential over the anode. This phenomenon can be understood by the diffusion of the \text{PF}_6^- counter ions over the metal electrode. This action causes measurements on unpatterned planar devices to fail to capture the relevant device physics of LEECs.

In order to establish the same boundary conditions as in sandwich-type devices, the \([\text{Ru(bpy)}_3]^{2+}\text{(PF}_6^-)_2\) layer needs to be patterned to avoid overlap with the metal electrodes.

Figure 1: In situ potential profile across an unpatterned device.
This was achieved by using a variation of the parylene lift-off technique developed by DeFranco, et al [2]. Parylene is a chemical-vapor deposited polymer which offers conformal, pinhole-free coatings that adhere weakly to a variety of substrates, including freshly cleaned gold [2]. Planar devices were prepared using the process illustrated in Figure 2. In short, gold was deposited on an oxidized silicon wafer. A layer of parylene was then deposited on top and patterned by photolithography and reactive ion etching. An ion mill was used to etch the gold, with the parylene layer acting as an etch mask. A layer of [Ru(bpy)]$_2$(PF$_6$)$_2$ was then deposited from solution and patterned by peeling off the parylene with the help of adhesive tape. The advantage of this technique is that the [Ru(bpy)]$_2$(PF$_6$)$_2$ film is patterned in a self-aligned manner between the electrodes, having sufficient contact to inject electrons and holes, while never coming into contact with solvents or developers. A wide range of materials can be patterned in this manner, including vapor-deposited thin films.

Figures 3 and 4 show the distribution of the potential and the electric field, respectively, in devices with a [Ru(bpy)]$_2$(PF$_6$)$_2$ layer patterned with the technique outlined above. As with the unpatterned devices, the first measurement (thin solid trace), shows that the electric field is approximately constant between the two electrodes. After one hour of continuous application of the 5V bias, however, a large electric field enhancement is visible near the cathode, and a smaller field is located near the anode (thick solid trace). At the same time, the electric field in the bulk of the device is suppressed from 5.4 ± 1.0 kV/cm to 0.9 ± 0.8 kV/cm, a reduction by nearly a factor of 6. A small electric field is also apparent near the anode. A closer inspection shows that the potential over the electrodes is constant, indicating that the patterned samples effectively confine ions between the two electrodes.

The observation of high interfacial fields has important consequences for the design of LEECs. It explains the ability of these devices to function efficiently even when high work function (hence air-stable) cathodes are used. The high energy barrier at the cathode, which would normally prohibit efficient electron injection, is reduced in width by the interfacial electric field. Smaller counter ions can pack near the electrodes with greater density and produce higher electric fields at the electrodes that help inject electrons and holes more efficiently. On the other hand, if too many ions pile up near an electrode, the resulting electric field will be very high, leading to local electrochemical breakdown of the device. Such phenomena are not only important for LEECs, but for all solid state ionic devices. EFM measurements on planar and appropriately patterned devices can help to quantify these effects, understand the underlying physics, and improve the performance of mixed conductor devices.

References


Plasma Wave Terahertz Electronic Devices

CNF Project # 778-99
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Abstract
Terahertz (THz) plasma wave detectors using the high density 2-D electrons in AlGaInN/GaN-based quantum well heterostructure field effect transistors (QW-HFETs) with submicron single gates and multiple gate arrays have been fabricated to achieve high sensitivity, very fast temporal response and precise tunability. We have demonstrated detection of sub-terahertz and terahertz radiation by GaN HFETs in the 0.2 THz-2.5 THz frequency range (much higher than the cutoff frequency of the transistors) with the noise equivalent power (NEP) of $10^{-8}$ W/Hz$^{1/2}$.

Summary
Potential applications of terahertz (THz) technology in imaging, medicine, biology, space exploration, covert communications, compact radar ranges, industrial controls, THz microscopy, THz tomography, and homeland security require high sensitivity, high speed, portable and tunable THz detectors. Today, the most sensitive detectors such as Golay cells, pyroelectric detectors and bolometers are either very slow, not tunable and/or not portable. New ideas of using plasma resonances of two-dimensional electrons for tunable emission and detection of terahertz radiation have been proposed [1]. The theory in Ref. [1] predicts that such detectors should have a much higher sensitivity than more conventional terahertz detectors. Also, the plasma wave electronics terahertz detectors and detector arrays tunable by DC bias over a wide frequency range will detect not only the intensity but also polarization and direction of a terahertz beam [2].

The objective of this project was to develop novel GaN-based plasma wave electronics terahertz detectors and sources utilizing the high-density 2-D electrons in submicron HFETs. GaN-based HFETs are probably most preferable for fabrication of plasma-wave-based THz devices. The main advantage of such materials comes from the large value of built-in electric field induced by spontaneous polarization. Due to this field, AlGaInN/GaN-based HFETs have an extremely high sheet electron density (over a factor of 10 higher than that in comparable GaAs-based devices). Such high electron density results in a high velocity of plasma waves that allows to dramatically increase the operating frequency and to achieve operation in the THz range of frequencies for much larger sample sizes. In the course of the project, we designed and fabricated devices with different broadband antenna geometries. Figure 1 shows a plasma wave HFET with monolithically integrated coplanar log-periodic antenna.

The typical response of the 150 nm gate length device to sub-THz and THz radiation is shown in Figure 2. Above 70K, only non-resonant detection was observed. At lower temperature, resonant detection was found. Figure 2a and Figure 2b show the response, $\Delta V$, of the transistors to 0.76 THz and 2.5 THz radiation, respectively, as a function of the gate voltage. The resonant detection reveals itself as...
a shoulder or maximum on the monotonic background of the non resonant detection. We attribute this behavior to the resonance detection of THz radiation by plasma waves. One of the most important parameters of the detectors is the Noise Equivalent Power (NEP), which can be found as \( N/R_W \), where \( N \) is the noise of the transistor in V/Hz\(^{0.5} \) and \( R_W \) is the responsivity. Figure 3 shows the NEP for different transistors at 4K and at 300K for different frequencies of radiation. Since detection was studied at zero bias, the noise was taken equal to the thermal noise. As seen from the Figure 2, at frequencies \( f < 0.76 \) THz, the NEP is of the order of \( 10^{-8} \) W/Hz\(^{1/2} \) or smaller. This value is slightly higher than for such commercial detectors as Golay cell, pyroelectric detectors and Schottky diodes, having meantime the potential advantage of operation at very high sampling frequency of several tens of gigahertz. At \( f = 2.5 \) THz, the NEP is higher because of low responsivity at that frequency. The level of sensitivity can be further improved by utilizing multi finger gate arrays in FETs and optimized antenna structures [3].

We also fabricated GaN lateral heterodimensional Schottky diodes (HDSD) formed between 3D metal and 2DEG, and studied their THZ response. The performance of conventional Schottky diodes used for detecting THz radiation is limited by the RC product. The advantage of HDSD is a small junction capacitance, and hence, much higher cutoff frequencies than for a conventional Schottky diode. High concentration of the 2DEG in the channel of the device allows for high forward current (200 mA/mm at 2 V) and relatively low series resistance. Figure 4 shows the response to the 200 GHz radiation. The position of the maximum is determined by the tradeoff between the maximum nonlinearity of the diode and match of the load resistance to the resistance of the diode. As seen, the position of the maximum shifts to lower voltages with the increase of the load resistance. The strongest response was observed when the diode operated in the open circuit configuration. The detection was observed in the frequency range from 200 GHz with the noise equivalent power (NEP) of \( 10^{-9} \) W/Hz\(^{1/2} \) up to 2.24 THz with \( \text{NEP} \approx 10^{-4} \) W/Hz\(^{1/2} \) [4].

References

Fabrication of Sub-22 nm Bistable Devices

CNF Project # 1205-04
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Bistability in 500+GHz sub-22nm quantum dot gate InGaAs-InP FETS for next generation analog and digital circuits for advanced radars and communication systems

This project describes a methodology to design and fabricate digital and RF circuits (requiring lower voltage and significantly reduced device count than conventional CMOS designs) via the use of novel quantum dot gate InGaAs-based 500+ GHz Field-Effect Transistors (FETs), which will exhibit three states in a similar manner to the quantum dot gate Si FETs recently fabricated by our group. Project Sponsor: Office of Naval Research

Information Theoretic Foundation of Molecular Computing

Performance Limits and Design Optimization: This project investigates usage of nanostructure devices for realizing computing systems. The objectives include building molecular logic blocks and memory unit based on quantum dot nanodevices fabricated in our UConn laboratory. Fault tolerant high parallel non-deterministic nanoarchitectures are being investigated. Project Sponsor: National Science Foundation. [With L. Wang.]

A mosaic layout with the integration of FETs and memory devices is being developed to create advanced nano-architecture to implant molecular computing systems.

Bistable Quantum Dot Gate Field-Effect Transistors Exhibiting a Multi-State Operation

A Novel Approach to Reduce Device Count in ICs: This project aims at designing and fabricating Si sub-22nm FETs having 3-state transfer characteristics for multi-valued logic and analog systems. The circuit implications of 3-state devices include lowering number of devices used to achieve a given functionality. Project Supporter: National Science Foundation.

CNF e-beam, RIE and other facilities are being used to implement a design file that will enable fabrication of sub-22nm Si FETs. We are currently working with Daron Westly to show the feasibility of various steps. This work is in progress.
Fabrication of Si Set Transistors for Electrical Metrology and Ultra-Sensitive Electrometry

CNF Project # 1248-04
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Abstract
This project entails fabrication of silicon (Si) nanotransistors with multiple levels of gates, for two purposes: 1) Silicon single-electron tunneling (SET) devices at low temperatures, and 2) Electrostatic sensing of charge recongurations in fluid at room temperature. We are fabricating these devices on silicon on insulator (SOI) wafers; with back gate, the active layer, two layers of poly-Si gates, and metallization, the process flow has about 60 steps in it. So far, we have made two sets of wafers which have shown promising results, although they are clearly far from optimum.

Summary of Research
Single-electron tunneling (SET) devices, which are based on Coulomb blockade physics, offer the amazing possibility of moving electrons one-by-one. For NIST, these devices offer the potential to form fundamental standards of current, capacitance, or charge [1].

SET devices have been made in a variety of materials, including metal (Al/AlO/Al), semiconductor (Si and GaAs), nanotubes, granular materials, and etc. Si-based SET devices [2] offer some advantages, including both the potential for higher speed as well as a substantial reduction in the problematic “charge offset drift”. This latter problem refers to the unpredictable random fluctuation in time of the operating point of the device [3], which makes it difficult or impossible to integrate or parallelize these devices. We have shown that, in Si-based SET devices, this fluctuation is reduced by a large factor, of order 1000 [4].

The basic device element for SET devices is a quantum tunneling barrier. In many Si-based SET devices, the tunnel barrier has a fixed value of conductance, set by the material architecture. We have shown that Si-based SET devices in which the tunnel barrier is produced and controlled electro-statically by a “finger” gate (a finFET), the device has better performance and in particular better homogeneity and uniformity [5].

The project at CNF is to fabricate such devices with two levels of gates, lower finger gates and a large upper gate—see Figure 1 for a schematic. Figure 2 shows both a top-

Figure 1: Schematic of single-electron tunneling multi-gate device.

Figure 2: Left; top-down view before upper gate fabrication. Right; cross-sectional view after complete fabrication process.
down view of the active single-crystal region plus the finger gates, as well as a cross-sectional view of the full device; the purple line on the left indicates the region where the cross-sectional view was taken. These micrographs indicate that this many-step process is working reasonably well, for both the Si layers as well as the oxide isolation layers.

In terms of the electrical performance, our major problem to date is a gross leakage problem at room temperature between many of the leads and the back gate. We have identified this as being due to an over-aggressive etch during the metallization part of the process; we plan to eliminate this in the new wafers. In addition, we are also having problems independently biasing all of the finger gates. This may also be due to the leakage problem to the back gate; we are trying to diagnose this now.

Also, given the leakage problem, we were especially pleased to find that at low temperatures the SET performance is reasonably good. Figure 3 shows a “diamond diagram”; this plot of stability regions demonstrates that we have a single dominant SET island which controls the electrical characteristics. This diagram is fairly good, but the lack of exactly regular peak spacing along the $L_{GS}$ axis is another indication of less-than-optimum performance.

Given this leakage problem, we were pleased to find that at low temperatures the SET performance is reasonably good. Figure 3 shows a “diamond diagram”; this plot of stability regions demonstrates that we have a single dominant SET island which controls the electrical characteristics. This diagram is fairly good, but the lack of exactly regular peak spacing along the $L_{GS}$ axis is another indication of less-than-optimum performance.

Also, given the leakage problem, we were especially pleased to find that, just as in previous Si-based devices, the charge offset drift problem appears to be quite small in these devices. Figure 4 shows a plot of the charge offset, in units of the electronic charge $e$, measured over many days.

In the coming year, after training a new user, we hope to fabricate multiple sets of wafers in which we have removed the problem of leakage to the back gate; we will then continue our low-temperature SET measurements and assess the performance of the new wafers.

**References**


Ballistic Transport Investigation and the Ballistic Deflection Transistor

CNF Project # 1301-04

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Abstract

In this work, we have been investigating the properties of non-linear ballistic transport and optimizing the design of a novel device, the ballistic deflection transistor (BDT). The electron steering effect required for an operational BDT has been demonstrated. A T-branch junction is also investigated.

Research Summary

The BDT utilizes a novel non-linearity first discovered by Song et. al. [1, 2], and is best understood as a shift in energy created by an artificial scattering mechanism rather than a PN junction. We are currently developing a transistor based upon this non-linearity in combination with electric gates that direct electrons to the appropriate portion of the artificial scatter. 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 enable 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. Figure 1 presents a scanning electron micrograph (SEM) of a recently fabricated design.

The fabrication of the device begins with the growth of an InGaAs-InAlAs heterostructure on an InP substrate by molecular beam epitaxy. 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 and a thin layer of SiO\textsubscript{2} followed by patterning using PMMA resist. Exposed areas are subjected to a CF\textsubscript{4} plasma that removes the SiO\textsubscript{2}, followed by an oxygen plasma that removes the carbon, creating a transfer mask. The etch is achieved using an ion mill. Lateral gates are formed from the semiconductor and are patterned at the same time as the rest of the structure. The contact layer is then formed using Ni-Ge-Au contacts.

Figure 1: SEM image of a ballistic deflection transistor.

Figure 2: Left and right output voltage response to a push pull voltage.
This structure will have 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 general structure of the transistor is essentially a differential pair rather than a single on/off switch, and this increases the compactness of subsequent circuits. 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 as such, very low voltage operation is possible (below 100 mV) with noise being the limiting factor. Recent measurements have demonstrated the steering effect, however gain has yet to be achieved (see Figure 2). We are currently modifying the design to reduce the induced built-in potential of the etched regions near the gates. This built-in potential has been shown to be impeding the steering effect and is the cause of the low gain at this time.

In addition to the transistor structure, we are investigating the fundamental properties of ballistic transport. Here we focus on one such device called the T-branch junction (TBJ) [3]. It has a large nonlinear input-output transfer function on the order of unity. Recent experiments [4] indicate that the intrinsic nonlinearity persists at high frequencies, to at least a terahertz. Therefore the nonlinear current mechanism of the TBJ could in principle be used to build electronic circuitry at unprecedented high frequencies. An SEM picture of one of our TBJs is shown in Figure 3 and typical electrical results are shown in Figure 4. We see that when a push-pull voltage \( \pm V \) is applied, the central probe voltage \( V_c \) is always zero or negative. If the TBJ were ohmic / diffusive, we would expect \( V_c = 0 \) at all \( V \). But to a good approximation, \( V_c \) in Figure 4 can be described as zero for \( V < 200 \) mV, a straight line with slope near \( \pm 1 \) for larger \( V \), and a gradual transition region between. The \( \pm 1 \) slope implies that \( V_c \) is to some extent pinned to the voltage at which electrons enter the T-bar, from whichever side. Also, the current through the top of the T is proportional to \( V \) (apparently ohmic) until it appears to partially saturate at the same transition region. This correlation between current saturation and onset of negative \( V_c \) is observed with all device geometries and temperatures. This strongly suggests that the two phenomena are caused by the same mechanism.

Our experiments and device structures have demonstrated that non-linear devices can be fabricated using ballistic transport. TBJs exhibit a near unity voltage-in over voltage-out response. The BDT has shown that it is possible to create a steering effect by using lateral fields without depleting the channel; as well it has been shown that the artificial scattering objects can perform as an active load in this device. In the near future, we expect to produce a BDT with gain.

References


Fabrication of SOI-Based Nanowire Sensors

CNF Project # 1353-05
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Abstract

Silicon nanowire sensors are fabricated from the active silicon layer of silicon-on-insulator (SOI) wafers and used for label-free sensing of specific proteins. A fabrication method is demonstrated which avoids the integration difficulties inherent in a bottom-up approach, without the decrease in carrier mobility usually associated with reactive-ion-etched nanowires. Nanowire devices are used to demonstrate the label-free detection of below 10 femtomolar concentrations of specific proteins.

Summary

Label-free sensing of specific proteins using semiconductor nanostructures is an important technological breakthrough that has been demonstrated recently, owing to improvements in the ability to fabricate semiconductor nanostructures. However, most if not all of the results published thus far have suffered from low-carrier mobility, resulting in reduced sensitivity, as in the case of most top-down fabricated nanowires, or they have not been easy to integrate into a complementary metal oxide semiconductor (CMOS) process, usually the case for “as-grown” nanowire devices [1-8]. We demonstrate a process to fabricate silicon nanowire metal oxide semiconductor field effect transistors (MOSFETs) with near bulk-mobility and using these devices, demonstrate label-free sensing of proteins.

Using electron-beam lithography with a combination of wet- and dry-etching techniques, we have successfully realized nanowires down to a few tens of nanometers in width on ultra-thin silicon-on-insulator wafers. The resulting nanowires are contacted using optical lithography.

Figure 1: SEM of SOI nanowire device [9].

Figure 2: $I_d, V_g$ at $V_{sd} = 1V$ for typical nanowire device [9].
and electrically characterized. Process details are reported in [9]. Figure 1 shows a scanning electron micrograph of the silicon nanowire portion of a typical device. The devices are back-gated using the SOI handle and have a threshold voltage of ~ 20 volts with a sub-threshold slope of ~ 1V. Figure 2 shows a typical drain-current versus gate-voltage characteristic. Field-effect hole-mobility is typically around 100 cm²/V-s for p-channel devices.

Silicon nanowire MOSFETs have been used to demonstrate sensing of a variety of different analytes. Un-functionalized nanowires are used as hydrogen ion sensors to measure pH of a solution, as shown in Figure 2. The steps represent pH values varying from 6.0-8.0. Sensing of specific proteins is also demonstrated using the familiar biotin-streptavidin system, as shown in Figure 3. The device is a p-channel nanowire FET, and streptavidin, a negatively charged molecule, is added at t = 0. Sensitivity below 10 femtomolar concentration is achievable. We are currently applying this new technology to a wide range of chemical and biological systems.

References
Morphological Evidence for Surface Pre-Melting on Silicon (111)

CNF Project # 317-87
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Abstract

We present what we believe to be the first morphological evidence for the occurrence of surface pre-melting on the Si(111) surface. Our results complement the extensive previous evidence from diffraction and ion scattering techniques for the presence of pre-melted (liquid-like) layers on Si(111) below the bulk melting temperature, and also suggest how atomic steps are involved in the initiation of such layers. Our results are based on atomic force microscopy studies of morphologies that are preserved when surfaces are annealed in a range of high temperatures and then rapidly cooled to room temperature for observation.

Summary of Research

Silicon (Si) surfaces, especially those vicinal to (001) and (111), are among the most studied surfaces due to their technological importance and to the rich set of phenomena occurring in these quasi two-dimensional systems. Control of the morphologies of these surfaces at the atomic level is vital for such applications as layer-by-layer growth of epitaxial overlayers or assembly of nano-scale devices.

High temperature annealing is extensively used to obtain chemically clean and flat surfaces, but even atomically smooth vicinal surfaces may contain a rather dense array of atomic steps. These atomic step distributions can be altered by lithographic patterning and annealing [1-3]. We have developed techniques to create large areas with no or widely spaced atomic steps on patterned silicon surfaces by direct current (DC) heating in ultra-high vacuum (UHV) [2-5]. A temperature gradient was intentionally induced by deliberately clamping some samples tighter on the lower side so that we could investigate the influence of a continuous range of temperatures on the same sample [4]. A unique feature of the experiments is the use of specially prepared atomically flat or very low step density surfaces; this allows us to see how the liquid-like morphologies are associated with the steps and also allows the high temperature structures to survive the cooling process without being absorbed into the steps, which normally would exist on a surface vicinal to (111).

The most interesting structures were observed in those regions of the samples with a holding temperature in the range 1200-1220°C, in which range the surface melting is known to initiate on surfaces vicinal to Si(111) (see for example [6]).

Figure 1 shows an atomic force microscopy (AFM) image of a set of steps which connect two step bunches (bright bands in the figure). Figure 2 shows elongated islands that extend from the edge of an otherwise step-free region which is actually a sublimation pit formed within one of the craters of the patterned region [2]. The most striking feature of these islands is their liquid-like morphology. Although they are extended, presumably due to electromigration, in the horizontal direction, which is also the direct current direction, they actually form all the way around the step-free region. AFM height measurements show that these protrusions also have a total height of 0.8-0.9 nm, i.e. ~ 3 Si bilayers. To try to get some clue to the
crystallinity of the structures seen in Figures 1 and 2(a) we have performed an RCA chemical processing step (SC1); this is known (see for example [7]) to preferentially etch away the surface oxide (by the OH- anions) and to produce a conformal new oxide layer over the whole surface (by the HOO- ions). After this etch, the height associated with the features in both Figures 1 and 2(a) was reduced to ~ 0.3 nm while their shapes were fully preserved, as shown in Figure 2(b). This implies that the two upper layers differ markedly from the lower layer, which is presumably well ordered. We can also speculate that exposure to oxygen more readily converts the disordered material to oxide.

We believe that the morphologies represented by these images provide evidence that the initiation of the surface melting transition in the 1200-1220°C range occurs selectively at atomic steps, or step bunches that surround extensive terraces. The liquid-like protrusions of Figure 2, which seem to have formed at the periphery of the terrace, are probably fed by atoms detaching from the edges and migrating rapidly across the liquid layer. For regions of the samples with holding temperatures slightly below 1200°C, the structures observed after quenching are similar to those reported earlier in our own studies [2] of Si(111).

Arrays of small islands, presumed to be due to adatom aggregation [2], are observed on (111) terraces at distances greater than about 50 nm from atomic steps; there are however no structures that can be ascribed to surface melting. For regions of the samples with holding temperatures above ~1220°C, the surfaces of the quenched samples again show no features that can clearly be associated with the surface melting transition. At these temperatures the quasi-liquid layer probably covers most of the Si(111) surface.

Quenched-in structures ascribed to pre-melting also act as sinks for diffusing ‘excess’ adatoms generated by the (1 × 1) to (7 × 7) transition [8] and this leads to the formation of dendritic islands one atomic bi-layer in height (Figure 3).

**References**


**Figure 2:** (a) AFM image showing liquid-like elongated islands extending from the edge of a crater. (b) The same region imaged as in Fig. 2(a), but after the sample was chemically cleaned (etched) following the standard RCA procedure.

**Figure 3:** Dendritic islands resulted from the ‘condensation’ of excess adatoms on preexisting protrusions.
Development of Molecular Glass Photoresists for Next Generation Lithography

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Abstract

In order to meet the growing demand of the electronics industry for smaller, higher resolution features, much attention has focused on next generation lithographic technique, such as extreme ultra violet (EUV) or e-beam lithography. Complementary to this field of research is the design of novel photoresist materials to produce sub-50 nm feature sizes. Chemically amplified molecular glass (MG) photoresists are among the most promising alternatives to traditional polymeric materials. A number of compounds which possess a rigid aromatic backbone were synthesized in our lab and evaluated for electron beam and EUV lithography. Herein, we describe the synthesis and lithographic evaluation of partially tert-butoxycarbonyl (t-Boc)-protected bulky phenol ‘CR1-60’. The resist CR1-60 is characterized by high glass transition temperature ($T_g \approx 130^\circ$C) and good film-forming properties.

Summary of Research

As the semiconductor industry moves to ever smaller feature sizes, extreme ultra violet (EUV) lithography is emerging as one of the most promising next-generation lithographic techniques for the production of sub-50 nm size features [1]. Photoresists designed for this wavelength must be more sensitive, in order to compensate for the typically low power of EUV light 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 photoresists has been the introduction of molecular glass (MG) resists [2]. Unlike conventional polymeric photoresists, MG resists are materials made up of small, discrete molecules rendered amorphous by functionalizing rigid core structures with bulky side groups [3].

In this report, we describe our work to develop a chemically amplified MG resist based on a bulky phenolic molecular structure (Figure 1). The structurally bulky phenol, CR-1, was prepared by conventional wet-chemistry processes and further protected with tert-butoxycarbonyl (t-Boc) group to produce a chemically amplified resist, CR1-60 [4]. Lithographic evaluation of CR1-60 was performed employing the tools at the Cornell NanoScale Science and Technology Facility (CNF) and Lawrence Berkeley National Laboratory (LBNL). The resist, CR1-60, was dissolved in propylene glycol mono-methyl ether acetate (PGMEA) containing triphenylsulfonium nonaflate as a photoacid generator (PAG). The resulting resist formulation was spin-coated onto a hexamethyldisilazane (HMDS)-primed wafer. No crystallization of the coated film was observed even after storage for several months, indicating the stability of the resist’s amorphous nature.

Positive-tone imaging properties were then tested under both deep UV (CNF) and EUV (LBNL) exposure conditions. These data are shown in Figure 2. CR1-60 resist
is highly sensitive to UV light at 248 nm. A clearing dose of 0.32 mJ/cm$^2$ ($E_0$) was found experimentally. A post-applied bake (PAB) at 130°C was strongly required to ensure good quality of the resist film. SEM images demonstrated that sub-50 nm resolution could be achieved under EUV exposure conditions. A post exposure bake (PEB) at 80°C combined with the use of 0.26 N TMAH solution was a suitable condition to produce high quality patterns.

In conclusion, t-Boc protected MG resist, CR1-60, was synthesized and examined as a positive-tone resist for the next generation lithography. The bulky phenolic material has been found to be amorphous with a high enough glass-transition temperature ($T_g$) and a good alternative to conventional polymeric photoresists thanks to its uniformity in size and structure. Sub-50 nm features were produced successfully after patterning with EUV lithography.

References


High Resolution, Versatile Block Copolymer Resists

CNF Project # 386-90

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Abstract

Our group has been working on a unique block copolymer, poly(α-methylstyrene)-block-poly(4-hydroxystyrene), PαMS-b-PHOST, which self-assembles upon spin-coating to form 20 nm cylindrical PαMS nanodomains in a matrix of PHOST. When small amounts of a photoacid generator (PAG) and crosslinker are added, the PHOST block can be crosslinked like a negative tone photoresist, and the PαMS cylinders may be thermally removed within the crosslinked area to create a periodic pattern of sub-lithographic lines or holes. This combination of top-down lithography and bottom-up self assembly is seen as a possible solution for patterning beyond the 22 nm node.

Summary of Research

Two distinct patterning methods use polymers for defining the elements of integrated circuits: the widely established process known as photolithography, and the emerging technique of block copolymer lithography. Photolithography is the process of forming a latent image by selective-area exposure of thin films of a photoactive polymer, causing either increased solubility (in the case of positive tone resists), or decreased solubility (in the case of negative tone resists) [1]. In contrast, block copolymer lithography relies on a self-assembly process and selective block removal to form the resist latent image [2]. Two polymer systems capable of being patterned by both of these methods are demonstrated here with self-assembling diblock copolymers in which the majority component block is a photoresist.

In the case of poly(α-methylstyrene)-block-poly(4-hydroxystyrene), PαMS-b-PHOST, cylindrical PαMS minor phase polymer spin-coated from PGMEA solution onto Si substrates self-assembles with cylinders perpendicularly oriented but without hexagonal packing [3]. When small amounts of a photoacid generator and crosslinker are added, the PHOST block behaves as a negative tone photoresist, and the PαMS block may be subsequently removed due to its low ceiling temperature.

Recently we have found that solvent annealing facilitates long range ordering at room temperature prior to crosslinking. The orientation of the pattern relative to the substrate – either parallel or perpendicular – can be controlled by the choice of solvent without additional substrate treatment. Figure 1 shows an atomic force microscopy (AFM) height image of the block copolymer film annealed in acetone.
Materials

The inset Fourier transform indicates a well-ordered hexagonally close-packed structure of vertically oriented cylinders. In the SEM shown in Figure 2, we demonstrate the potential of this technique as a nanofabrication tool by inducing highly ordered parallel orientations of the cylindrical nanodomains on lithographically pre-defined structures, further enhancing the nanofabrication potential of block copolymer self assembly.

Another system, poly(methylmethacrylate)-block-poly (2-hydroxyethylmethacrylate), PMMA-b-PHEMA, is being developed at this time for inclusion into a 193 nm lithography process [4]. In this case, PHEMA can be used in the same manner as a 193 nm photoresist with the inclusion of photacid generator and crosslinker, and the PMMA can be selectively removed to create a template of periodic holes or lines.

Additionally, we have shown through cross-sectional transmission electron microscopy (TEM) that polar PAG particles tend to sequester into the polar phase PHOST domain. Poly(styrene-block-hydroxystyrene) (PS-b-PHOST) was synthesized anionically as a model amphiphilic block copolymer to test its compatibility with ionic triphenylsulfonium hexafluoroantimonate PAG. The high atomic number of the Antimony within these PAG molecules enables optical contrast between the microphase separated domains, as seen in Figure 3. Being able to control the location of the PAG molecules through directed self-assembly means that we are able to effectively concentrate the PAG molecules where they are needed for crosslinking in the PαMS-PHOST system, and it provides an additional handle to control the photochemistry in other self-assembling photoresist systems.

References


Figure 2: Within a lithographically pre-defined trench, parallel-oriented cylinders form after annealing in Tetrahydrofuran.

Figure 3: PAG sequesters preferentially to the majority (hydroxystyrene) phase in PS-b-PHOST.
Thin Film Nucleation and Growth using Energetic Neutral Species

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Abstract
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. Pentacene is a promising candidate for applications in organic thin-film electronics owing to the ability to form highly ordered thin films with excellent electrical properties near room temperature. In this work we make use of both ex situ techniques, such as atomic force microscopy, and in situ techniques, such as synchrotron x-ray radiation.

Summary of Research
Organic thin-film field-effect transistors (OFETs) are of great interest because of their potential to offer light-weight, flexible, low-cost alternatives to devices currently utilizing the traditional silicon-based transistors. Among different organics being studied for applications as gate dielectrics in OFETs, pentacene has emerged as the leading contender owing to the ability to form highly ordered thin films near room temperature with excellent electrical transport properties [1, 2]. However, in order to control/improve the field effect mobilities demonstrated by pentacene thin films, it is essential to understand and control the nucleation of pentacene on the substrates. The nature of interface formation of pentacene on a substrate that can subsequently affect the electrical properties of the organic film may be controlled in two ways. One is by modifying the energy of the incident pentacene molecules, i.e., thermal (evaporative) vs. hyperthermal (supersonic beam) deposition; and second is by controlling the chemical environment offered by the substrate itself by depositing an interfacial layer prior to pentacene deposition. In order to achieve the former we have been carrying out the deposition of pentacene via energy tunable molecular beams. To control the latter, we have utilized inorganic substrates modified by interfacial organic layers such as those of hexamethyldisilazane (HMDS), HN[Si(CH₃)₃]₂, for pentacene deposition. 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]. For these experiments, approximately 300 nm thick silicon dioxide (SiO$_2$) films were grown by wet thermal oxidation at 1100°C and cleaned subsequently with UV-ozone. This gave a clean and reproducible hydrophilic surface. These samples were exposed to pentacene molecular beams in the UHV chamber. Following deposition, atomic force microscopy was conducted ex situ in tapping mode using a DI 3100 Dimension microscope at CNF.

Regarding the ex situ results we have obtained so far, 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. Regarding substrate modification, we have used interfacial organic layers of HMDS, HN[Si(CH$_3$)$_3$]$_2$, on SiO$_2$ for pentacene deposition [4]. Similar to growth on clean SiO$_2$ 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 SiO$_2$ 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 SiO$_2$. As these micrographs are acquired ex situ, some rearrangement of the layers post-deposition is possible.

The use of in situ x-ray radiation eliminates artifacts associated with post-deposition effects. It also can provide an incredible amount of information concerning the dynamics of growth. We have examined the growth of pentacene thin films on SiO$_2$ using supersonic molecular beam techniques and in situ real-time synchrotron x-ray scattering [5]. At low-to-modest rates of deposition [ca. 0.001 to 0.1 monolayer (ML)-s$^{-1}$] we observe an abrupt transition from 2D, layer-by-layer growth to 3D, islanded growth after deposition of the first few monolayers of pentacene on SiO$_2$. As may be seen in Figure 1, the oscillations in the intensity at the anti-Bragg condition are much longer lived at the highest growth rate (2 ML-s$^{-1}$), and a highly textured film possessing significantly flatter and smoother surface morphology is also formed for these conditions. Concomitantly, in this regime we also observe new polymorphs of pentacene (not shown), as significant changes occur in the crystalline structure of the thin films. The transition to this anomalous, yet promising growth behavior corresponds very strongly with the onset of the formation of van der Waals clusters of pentacene within the supersonic expansion.

In subsequent experiments, we have directly verified the presence of clusters using quadrupole mass spectrometry. We suggest that the presence of clusters in the incident flux acts to change the dynamics of growth due to a variety of reasons, including their effects on the step edge density, which in turn can affect the rates of interlayer transport.

**References**


**Figure 1**: X-ray intensities measured in situ at the anti-Bragg position (001/2) vs. time for the growth of thin films of pentacene on SiO$_2$ at three deposition rates: 0.008, 0.8, and 2 ML-s$^{-1}$. Atomic force micrographs of thin films of similar thickness (~ 35-40 nm) and histograms of height distribution obtained from micrographs at these same three rates are shown in the central and right-hand panels, respectively. $T_s = 30^\circ$C.
Low Heat Capacity Substrates for Calorimetry Measurements

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Abstract

Our research focuses on developing calorimetry techniques and instruments for thin film material, nanometer scale particles of metals, polymers and nanoliter volumes of proteins [1]. Development of these devices for such characterization techniques via the nanocalorimetry technique (NanoDSC) makes use of CNF micromachining [2] techniques. Many different sample configurations can be used including vacuum-deposited, spin-cast, or liquid solutions. A variety of materials phenomenon which occurs at the nanometer length scale have been investigated, such as the size-dependence melting point depression or sizes in metal nanoparticles [3], order-disorder transitions of self-assembled monolayers of alkanethiols (SAMS) [4], and thickness dependence of glass transition in polymers [5]. In this report we discuss work on two topics: (1) heat capacity measurements self-assembled monolayers (SAMs) on Ag substrate [6], and (2) preliminary results of a new liquid calorimeter using a model system.

Summary of Research

SAMs have tremendous potential in all types of technologies ranging from new type photoresists for microelectronics to surface conditioning for biomaterial applications. One monolayer of a molecule on a surface is a fascinating system to study, especially for long chain alkanes. How do they order themselves? How do they melt? Do they melt? Should they be different than bulk alkanes (alkanethiols)? Should the thermodynamic properties of a 3D SAM differ from a 2D SAM?

One of the most puzzling aspects of SAMs is the contrast of the heat of melting Hm of SAMS. The value of 3-D SAM/ Au is only 25% of bulk thiol, where as it is 100% when grown on 3-D SAM/Ag. Confinement of alkanethiols on a Au or Ag surface in the form of a self-assembled monolayer (SAM) generates a material apparently more stable than the bulk alkanethiols having a “melting” temperature 100K greater than the bulk.

The order-disorder transition in this system is unique in that it is done under the ultimate confinement of the length-scale of the molecule (nm). We report here about our recent work on 3-D SAM/Ag. Hexadecanethiol self-assembled monolayers (SAMs) grown on polycrystalline Ag planar surface (2D) and nanoparticles (3D) are studied by measuring heat capacity with NanoDSC. Sharp melting transitions at high temperatures were observed in this system, which indicate the presence of well ordered phases. SAMs on 3D Ag show melting characteristics that are comparable to that of Ag-alkanethiolate layered materials precipitated from solution. The different states of the system can be manipulated by changing the specific heating and cooling schedule.

We have begun developing a liquid-calorimeter which would be used for protein studies. Theoretical models have suggested that protein folding proceeds over an energy landscape described as a “folding funnel”, which has metastable intermediate states with entropic and/or enthalpic kinetic barriers. One limitation to the study of protein folding using conventional calorimetry has been the time scale for measurement. The high rates of the proposed liquid NanoDSC will allow us to observe these fast intermediate steps of protein folding. The design of the NanoDSC will also enable us to develop systems allowing...
combinatorial studies. Furthermore, the small sample sizes is ideal for characterization of new proteins which are usually made in limited quantities. Our preliminary design uses docosane as the test-sample.

This prototype biocalorimeter has a scan rate (10 K/min) which is much smaller than the scan rate (~2,000,000 K/min) of the nanocalorimeter used in the (previously mentioned) SAM work. However, it is much faster than conventional biocalorimeter (1 K/min). It also has a much smaller sample-volume than conventional biocalorimeters (> 100 uL). The plot in Figure 2 shows the Cp(T) data of n-docosane C_{22}H_{46} using of 22 uL sample vessel. The n-docosane was introduced to the sample holder as a fluid (melt) and then allowed to solidify before the calorimetry measurement. As expected melting occurs near bulk values T_m ~ 43-45°C. The sample also exhibits multiple melting peaks with H_m ~ 6 (J) which is expected for this system.

**Figure 1:** Nanocalorimetry results self-assembled monolayers (SAMs) grown on 3-D Ag surface. Two different melting transitions with peak temperature T_{M1} ~ 115°C and T_{M2} ~ 127°C are observed, which indicates two ordered phases exist. SAMs with T_{M2} are thermodynamically preferred, and the melting characteristics are comparable to that of Ag-alkanethiolate layered materials precipitated from solution. Different states of the system can be obtained by using different heating/cooling schedules: (1) upon fully annealing, a single phase with T_{M2} is observed; (2) partially annealing results in the coexistence of both phases; (3) upon rapid quenching from high temperature, a single phase with T_{M1} can be obtained.

**Figure 2:** Preliminary results using a new liquid calorimeter designed for fast-scan and low-volume protein analysis. The plot shows the C_p(T) data of n-docosane C_{22}H_{46} using of 22 uL sample vessel. The n-docosane was introduced to the sample holder as a fluid (melt) and then allowed to solidify before the calorimetry measurement. As expected melting occurs near bulk values T_m ~ 43-45°C. The sample also exhibits multiple melting peaks with H_m ~ 6 (J) which is expected for this system.

**References**


Novel Sensors, for Chemical and Biological Agents, Based on Electrochemical Detection Using Conducting Polymers

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Abstract

We report on a novel design of an organic thin-film transistor (OTFT) that is used as a platform for sensor applications. This transistor is based on conducting electroactive polymers and operates at low voltages. The sensor response is measured as the drain-source current of the channel while a potential is applied on the gate. The behavior of the transistor can be understood in terms of an electrochemical mechanism which is proven to depend on the ionic concentration of the electrolyte.

The possible applications of these devices in sensing, e.g. sensing of harmful chemical and biological agents, are explored as the advantages are many. Organic materials offer facile processing, e.g. roll-to-roll printing, which can result into low cost fabrication onto flexible substrates and therefore disposable sensors. Chemical synthesis can be used to tune their electronic properties and provide selectivity and specificity through their integration with biological systems. Low end performance which generally limits the range of applications of organic transistors is not an issue as they provide fast enough responses for sensors. Furthermore, by fabricating disposable sensors, the lifetime and fouling issues are avoided. Examples of biological sensing applications include the utilization of enzymes for the detection of several target analytes, and utilization of epithelial cells for the detection of harmful enteric pathogens, e.g. bacteria, viruses, toxins, etc. Use of different recognition elements in these devices can result in sensors for different applications. Current work includes the transition from photolithography to roll-to-roll inkjet printing, as well as the development of arrays of sensors where many different agents can be detected at the same time.

Summary of Research

The application of transistors based on poly(3,4-ethylenedioxythiophene) doped with poly(styrene sulfonic acid) (PEDOT:PSS) in chemical and biological sensing is realized. These devices offer enormous potential for facile processing of small, portable, and inexpensive sensors. These capabilities are ideally suited for point-of-care analysis. Organic thin film transistors can be used to detect a wide range of analytes for a variety of possible applications in many fields, e.g. environmental monitoring (air-borne chemicals, water contamination, etc.), health care (medical diagnostics), and food industry (smart packaging). These devices are excellent candidates for sensor applications because they have the ability to efficiently translate chemical and biological signals into electronic signals with high sensitivity. Furthermore, functionalization of PEDOT:PSS films with a chemical or biological receptor can provide high specificity in regards to the target analyte. The advantages of using PEDOT:PSS electrochemical transistors are many, as well as the possible applications for sensing analytes in both gaseous and aqueous environments.

Organic thin-film transistors (OTFTs) have been long used for sensor applications. They offer several advantages for use as transducers due to their inherent signal amplification that allows for use of small sample volumes, and ease of incorporation into circuits. Among these transistors, the organic electrochemical ones offer additional advantages, such as low operating voltages, the ability to operate in aqueous environments that are essential for biological applications, and simplified device structures. These key properties enable organic electrochemical transistors to be integrated with microfluidic devices, and ultimately move the technology closer to the “lab-on-a-chip” concept.
Typically, an electrochemical transistor consists of the channel, the source (S), drain (D) and the gate (G) electrodes, and an electrolyte medium in contact with the channel and the gate, as shown in Figure 1. The channel is a thin film of semiconducting polymer (in its doped state) which has been deposited onto a supporting substrate. In case the channel is composed of a highly conducting polymer, such as PEDOT:PSS, the same material can be used for the source and drain contacts as well. Electrochemical transistors operate in an electrochemical mode where the conductivity of the polymer film is modulated by doping, i.e. oxidation or reduction. This doping (de-doping) process is reversible and involves the diffusion of mobile ions from the electrolyte solution out of (into) the polymer film. This results in the switching of the polymer between its redox states.

In recent years, the demand for low cost fabrication techniques of organic semiconducting devices has grown tremendously. This demand is driven by the need for small, portable, and inexpensive sensors which will realize the point-of-care analysis. PEDOT:PSS-based electrochemical transistors have been so far patterned by standard photolithography and dry etching. Then, thin layers of PEDOT:PSS can be deposited onto a variety of solid substrates using the spin-coating technique. If a metal (Pt, Au, or Ag) is used for the gate, source, and drain contacts, then additional deposition and patterning steps are required. However, although the use of photolithography provides high quality devices, it’s not suitable for mass fabrication of disposable devices. This technique can be expensive, and time consuming. Hence, simpler and more versatile patterning techniques are considered necessary.

Soluble organic conducting materials such as PEDOT:PSS offer options for simpler patterning techniques due to their ease of processing. We are currently working on optimizing the Dimatix inkjet printer parameters in an effort to move from the traditional multi-step photolithography patterning of the devices described above to one step printing process. The Dimatix inkjet printer, which was recently added to the CNF labs, is a promising tool for the fabrication of disposable semiconducting devices. Figure 2 shows a picture of an old array of devices on glass that were fabricated using photolithography, and Figure 3 shows a recent array of devices on paper that were fabricated by the Dimatix inkjet printer.
Nanoporous Gold Thin Films

CNF Project # 1163-03
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Abstract

Nanoporous gold thin films (NPG) have potential for diagnostic applications [1] or in MEMS devices [2] that require inertness, conductivity, or increased surface area. We have deposited silver-gold (Ag-Au) alloy films on silicon substrates and measured the stress induced in the films during dealloying. Changes in stress as a function of temperature were also measured. Scanning electron microscopy (SEM) imaging was used to determine porosity changes due to exposure to various concentrations of nitric acid and as a function of annealing. Surface-enhanced Raman spectra of alkanethiol self-assembled monolayers (SAMs) on the NPG were acquired.

Experimental Details

Nanoporous gold thin films (Figure 1) were prepared by selective leaching of silver from a Ag₇₀Au₃₀ composition ratio [3] (by atomic mass), resulting in a mesoporous metal having pore dimensions of 10-30 nm. Three-inch silicon wafers were cleaned in a standard metal-oxide semiconductor (MOS) cleaning process for 30 minutes and rinsed with ultrapure (18 MΩ-cm) water. After undergoing an additional rinse/dry cycle, the wafers were placed in a 1000°C thermal oxidation furnace for an hour to facilitate growing of 300 nm of oxide on substrate. Upon cooling, an adhesion layer consisting of 30 nm Cr / 50 nm Au was evaporated and approximately 250-300 nm of a Ag₇₀Au₃₀ alloy was deposited using an argon-ion sputter system (Baltec). Wafers were then immersed in various concentrations of nitric acid (ranging from 15% to 40% v/v), rinsed with DI water, blown dry with nitrogen gas, and all owed to dry overnight. Substrates prepared in this manner were a dark brown in color, with increasing darkness upon immersion in higher concentrations of acid.

Summary of Research

Stress Analysis: Stress measurements were acquired by a common laser scanning technique [4], which measures substrate curvature changes associated with thin film stresses. It was found that the tensile stress decreased after etching. Average stress values measured in the sputtered film were around 340 MPA, which dropped to 60 MPA in the porous film after the etching process. Relaxation of stress also occurred as a result of annealing (Figure 2). Thermally activated motion of gold atoms allows atoms to relieve local stresses through rebonding.

Morphology: Pore and ligament evolution was monitored as a function of annealing at 450°C for 15 hours (Figure 3). Statistical analysis (computed via image processing...
Materials software) showed that the first 10 hours of annealing resulted in a near-exponential growth in the average pore width (in nanometers), and from 10-15 hours, the average pore width appears to be closing. The complete duration of annealing resulted in a linear increase in the ligament width. This evolution is also accompanied with ligament coarsening, which affects the material’s ability to be used as a surface-enhanced substrate (see Raman discussion).

**Raman Spectroscopy:** Surface-enhanced Raman spectroscopy (SERS) is a technique that uses inelastic (Raman) scattering of monochromatic light for the detection of adsorbed molecules on roughened metal surfaces (typically gold or silver). Increased intensity of the Raman signal is dependent on the electromagnetic enhancement (associated with the roughened metal surface produced by wet-chemical etching) and also on the electronic coupling of the molecules to the surface via a surface plasmon in the metal. In this experiment, the vibrational modes of 1-dodecanethiol (98%) and 2-mercaptopyridine (99%) self-assembled monolayers (SAMs) chemisorbed on NPG substrates produced in various acid concentrations were analyzed using Raman. These molecules were chosen because of their large inherent polarizability that results in strong Raman scattering.

Enhancement for both molecules is visible on nPG substrates formed by etching in 15% and 20% v/v HNO₃ concentrations and not visible in increased acid concentrations (Figure 4). This characteristic may be due to residual silver in the alloy etched at lower acid concentrations, which could enhance the electronic coupling of the molecule to the surface through surface plasmons. Such enhancement is not visible on substrates that were annealed before thiol deposition. SEM images (Figure 1c) show that annealing results in an increase in coarsening due to surface smoothing, which may reduce the electromagnetic enhancement associated with a rough surface.

**References**


Shape-Selectivity in the Aggregation of Lithographically-Designed Colloidal Particles

CNF Project # 1177-03
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Abstract

We have developed a method to produce well-defined, lithographically-designed, non-spherical colloidal particles. We demonstrate here that the formation of a highly anisotropic structure in a model system of Brownian cylindrical particles can be achieved by precisely controlling electrostatic, van der Waals and depletion interactions, as well as the surface properties of the particles. We show that this method can also be used to produce a diversity of particles of more complex shape, and thus opens the road to the formation of a broad diversity of structures at the colloidal scale.

Report

The development of several promising fields, such as micro-mechanics or photonics, requires precise control of structure from the nano-scale up to the micro-scale (100 nm - 100 µm). An appealing approach consists in using colloidal particles as building blocks to form a diversity of structures by self assembly; this strategy exploits concepts of chemical kinetics and thermodynamics to direct spontaneous formation of structure. To date, this approach has been limited by the paucity of available particles and the lack of a robust method to control structure formation.

To generate a diversity of particle building blocks, we have developed a photolithographic procedure (Figure 1) to obtain particles of well-defined shape and size, directly from a negative photoresist, SU-8 2002 (Microchem Corp.). We have focused our study on a model system of cylindrical particles to have a better understanding of the mechanisms at play in the process of self-assembly; we have also successfully used this method to produce particles of more complex shape (Figure 2). The key aspects of the process are: 1) Spin coating of a sacrificial layer (Omnicoat, Microchem corp.) on a 100 mm silicon wafer—this layer is later dissolved in order to release the particles; 2) Spin coating of a layer of SU-8—this step defines the height, \( h \), of the particles that will be formed; 3) Exposure of the SU-8 layer to ultraviolet light (GCA Autostep 200 DSW i-line Wafer Stepper) through a photomask—this step defines the diameter, \( d \), of the particles; 4) Development of the resist, release of the particles via dissolution.

Figure 1: (A) Schematic diagram of formation of cylindrical particles in an epoxy-based negative photoresist (SU-8) via projection photolithography. (B and C) SEMs of SU-8 particles produced by this process and redeposited on a silicon wafer from water.
of the sacrificial layer, washing via repeated centrifugation, and resuspension in a buffered aqueous solution of Tergitol NP40, a non-ionic surfactant. This route to the formation of particles generates $\approx 10^9$ colloidal particles on a 100 mm wafer. We have formed particles with thicknesses in the range $0.3 < h < 3 \ \mu m$, and diameters in the range $0.8 < d < 8 \ \mu m$.

Analysis of the lithographically-designed particles by scanning electron microscopy (SEM) indicates that the size distribution is narrow within a single wafer ($\Delta d$ and $\Delta h \approx \pm 5\%$). Atomic force microscopy (AFM) performed on aspect ratio 1:1 particles ($h = 1.2 \ \mu m, d = 1.2 \ \mu m$) reveals that there is a contrast in the roughness of the particles: the flat top and bottom are much smoother (4 nm RMS) than the curved side (12 nm RMS). This contrast in the roughness likely plays a role in defining the interactions between the particles; we are currently investigating this effect both theoretically and experimentally.

We employed these particles to explore the use of depletion and DLVO (van der Waals + electrostatics) forces to generate shape selectivity. Depletion interactions are attractive interactions that are due to excluded volume effects when a non-adsorbing polymer, for example, is added to the solution. The distinction in shape, both global curvature and roughness of the sidewalls relative to the ends of the particles implies distinct steric constraints on their interactions when oriented end-to-end, end-to-side, and side-to-side: the smooth, flat ends will be able to form closer, more extended contact than the rough, rounded sides. An evaluation of the electrostatic, van der Waals, and depletion interactions for smooth cylindrical particles predicts orientationally selective interactions: end-to-end interactions will be favored over end-to-side and side-to-side.

To explore this prediction experimentally, we observed the evolution of the behavior of particles of aspect ratio 1:1 ($h = 1.2 \ \mu m, d = 1.2 \ \mu m$, Figure 1B) and 1:3 ($h = 0.4 \ \mu m, d = 1.2 \ \mu m$, Figure 1C) when both the ionic strength and the depletant volume fraction in the suspension are raised. We find that as the electrostatic repulsion between the particles is diminished with rising ionic strength and the depletion interaction is increased with rising concentration of the non-adsorbing polymer, the particles pass through a columnar state in which aggregation occurs with strong selectivity for end-to-end assembly (Figure 3). This result demonstrates that our strategy, based on shape considerations and the use of selective interactions, is a powerful one to induce and control structure in a system of well-defined cylindrical particles. More generally, we think that this strategy could be used with a diversity of lithographically-designed particles of more complex shape to obtain a diversity of promising structures at the colloidal scale.

This work has been published in the Journal of the American Chemical Society (Badaire et al., J. Am. Chem. Soc., 129, 40-41, 2007).

Figure 2: SEM picture of different shapes that can be obtained by photolithography in 1 \ \mu m thick SU8-2002 photoresist. Scale bar = 2 \ \mu m.

Figure 3: State diagram of the different states observed for particles of aspect ratio 1:1 (Figure 1B) and optical picture representative of the columnar regime. Scale bar = 10 \ \mu m.
Compact Hydrogen Generation through JP-8 Fuel Processing Using Microplasmas

CNF Project # 1184-04
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Abstract

Fuel cells have been researched extensively as means of converting high energy density fuels to provide long-lasting, compact power supplies for military and domestic applications. Reforming of hydrocarbon fuels leads to the highest system energy densities since these liquids are excellent sources of hydrogen at high density. While typical reforming operations rely on catalyst-driven processes, unfortunately fouling and lifetime issues have limited the effectiveness of most of these processes to date, especially for compact systems with greater restrictions on size, weight, operating temperature and materials of construction.

This project aims to study reforming of a heavy hydrocarbon military fuel (diesel-like JP-8 fuel) using a microplasma. Plasmas confined to microscale geometry exhibit extremely high ion, electron, and reactive radical densities and hence can activate chemical processes in place of catalysts and at lower reactor temperatures. We intend to implement microfabricated plasma reactors in order to benefit from scaling laws permitting processing at pressures greater than atmosphere.

Fabrication

We have fabricated the first set of microplasma reactors at CNF and will characterize them for their plasma operation with inert and well-characterized plasmas like xenon (Xe). We will ultimately introduce characteristic hydrocarbon species representative of those found in JP-8, and analyze the effects of the plasma. Optical emission spectroscopy will be used to analyze intermediates. Gas chromatography will be used to analyze the product distribution and to assess conversion. FTIR will be used for in situ chemical analysis within the reactor.

Plasma reactors were designed in AutoCAD (Figure 1) and microfabricated using various silicon processing techniques. Silicon wafers, 500 µm thick, served as the starting substrate. Silicon dioxide of substantial thickness (2 µm and 4 µm, on different wafers) was thermally grown on the silicon substrate using an MOS oxide furnace operating around 1200°C. Nickel anodes were created using lift-off photolithography and metal evaporation. Next, microchannels between the anode contacts were dry-etched into the dielectric layers using fluorine chemistry, and the etching continued in the silicon substrate by switching over to the Bosch process for deep reactive ion etching (DRIE). Finally, gold was evaporated onto the backside of the silicon substrate, and the silicon wafers were sawed into the final device size (1 cm by 0.8 cm).
Summary

The devices recently microfabricated at CNF are currently in the process of being evaluated for plasma performance by attaching them with their backside cathode against a platform onto which a negative voltage is imposed while grounding the nickel topside anode. The entire platform sits inside a small pressure-controlled chamber (see Figure 2) which can be filled with various ambient gases. Initial testing has begun using Xe gas. A microplasma was generated at atmospheric pressure and contained within the microtrench of the device (see Figure 3). Initial test results are very encouraging.

References


Carbon Fiber Integrated MEMS for Optical Scanning

CNF Project # 1241-04
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Abstract

Optical scanners are used in a variety of systems to transmit or convert information encoded in light. Employed in telecommunications networks, optical storage systems, projection displays and image recording systems remain critical components that have not seen the miniaturization and cost and performance improvements as other optoelectronic devices. The development of microelectromechanical systems (MEMS) technology, has promised inexpensive, very low power requirement, miniature scanning mirrors capable of very high speed scans and large deflection angles. However, the mechanical property limitations of traditional materials used in MEMS technology has failed to produce scanners that can simultaneously scan large angles (mechanical angles > ± 45°) and high speeds (5-100 KHz).

We have developed a novel category of MEMS optical scanners that make use of fibrous materials—in particular high performance carbon—as mechanical elements, cultivating the development of a novel, more robust MEMS material category capable of fatigue free behavior and vastly superior elastic deformations. Carbon fiber integrated MEMS optical scanner devices have been successfully fabricated leveraging existing semiconductor and MEMS processing, and exhibit fatigue free mechanical scans of > ± 45° at speeds exceeding 5 kilohertz (KHz).

Summary of Research

The goal of this project is to develop a novel materials technology based on fibers to address the mechanical limitations of the current silicon-based MEMS technology with a particular emphasis on high performance applications that place severe demands on materials performance, such as optical scanners. The use of fibers in MEMS scanner devices permits the use of very high performance materials that can be tailored towards the performance needs of the device. By using fibers as the flexure elements within a MEMS micro-mirror, specific property enhancements can be obtained with virtually limitless control of the strength and stiffness of the flexures resulting in enhanced speed and scan angle performance of the MEMS scanner device. The large bending capabilities of fibers even at micro-scales permits the creation of micro-mirror elements with very large deflection capabilities, resulting in scan angles far surpassing existing silicon based devices. Additionally, by simply varying the number of fibers within a flexure element the speed response of the micro-mirror can be modified, without any change in the large scan angle capability. The carbon fiber integrated MEMS micro-mirror technology developed through the present research also shows greater resistance to fatigue due to cyclic stresses [1].

We have developed reliable methods to deposit precise numbers of oriented layers of fibers onto silicon substrates, and have developed methods to lithographically pattern and etch the fibers into desired MEMS structures. Etch chemistries optimizing fiber etch rates (> 0.7 µm/min) have been developed [2]. Epoxy and metal based coupling agents have also been explored to form composite structures coupling the carbon fiber flexure elements to silicon structures on the substrate. Surface treatments to enhance the coupling of fibers to both thin film metallic matrices and photo-curable epoxy based materials have also been developed.

Using carbon fibers integrated with silicon structures, we have successfully developed a novel MEMS micro-mirror technology capable of scanning laser beams at very fast speeds and large scan angles. This performance behavior has been extended towards a prototype miniature projection system capable of scanning an 8” x 11” monochrome video image from a 1.5” projection distance.
Materials

References


Freestanding Alumina Membrane by Double-Layer Anodization

CNF Project # 1310-05
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Abstract
Anodic alumina membrane has been of great interest because of its unique nano-porous structure. Here a double-layer anodization (DLA) [1] has been developed for fabricating full or partial freestanding anodic alumina based on a silicon substrate. A microfluidic channel formed by alumina membrane is also demonstrated. For its simplicity and flexibility, DLA is expected to be a useful approach in anodic aluminum oxide (AAO)-related applications.

![Figure 1: Illustration of a freestanding anodic alumina membrane on Si chip.](image)

![Figure 2: Porous structure of the freestanding alumina membrane.](image)

Experimental Research
In our DLA method, a sacrificial metal layer (Cu or Ag, 50 nm thick) is introduced between an Al film (200 nm thick) and an Si₃N₄ substrate by evaporation. A freestanding alumina film at wafer scale is successfully achieved by anodizing the double metal layer at 20 V dc in 0.3M H₂SO₄ at room temperature, during which the alumina is spontaneously stripped off the Si₃N₄ substrate due to the anodic oxidation of the sacrificial layer (Figure 1). The barrier oxide at the bottom of the alumina film is effectively removed either by H₃PO₄ dissolution or by CF₄ reactive ion etching, making the alumina film a through-hole membrane (Figure 2). The freestanding alumina film is utilized as a contact mask to transfer its nanoporous pattern to a Si substrate. By patterning the sacrificial metal layer with contact lithography, a partial freestanding alumina film is successfully achieved on the silicon chip, producing a
unique micro/nanofluidic channel (Figures 3-4). Compared with previous techniques, the method reported here is advantageous for its simplicity and flexibility.

**Summary of Research**

The difficulty of fabricating freestanding AAO films is essentially the stripping of the AAO from the substrate. DLA makes this stripping process simple by adding an extra metal layer for lift-off purpose. The Al anodization and the AAO stripping are simply merged as a one-step fabrication, requiring no additional process. Another unique feature of DLA is its capability to produce partial freestanding AAOs, which would be interesting to the fields such as nanofluidics.

**References**

Silicon Nanostructures from Block Copolymer Derived Thin Films

CNF Project # 1356-05
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Abstract
We are currently developing a novel method to structure silicon (Si) down to 10’s of nm using organic-inorganic hybrid thin films. Using a block copolymer as a structure-directing agent for aluminosilicate sol, monolayer films are prepared on a Si substrate. Removal of the block copolymer leaves a matrix of aluminosilicate with cylindrical pores standing up on the Si substrate. These inorganic porous films are then used as a template to make epitaxially grown Si nanostructures. This method may allow the cost-effective and rapid formation of Si nanostructures down to the tens of nanometer regime.

Summary of Research
Thin films of block copolymer are well studied and have shown promise to be used in several applications [1]. In the Wiesner group, we use block copolymer as a structure-directing agent for inorganic precursors [2]. Monolayer films are prepared using self-assembly of a mixture of a PI-b-PEO block copolymer and an aluminosilicate sol, spin coated on a Si substrate. The hydrophobic phase (PI) forms hexagonally packed spheres on the substrate and surrounded by the hydrophilic phase (PEO and aluminosilicate). The calcined (organic free) aluminosilicate films are typically 15-20 nm thick, over a 4” (~100 mm) Si substrate, as indicated in the cross-section STEM (Figure 1).

These inorganic films are then used as a template to make Si nanostructures, see schematic (Figure 2). The pores of the film are first filled with amorphous silicon using the CVC sputter deposition system in CNF. This is followed by pulsed laser annealing which selectively melts silicon. On cooling, silicon crystallizes inside the pores, epitaxially from the substrate, confirmed by cross-section high resolution TEM. Subsequent removal of the...
inorganic film by HF treatment leaves the crystalline Si nanostructure on Si substrate. Further radial distribution analysis confirmed a perfect congruence between the pore-pore spacing on the template and center-center spacing on the final nanostructures. See atomic force microscopy (AFM) images (Figure 3) and radial distribution function (Figure 4).

We are currently working to control the aspect ratio (height/diameter) and center-center spacing of the nanostructure by changing the block copolymer molecular weight and inorganic-organic ratio in the spin coating solution.

References

Figure 3, top: AFM images of film and nanostructures.

Figure 4, bottom: Radial distribution of pore-pore spacing of the template and center-center spacing of the nanostructure.
Examining and Influencing Order in the Flow of Worm-Like Micelles Through Porous Media

CNF Project # 1405-05
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Abstract

Surfactants are widely used 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. An additional design has been created to allow the study of these novel fluids in expansion / contraction chambers [3].

References

**Crystalline Monolayers from Convectively Self-Assembled Non-Spherical Colloids**

CNF Project # 1417-06  
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**Abstract**

Colloids with different morphologies were self-assembled into monolayers on silicon and glass substrates by a heat-assisted convective assembly technique. The monolayers show a high degree of positional and orientational order.

**Summary**

Assembling non-spherical two dimensional (2D) particles is motivated by the potential of achieving colloid-based materials that have a higher degree of structural complexity and produce stronger light-matter interactions due to the complex particle shape. We have recently demonstrated that several different non-spherical colloid shapes can assemble within a thin wetting layer formed by aqueous particle suspensions on a hydrophilic surface.

The figure shows scanning electron microscopy (SEM) micrographs of the crystals produced from the non-spherical particles used in this project. The shapes are classified as disc, mushroom-cap shape, sphero-cylinder and asymmetric dimer. The particles were positioned on either a hexagonal or oblique lattices, depending on the particle shape. The lattice became more oblique as the aspect ratio of the particle increased. The monolayers have positional order and varying degrees of orientational order. Single crystalline regions were as wide as 100 μm. The crystal structure was also changed by varying the suspension concentration from which the assemblies were made. This commonly produced hexagonal lattices from the sphero-cylinders and asymmetric dimers, with the particle long axis aligned perpendicular to the substrate.

Future work will focus on producing larger scale crystalline films. These films will then be infiltrated with a higher refractive index medium such as silicon or gallium arsenide, in order to enhance the refractive index contrast necessary to yield strong light-matter interactions.
Figure 1: SEM images of crystalline monolayers from 1-3 micron size non-spherical colloids.
Directed Linking of Carbon Nanotubes with Single CdSe Quantum Dots

CNF Project # 1455-06
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Abstract

Metallic single-walled carbon nanotubes (SWNTs or NTs) are the ideal nanometer-scale wire, as they can withstand current densities up to 2 to 3 orders of magnitude higher than copper currently used in electronic chips [1]. These conductive NTs can be utilized as nano-electrodes to electrically contact another nanoscale object, such as a single semiconductor quantum dot (QD) or metallic nanoparticle (NP). We have designed a strategy for directed assembly of fabricated QD–SWNT devices. NTs were grown across patterned catalyst islands on a silicon wafer followed by electrode placement. After cutting the NTs, the resulting carboxyl group moieties found at the cut NT edges were used to covalently attach amine-functionalized cadmium selenium (CdSe) QDs or gold (Au) NPs. Electrostatic force microscopy (EFM) was used to monitor NT conductivity before and after cutting, as well as after NP attachment.

Summary of Research

Combining the technique of NT chemical vapor deposition (CVD) synthesis with photolithography and metal deposition has resulted in the successful fabrication of a device with an active NT component, as shown in Figure 1a. Device validity is monitored by applying a voltage to the NT, followed by capturing an EFM charge image, as shown in Figure 1b. After NTs are grown on a substrate and device validity is assessed, the difficult process of NT cutting occurs. NTs have been cut successfully using two different methods: by applying a sequence of voltage pulses across a metal-coated atomic force microscopy (AFM) tip in close proximity to a grounded NT [2] and by using electron beam (e-beam) lithography in conjunction with an oxygen plasma to remove small sections of a NT (Figure 2) [3]. Whereas using an AFM tip yields gaps as small as 80 nm, the smaller (< 20 nm) gaps produced using e-beam lithography are necessary in order to successfully attach a single NP of less than 20 nm in diameter. EFM images captured before and after the cutting events clearly indicate the location of the cut.

When either cutting method is utilized, the exposed NT edges oxidize to form carboxyl groups that will be used to anchor a single NP. Previously, our group functionalized the ends of commercial NTs in solution with thionyl chloride and then covalently attached CdSe QDs coated with amine-terminated surface ligands [4]. In this project, similar chemistry has been used to attach single CdSe QDs to severed NTs on a silicon substrate (Figure 3). The hydrophobic surface ligands on CdSe QDs are exchanged with amine-terminated ligands, which can form amide bonds with carboxyl groups found on the cut SWNT edges to yield the consummate NP-NT circuit [5]. CdSe QDs have been attached along the NT sidewalls.
successfully; however, due to the small size (~ 4 nm) of a single CdSe QD, conjugation across the larger NT is impossible.

Two possible solutions to the size mismatch problem are to create a smaller cut in the NT or to use larger NPs. Since e-beam lithography is currently the best method for patterning the smallest features and its resolution limit is < 20 nm, choosing larger particles to fit into the NT gap is the most plausible solution. CdSe nanorods (NRs) are a better candidate for filling the NT gap due to their elongation along the c-axis. These NRs can be coupled to NTs using the same procedure as for CdSe QDs (Figure 3).

The difficult task of exchanging the hydrophobic surface ligands on NRs for hydrophilic ones is currently being investigated in our group. Another candidate for placement into the gap is an Au NP, which can be synthesized in sizes ranging from a few to hundreds of nanometers (Figure 4) [6, 7]. While it has been reported that dielectrophoresis can be used to move Au NPs between gold electrodes [8], placing an individual Au NP between NT nano-electrodes would be a major breakthrough.

After device optimization on silicon substrates is completed, fabrication of NP-SWNT structures on quartz substrates will allow for laser excitation and fluorescence measurements studies. The finished device will be used to study charge-transfer effects between the NPs and the NT, with possible applications in solar cell technology.

References

Graphene as an Atomic Membrane Interface

CNF Project # 1486-06
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Abstract
We fabricated silicon devices for the study of graphene as an atomic membrane interface, with potential applications in nanoscale sensing and imaging. The devices consist of holes on silicon nitride on which graphene can be suspended and accessed from both sides of the membrane, to create two isolated environments separated by less than a nanometer.

Summary
Graphene, a flat material consisting of carbon atoms packed in a hexagonal honeycomb lattice, is part of the family of fullerene molecules such as buckyballs and carbon nanotubes which have been discovered in the last 20 years. The goal of this project is to develop a method to use graphene as an atomic membrane interface between two different mediums such as a liquid and gas or vacuum. Since a single layer graphene membrane is only one atom thick, two isolated mediums can be brought very close together and yet remain physically separated from each other by less than a nanometer. We are attempting to achieve this by mechanical exfoliation of bulk graphite directly onto previously fabricated devices. We are using two different approaches to fabricate an atomic membrane interface. The fabrication steps for these two approaches can be performed on a single wafer, since all of the steps involved are the same.

The first approach is to open backside windows on silicon wafers using the benchmark potassium hydroxide (KOH) wet etch with a silicon nitride mask. A graphene membrane, suspended on top of a hole in the silicon nitride, can be accessed from the back of the device. This design provides the opportunity to separately control the two isolated environments, above and below the graphene membrane.

The second approach consists of fabricating columns of holes etched on a low stress silicon nitride film. By taking advantage of undercut effects, different holes can become connected from underneath; KOH will etch silicon to form a channel shaped structure below the patterned silicon nitride (Figure 1). After selecting a hole that has been successfully sealed by a graphene membrane, a polydimethylsiloxane (PDMS) layer with a slightly larger hole can be added on top, allowing access to the graphene membrane but effectively sealing neighboring holes. A liquid containing biomolecules can then be injected into the channel through the holes which are farther away from

Figure 1: Optical microscope image of 3 columns of trenches and holes on silicon nitride, with sides of 2, 3.5 and 5.5 µm respectively (left to right).
the graphene membrane. In this way, graphene becomes an atomic interface between the injected liquid and the gas or vacuum outside of the device.

Low-stress silicon nitride is grown on silicon wafers in a low pressure chemical vapor deposition (LPCVD) silicon nitride furnace. It is of great importance to be able to discern a graphene membrane optically, in order to swiftly identify useful devices. The contrast created by the presence of graphene will depend on the thickness of the silicon nitride film; we are in the process of determining the optimal thickness of this film. Arrays of trenches facilitate the localized exfoliation of bulk graphite. The depth of these trenches, etched using reactive ion etch (RIE), is roughly half of the total thickness of the silicon nitride. Holes of sizes ranging from 2 to 5.5 µm per side are etched on silicon nitride, also using RIE. The wet etch using KOH is then performed. During the time it takes for the backside windows, described for the first type of devices, to etch all the way through the wafer (more than 3 hours at 90°C using 25% KOH), the column of holes become connected because KOH undercut etching forms a channel-like structure in the silicon underneath the silicon nitride holes. If the separation of individual holes in a column increases, the etch time for the holes to become connected from underneath will evidently increase. The width of the resulting silicon nitride membrane column will also increase with time, resulting on a more fragile device. This is problematic since the silicon membrane must not break during the graphite mechanical exfoliation process, which is equivalent to writing with a pencil on a delicate membrane. However, if the separation between holes is too small, not only will the silicon nitride ‘bridge’ between the two sides of the column become thin and fragile, but isolating a specific graphene-sealed hole with PDMS would become exceedingly difficult; we will work on optimizing this parameter.

We believe there is potential for the fabrication of nanoscale sensing devices using this geometry, as well as the development of a novel imaging platform in which a scanning tunneling microscope/atomic force microscope (STM/AFM) tip can be driven in vacuum to enhance image resolution, while allowing biomolecules such as deoxyribonucleic acid (DNA) to remain in their native liquid environment. Interactions between biomolecules and graphene would induce a local deformation of its electronic structure. Thus, the atomically thin interface created by graphene would act as a window through which an STM/AFM tip could gather information that can be used to construct an image of a molecule on the other side of the membrane. Using this technique, images of individual molecules in solution could be obtained while taking advantage of the optimum performance of STM/AFM imaging techniques when these tools are operated in vacuum.

**References**


**Design of a Selective Gas Sensing Device Using Platinum Electrode Interdigitated Substrates**

**CNF Project # 1490-06**

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**Abstract**

Alumina substrates with interdigitated platinum (Pt) electrodes for resistive sensing applications and heating elements on its back have been successfully fabricated using photolithography method. Molybdenum oxygen (MoO₃) nanoparticles synthesized by alcoholic hydrolysis method have been deposited onto the substrates for nitrogen hydrogen (NH₃) gas detection. The synthesized resistive NH₃ sensors are used to integrate into a hand held device applied for breath analysis and disease detection.

**Summary of Research**

Semiconducting metal oxides have been used for decades in gas sensing applications. The basic principle behind the gas sensing mechanism by metal oxides is the change in their electrical resistance on exposure to a reducing or oxidizing gas, due to electronic change. On the other hand, NH₃ sensors are being used in diverse applications such as food technology, chemical plants, and medical diagnosis and for environmental protection.

Among many kinds of semiconducting metal oxides, MoO₃ is a promising material for NH₃ detection. MoO₃ has a weakly-bonded layered structure. In addition, it has been demonstrated by our group [1] that NH₃ tends to remove MoO₃ layer when it is exposed to MoO₃, replacing O with N. This reaction causes an ultra high sensitivity.

In this project, we successfully synthesized MoO₃ nanoparticles using alcoholic hydrolysis method. In this method, molybdenum isopropoxide mixed with butanol are ultrasonicated at room temperature to form MoO₃. The materials are then deposited onto the sensing substrate. Figure 1 shows the sensing response of a MoO₃ sensor when it is exposed to NH₃. The result indicates that our sensor can detect NH₃ with the concentration down to 100 ppb. The detecting signal is stable and the response time for each gas exposure is less than 100 s.

In order to make a hand-held NH₃ detection device, the substrate with measuring electrodes on which the MoO₃ nanoparticle sensing film is deposited should be small enough. Here, we use alumina substrates with the size of 3 mm x 3 mm x 0.1 mm. By using a photolithography method, a layer of Pt electrode used for resistive sensing applications has been deposited on one side of the substrate, as well as another Pt electrode layer on the other side for heating the substrate. Figure 2 and Figure 3 show the design of the sensing electrode and heating electrode, respectively. The deposition thickness is about 100 nm.

Finally, the fabricated MoO₃ sensing element is integrated into a hand held device. This device collects and analyzes human breath and is only sensitive to NH₃.

**References**

Figure 1: Sensing response of MoO$_3$ nanoparticle sensor to NH$_3$ gas.

Figure 2: Design of sensing electrode on the alumina substrate.

Figure 3: Design of heating electrode on the alumina substrate.
Thin Film Bulge Test of Nanocrystalline Metals

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Abstract
Free-standing nanocrystalline copper (Cu) films with grain size around 39 nm are fabricated by thermal evaporation and characterized by the plane-strain bulge test. Young’s modulus and yield stress at a 0.2% offset are about 110 ~ 130 GPa and 400 MPa, respectively. Results show that no grain growth is observed and the predominant plastic deformation mechanism is grain boundary sliding accompanied by dislocation mechanisms within the grains to accommodate grain boundary sliding.

Summary of Research
In the present study, nanocrystalline copper (n-Cu) thin films are synthesized by thermal evaporation and the mechanical properties are explored using the “plane-strain bulge test” introduced by Vlassak and Nix [1]. The results are compared to previously published studies on the mechanical properties of similar free standing thin films with a micrometer grain size [2] and with nanometer grain size [3].

The n-Cu films are prepared by thermal evaporation (Edwards Auto 306 Vacuum Evaporator). The base pressure in the chamber is 1 x 10^{-6} Torr and high purity (99.995%) Cu is used. A current of 2.4 A is applied through the tungsten boat to melt the Cu source which yields a deposition rate of 0.1~0.2 nm/s. During the process, the substrate temperature is no higher than 70°C.

As shown in Figure 1, the fabrication procedure is modified from that of Xiang et al. [4]. A (100) Si wafer with a 500 nm thick Si₃N₄ layer deposited on both sides by low pressure chemical vapor deposition (LPCVD) is used. Positive photo-resist AZ5214 (CLARIANT), ultraviolet photolithography (wavelength of 365 nm) and reactive ion etching (RIE, 250 mTorr, 250 W in CF₄/O₂ atmosphere, TECHNICS Series 800) is employed to open a rectangular window through the Si₃N₄ on one side of the wafer.

Next, the Si wafer is anisotropically etched in 30 wt.% potassium hydroxide (KOH) solution at 80°C until a free standing rectangular Si₃N₄ membrane remains on the opposite side of the wafer. The n-Cu is then deposited on the Si₃N₄ membrane by thermal evaporation and another RIE etching with lower pressure and power (150 mTorr and 150 W) is used to remove the Si₃N₄ so that a freestanding n-Cu film is obtained. A schematic of the specimen is shown in Figure 2.

Figure 1: Sample preparation.

Figure 2: Schematic of the specimen.
Scanning electron microscope (SEM) micrograph and x-ray diffraction (XRD) studies show the average grain size of the specimens are in the range of 36 nm ~ 41 nm.

Figure 3 shows the plane-strain stress-strain curves for the six n-Cu films examined and three curves of the coarse-grained Cu films from [2] are also included for comparison. The Young’s moduli of the n-Cu films, compiled in Table 1, are in the range of 111 ~ 132 GPa, in good agreement with published values [5].

There does not seem to be a pronounced thickness dependence of the yield stress for the n-Cu films as there is for the coarse-grained films at the strain rate of 5 x 10^{-6} s^{-1}. Most of the specimens show a consistent range of 410 ± 10 MPa. As shown in Figure 3, the n-Cu films can achieve a ultimate stress of about 450 MPa, which is 50% higher than that of the coarse-grained Cu, 300 MPa, reported in [2]. Moreover, the results show that the n-Cu has less macroscopic ductility than the coarse-grained thin films and there is no obvious yield point in the data of Figure 3.

Figure 4 shows the progression of a crack-like feature near the edge of the free standing film. There is an uplift of material near the tip consistent with a Mode III ripping behavior. This is evidence of grain boundary sliding, however close inspection reveals a series of highly planar strain localizations which pass through several grains parallel to the Mode III crack tip, which suggests the possibility of dislocation activity. The deformation mechanisms associated with both types of failure are consistent with studies [6, 7] which state the deformation mechanism in n-Cu with grain sizes of those in the present experiments are thought to be grain boundary sliding accompanied by dislocation mediated plasticity within the nanoscale grains to accommodate grain boundary sliding.

Gianola et al. [3] reported two classes of stress-strain response for n-Al. The specimens in the present study are quite similar to one class which had been deposited using sputter deposition at a similar vacuum to present study (10^{-6} Torr). The specimens exhibited a high yield stress, and limited ductility due to shear localization which occurred at a very low level of plastic strain. The authors [3] show that the differences in response can be attributed to an effective absence of grain growth in specimens while stress-assisted grain growth occurred in another class of specimens which had been deposited at higher vacuum. That, combined with an absence of perceptible grain growth in our specimens, suggests that impurities in the grain boundaries tend to pin grain boundaries to limit grain growth just as in [3, 8].

References

Fabrication of Micropost Arrays for use as a Substrate for the Epitaxial Growth of Semiconductor Nanowires

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Abstract

Individual coupons of micropost arrays were fabricated using a deep reactive ion etch process. Each coupon consisted of a 10 x 10 square array of 5 µm diameter posts with a height of approximately 50 µm and a pitch of 400 µm. Each 5 µm diameter micropost top will serve as a substrate for the epitaxial growth and subsequent atom probe tomographic analysis of individual semiconductor nanowires.

Summary of Research

One major challenge to developing structure/property relationships in semiconductor nanostructures is the analysis of composition at the atomic scale. This challenge has recently been met by the use of atom probe tomography [1] to map the composition of single semiconductor nanowires with single-atom sensitivity and sub-nm resolution.

Pulsed-laser local electrode atom probe tomography (PL-LEAP) will be used to map the 3D composition of individual semiconductor nanowires [2] grown epitaxially on posts fabricated at the Cornell NanoScale Facility (CNF). In PL-LEAP analysis of nanowires, picosecond laser pulses are applied to the tip of a biased nanowire to generate thermally-assisted field evaporation of the surface atoms. The evaporated ions impinge on a position-sensitive detector from which the original position of each detected ion within the nanowire can be reconstructed in three dimensions. At the same time, the chemical identity of each detection event is accomplished through time-of-flight mass spectrometry. To minimize interactions between neighboring nanowires and the substrate during analysis, individual wires are to be grown epitaxially on the top surface of a micropost array.

Micropost arrays from Si(111) were fabricated using the tools available at the CNF. Individual coupons consisting of a 10 x 10 square array of 5 µm diameter posts with a height of approximately 50 µm and a pitch of 400 µm were made using a deep reactive ion etch process. An example portion of such an array is shown in Figure 1. The inset shows a detailed view of an individual micropost. The surface texture of micropost top is due to roughening of the oxide etch mask during the etch process. This roughening can be removed, revealing a pristine (111) surface for the epitaxial growth of an individual semiconductor nanowire.

References

Understanding Dissipation in Nanostring Resonant Sensors

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Abstract
We have used optical drive and detection to study dissipation in silicon nitride beam resonators. We are able to tune resonant frequency as well as quality factor in a given nanostring resonator by several hundred percent by tuning the stress in real time, providing a unique look into the sources of dissipation for this class of nanoresonator. A record room temperature quality factor of 390,000 at 3.7 MHz has been attained in vacuum with a silicon nitride nanostring under tension. Nanostrings have also been resonated in viscous liquids including water and biological buffer. This is the first demonstration of the operation of truly nanoscale flexural resonators in viscous liquids.

Summary
Optical drive and detection have been used to actuate and detect the resonance of doubly-clamped nanostring resonators. These resonators have been made from both high and low-stress silicon nitride. Devices in a high stress state have been operated with quality factors at radio-frequencies in excess of $10^5$ MHz. In Figure 1, we show the resonance of a 75 µm long, 500 nm wide, 105 nm thick string, with a quality factor of 390,000 at 3.7 MHz [1]. The stress state can then be tuned for a device of a given size by bending of the resonator chip, as shown in the schematic in Figure 2. Using this chip-bending technique, we have been able to drastically tune beam stress, resulting in tuning of frequency by several hundred percent. Over this wide range of frequency tuning, the quality factor is also tuned by several hundred percent, as summarized in the plot in Figure 2 in which frequency is increased while dissipation is decreased for several devices of a given size (colored symbols), and frequency and dissipation are pushed towards the values for the as-fabricated high stress devices (black squares).

Future work on the source of this drastic quality factor tuning will include investigations into the influence of the nanostring clamping, which may be influenced by the addition of stress. Nanostring resonators have also been operated in gas, of potential utility for real-time chemical sensing, and viscous liquids (schematic shown in Figure 3) of potential utility for the sensing of biological molecules in real time. A 2 µm long, 160 nm wide, 125 nm thick metalized string was operated with a frequency of 145 MHz and quality factor of 400 in room air, and with a reduced frequency of 95 MHz and quality factor of 5 in water [2]. The resonance responses for this device are shown in Figure 3.

References
Figure 2

Figure 3
Resonant Micro- and Nanomechanical Biosensors

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Abstract

In this work we fabricate micro- and nanoscale devices, and use them to detect biological analytes from solution. Masses that bind to these devices can be weighed by monitoring the frequency response of the resonating devices. Careful functionalization of sensor surfaces gives them high specificity, allowing frequency shifts to be associated with analyte detection. These devices are promising candidates for compact test platforms in the detection of disease, harmful biological substances, and important biomarkers in a low-cost and rapid way.

Summary

Micro and nanoelectromechanical systems (MEMS and NEMS) are of interest for chemical and biological sensor applications due to their high sensitivities and small sizes [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, sensitivity to added mass can be increased sufficiently for the detection of single bacteria [2], viruses [3] or molecules [4]. Resonant biosensors have demonstrated sensitivity to changes in mass as small as attograms [4]. The small scale of the devices encourages large scale arraying of sensors, building in appropriate redundancy and statistical samplings on a single chip. In addition, the high fill fraction of devices on a surface suggests that multiplexed detection of several different analytes from the same sample solution is possible.

Our devices are fabricated from low stress silicon nitride, with thicknesses on the order of 100 nm. Typical devices have lengths and widths on the order of hundreds of nanometers to a few microns. Variants of the standard cantilever beam shape, in addition to other interesting structures, are being investigated to improve sensitivity in the detection of dilute analytes. 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. Then we functionalize our devices with a linking chemistry specific to the desired analyte. A schematic of the fabrication process is shown in Figure 1 for an immunospecific cantilever sensor.

We use an optical system to both excite and detect device resonance. Light modulated at the resonant frequency of the sensor is used to periodically heat the device and drive the oscillations. Optical interrogation of devices is performed using optical interference techniques. As the sensor is resonating, the intensity of reflected light changes due to the varying gap between the device and silicon wafer. This method allows the exploration of large arrays of devices as well as creative and nontraditional device structures that would be difficult or impossible to detect using other techniques.

Figure 1: Fabrication schematic for a cantilever sensor functionalized with an immunological receptor chemistry.
For sensitive detection of biological entities, 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 like antibodies, for example, that are 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. Current work is directed towards the detection of small (on the order of 10s of kDa) proteins important in the early detection of disease. The small mass of these analytes exacerbates nonspecific binding issues and requires further optimization of blocking chemistries in order to prevent false positive signals.

References
Vibrational Manipulation of Microspheres Using Optically Excited In-Plane Motion of NEMS Oscillators

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Abstract
Optical excitation plays an important role in the actuation of higher flexural and torsional modes of nanoelectromechanical oscillators. We show that optical fields are extremely efficient for excitation, direct control and measurement of in-plane motion of cantilever-type nanomechanical oscillators. As a model system, 200 nm and 250 nm thick single crystal silicon cantilevers with dissimilar lengths and widths ranging from 6 to 12 µm and 500 nm to 1 µm, respectively, were fabricated using surface micromachining and dynamically analyzed using optical excitation and interferometric detection. Higher order modes of slender cantilevers, calculated using linear Euler-Bernoulli beam model, differed significantly from the measured values. Three dimensional finite element analysis incorporating shear, rotational inertia, cross-sectional deplanation and non-ideal boundary conditions due to the structural undercut, are shown to adequately describe the dynamics of the nanomechanical structures. The quality factor of a particular in-plane harmonic was consistently higher than the transverse mode. The increased dissipation of the out of plane mode was attributed to material and acoustic loss mechanisms. The in-plane mode was used to demonstrate vibrational detachment of sub-micron polystyrene spheres on the oscillator surface. In contrast, the out of plane motion, even in the strong non-linear impact regime, was insufficient for the removal of bound polystyrene spheres. Our results suggest that optical excitation of in-plane mechanical modes provide a unique mechanism for controlled removal of particles bound on the surface of nanomechanical oscillators [1].

Summary
Our cantilevers were fabricated from commercially available silicon-on-insulator (SOI) (100) wafers (SOITEC). The top structural silicon layer was 200 nm and 250 nm thick with a measured uniformity of less than 2%. Devices of varying length \( l = 6-12 \, \mu m \) and width \( w = 500 \, \text{nm-}1 \, \mu m \) were defined using optical projection lithography and etched with a CF\(_4\) plasma. The exposed 1 µm thick sacrificial SiO\(_2\) layer was removed using 49% hydrofluoric acid. Dynamic properties of the cantilevers were calculated and modeled for the geometry determined from optical and scanning electron microscopy. Rounding of the corners at the clamped and free end, due to optical proximity effects during lithographic definition of the beam, along with the undercut was incorporated into the three dimensional finite element model.

In order to identify the type of vibrational mode, the eigenfrequencies of the cantilever were first calculated and the corresponding eigenmodes were built using beam theory. The ratio of the two oscillatory modes (in-plane and out-of-plane) yields a dimensionless quantity \( \phi = w/t \). For the fabricated devices of width 1 µm and thickness 200 nm, \( \phi = 5 \). For the devices with dimensions \( l = 6.6 \, \mu m \), \( w = 1 \, \mu m \) and \( t = 200 \, \text{nm} \), the first eigenfrequencies for the transverse and in-plane modes are shown in Figure 2a and 2b respectively. The measured ratio between the two modes \( \phi_{\text{measured}} \approx 5.12 \) was in good agreement with the Euler-Bernoulli theory. With this oscillator length, second and third transverse harmonics were also measured, however, higher in-plane vibrational modes were not detected. Additionally, excitations above the third out-of-plane harmonic were not observed. This is attributed to the dramatic contrast of the compliance between the two modes. Neglecting rounding of the corners at the clamped and free end, calculated compliance for the transverse and in-plane directions were \( c_o = 0.855 \, \text{m/N} \) and \( c_i = 0.043 \, \text{m/N} \) respectively. However, with fabricated devices of lengths exceeding 8.5 µm, we measured up to the second in-plane mode.
In the following treatment, as a model system, evaluation of employing in-plane oscillations to remove weakly bound polystyrene spheres was considered. First, polystyrene spheres with diameter ranging from 500 nm to 1 µm diameter were randomly dispersed on a chip containing a periodic array of a large number (> $10^6$) of NEMS resonators. Oscillators containing single latex spheres were then identified and placed into a chamber that was evacuated to a pressure of $2 \times 10^{-7}$ Torr. NEMS devices with single latex spheres were baseline measured using a low excitation signal. In this work, vibrational excitation and motion detection were accomplished using an optical setup comprised of an amplitude modulated 415 nm diode laser in conjunction with a HeNe interferrometric system (Figure 1). The incident HeNe laser power was 56.4 µW during the baseline measurements. The excitation source was then swept from 500 µV to 30 mV. In most cases, the excitation source had to be moved to one side of the clamped end (base of the cantilever) in order to introduce a thermal gradient in an effort to amplify the in-plane vibrations.

Figure 2 shows the in-plane first vibrational mode with the sphere and following the removal of the sphere by shaking. Here the oscillator had dimensions $w = 601$ nm, $l = 10.12$ µm and $t = 250$ nm. Figures 3a and 3b show oblique angle scanning electron micrographs of the cantilever with a polystyrene sphere near the free end and directly following the removal of the bead respectively. Baseline measurements on a collection of oscillators with these dimensions had a natural out-of-plane harmonic of $3.16 \pm 0.12$ MHz. This was in excellent agreement with the results following the removal of the sphere.

References


Figure 1: Schematic of the optical excitation setup.

Figure 2: Measured in-plane harmonic spectra.

Figure 3: SEM of cantilever (a) with a sphere and (b) following its removal.
Dynamic Characterization of Nanomechanical Oscillators

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Abstract
Dynamic detection of vibrational characteristics of nanoelectromechanical systems (NEMS) was investigated through direct coupling with a micromechanical probe. The nanomechanical structures were harmonically driven using piezoelectric transducers and the resulting out-of-plane excitations were monitored with a conventional atomic force microscope (AFM) probe. Intermittent contact imaging data show quantitative linear classical resonance behavior. Additionally, non-contact AFM interrogation revealed the initiation of interaction between the two oscillators, providing a qualitative description of the resonant response. The vibrational spectra measured through optical excitation and detection was in good agreement with the coupled NEMS-AFM system measurement results. The dynamic response of the coupled system was modeled through a combination of long range van der Waals and contact forces using the Derjaguin-Muller-Toporov model. These results collectively demonstrate that this is a viable method for detecting the dynamic behavior of nanoscale suspended mechanical structures [1].

Introduction
The desire to understand and control dynamical properties of micro and nanoelectromechanical systems [1–4] (MEMS and NEMS) has inspired great interest in a wide spectrum of applications, including scanning force microscopy, ultrasmall force detection, single charge detection, magnetometry, cavity quantum electrodynamics, chemical and biological sensing, parametric resonance, data storage, and mechanical mixers and filters. Dynamic detection of forced deflection or out-of-plane translational vibrations is generally accomplished by either optical interferometry or optical deflection techniques. A fundamental constraint in optical detection is encountered when the device dimensions approach the diffraction limit of the optical system. In this regime, for a diffraction limited laser spot size at the position sensitive detector and noise dominated by shot noise, reduction of the total laser power reflected from the surface of the nanomechanical device significantly degrades the signal to noise ratio. To circumvent these restrictions, a number of detection techniques have been implemented, including, electrostatic, magnetic, piezoelectric, optical pumping and mechanical actuation and detection utilizing a combination of scanning tunneling and electron microscopes, respectively. These methods of forced actuation generally require supplementary fabrication steps such as deposition of additional layers on the oscillator surface, which in turn alters the mass and hence the dynamical properties of the resonator. Scanning probe microscopy represents an alternate approach to enhance and exploit the dynamical response of nanoscale systems through coupled mechanical interactions. Methods of measuring frequency response of large micromechanical structures using contact mode atomic force microscopy (AFM) have been reported. In these cases, the mass and stiffness of the structure under consideration were much higher than the AFM cantilever. With structures of reduced dimensions, the strongly interacting contact mode cantilever dominates the dissipative dynamics by dramatically modifying and broadening the vibrational properties of the probing NEMS device. Furthermore, due to this coupling, forces imposed during contact force imaging are sufficient to destroy small size and volume-to-surface ratio suspended structures.

Summary
In this article, we have studied the interaction between coupled MEMS and NEMS oscillators both experimentally and theoretically. This was based on an approach in which interactions between harmonically driven nanomechanical structures and an AFM probe in tapping and non-contact modes were used to illustrate the dynamics of the coupled system (Figure 1). For these experiments, suspended, surface micromachined single crystal, high frequency ($f_o$ 1 - 15 MHz) silicon NEMS cantilevers were fabricated.
and used in conjunction with commercially available AFM probes. The natural harmonic of the NEMS cantilever was much higher when in contrast with the probe with dimensions of length, $l = 225 \ \mu m$, width, $w = 30 \ \mu m$, and thickness $t = 3 \ \mu m$ and a resonant frequency in the range of 50-100 kHz.

Low force, tapping mode imaging near the free end of a nanomechanical cantilever subject to a harmonic excitation revealed the frequency response of the NEMS device. The spectral response was most pronounced in the phase and amplitude images of the oscillating probe. Figure 2 displays amplitude of the probe dynamics in the vicinity of the cantilever natural harmonic. The Lorentzian fit reveals a resonant frequency of 5.01 MHz with a mechanical quality factor, $Q = 3.9$ for a single crystal silicon device with dimensions $l = 7.92 \ \mu m$ and $w = 526 \ \text{nm}$.

In addition to topographic detection, the natural harmonic was measured using dynamic probing in intermittent and non-contact modes. In the former, the probe was positioned directly above the free end of the cantilever. The z-position of the probe was controlled by the extension and retraction cycle of the z-piezo. During the cyclic loading, in the fully retracted mode, the probe undergoes free oscillations. As the probe extends, it is lowered and brought in close proximity of the surface, the amplitude decays and finally jumps into contact with the surface. The point of contact marks a significant decrease in the vibrational amplitude of the probe (see Figure 3). The amplitude reaches a plateau throughout the range of the contact extension cycle. During the probe-cantilever retracting cycle, the vibrational amplitude remains approximately constant until the point when the probe breaks contact with the surface. The amplitude gradually increases and reaches a steady state value within the free vibration regime. Figure 4 shows the measured frequency spectra of the driven cantilever structure within the intermittent contact regime.

References

Mechanics of Nanometer-Thick Suspended Carbon Materials

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Abstract
We fabricated nanoelectromechanical systems (NEMS) from graphene sheets by mechanically exfoliating thin sheets [1, 2] over trenches in silicon dioxide substrates. The thinnest resonator consists of a single suspended layer of atoms and represents the ultimate limit of a two dimensional NEMS.

Background
In collaboration with the McEuen and Davis research groups, we continue to work on patterning, control and suspension of carbon nanotubes, but our major focus has been on suspended thin films of graphite, down to single atomic layers of graphene.

![Figure 1: An optical micrograph of a suspended few-layer graphene sheet also measured to be 2 nm-thick by AFM.](image1)

Summary of Research
An optical micrograph of a suspended few-layer graphene sheet is shown in Figure 1. This particular membrane was measured to be 2 nm-thick by atomic force microscopy (AFM). Vibrations with fundamental resonant frequencies in the megahertz (MHz) range are actuated either optically via thermal expansion and contraction [3], or electrically by applying a radio frequency voltage relative to a doped silicon back-gate [4]. Motion is detected optically by laser interferometry. We detect the thermal motion of the resonators, and use the equipartition theorem to calibrate the amplitude of motion with the optical signal. AFM and spatially resolved Raman spectroscopy are used to determine the thickness of the suspended sheets [5].

![Figure 2: A surface plot of the spring constant of a suspended graphene sheet vs. the location of the AFM tip.](image2)
Mechanical properties are measured using calibrated AFM probes [6] to measure static deflections in response to a known point force. We have made a detailed study of the mechanical properties of these resonators including resonance frequency, spring constant, built in tension, and quality factor. Figure 2 shows a series of measured values for the effective spring constant across the surface of a stack of suspended graphene sheets which are shown in an AFM image in Figure 3 with a corresponding grid. On layered graphene sheets of thicknesses between 2 nm and 8 nm, we measured spring constants ranging from 1 to 5 N/m. Our data is fit to a model for doubly clamped beams under tension by plotting the spring constant, as measured in the center of the device, vs. $w(t/L)^3$ for eight different suspended graphite sheets. As shown in Figure 4, we can use this fit to extract a Young’s modulus of 0.4 TPa, compared to 1 TPa for bulk graphite along the basal plane, and tensions on the order of 10-7 N [7].

The unusually small mass, electrically active material and reasonable dynamic range indicate that graphite resonators would make excellent mass and charge sensors.

References

An Electronic Nonvolatile Memory Device Based On Electrostatic Deflection of a Bistable Mechanical Beam

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Abstract

The speed of memory structures is considerably slower than that of logic thus permitting a variety of approaches to achieving and using bistability. We used the bistability of a doubly clamped beam to attain two states that can potentially be used in the implementation of a nonvolatile memory [1]. The device is designed such that a bistable beam acts as the gate of an air gap transistor. In theory, in scaled structures, the threshold voltage shift corresponding to a gate deflection of nanometer dimensions provides sufficient change to allow sensing of the state of the nonvolatile memory cell.

Summary of Research

The bistable mechanism has found numerous applications in the microelectromechanical systems (MEMS) field related to mechanical switches and valves. Experiments by B. Halg [2] have demonstrated that it is also conceivable to use mechanical bistability in a nonvolatile memory cell. Similar work [3, 4] has shown simulations of the electrical characteristics of a metal oxide semiconductor field effect transistor (MOSFET) with a buckled floating gate. We have refined these concepts further and put them into practice by designing and fabricating a novel structure suitable for the integration of an FET (Figure 1). In addition, the process developed for this device is completely complementary metal oxide semiconductor (CMOS) compatible.

The write/erase mechanism of this memory device operates on the principle of electrostatic attraction. The plate is incorporated in the device to help perform the write function. The upper plate is nearly four times thicker than the gate thus making it highly stiff by comparison. This establishes the gate as the only deformable element of the device. Therefore, in order to obtain the “down” state (Figure 2), a large bias is applied between the gate...
and the source/drain, thus attracting the gate towards the channel. Upon removal of the voltage, the gate remains in the “down” position due to it being energetically favorable as a result of the bistable mechanism. On the other hand, in order to obtain the “up” state (Figure 3), a large bias is applied between the upper plate and the gate so that the gate is pulled up to the other stable position. The read function is executed using small biases between the gate and the substrate. In read mode, the device operates like a MOSFET. The read voltages must be lower than the write voltages in order to prevent any additional deflection of the gate. The device’s electrical characteristics substantially change as the gate is moved between the two stable states. The “down” state corresponds to a lower threshold voltage and sub-threshold slope, whereas the “up” state is described by a higher threshold voltage and sub-threshold slope.

Our device features a bistable polysilicon beam sandwiched between two silicon dioxide layers as seen in Figure 1. The bistable beam is obtained by patterning and releasing a compressively stressed thin film with sufficient internal stress to cause buckling upon release. This enables the bistable mechanism; the beam can be deflected either up or down, thus creating two stable states. The middle polysilicon layer is deposited using the low pressure chemical vapor deposition (LPCVD) furnace at 620°C for 12 minutes which results in a film thickness of 80 nm. In addition to the beam used for the gate, another beam or plate is suspended above the gate. The plate is also comprised of LPCVD polysilicon and has a thickness of 330 nm. The two silicon dioxide layers are both 1.1 µm thick and are made of low temperature oxidation (LTO). These dual LTO films serve not only as the sacrificial release layers, but also act as the anchors for the suspended beams. Furthermore, they also provide electrical isolation between the plate, beam, and substrate. After depositing a stack of the previously stated materials, a top down approach is taken to fabricate the devices. A bi-layer resist liftoff process is performed to leave 40 nm thick Cr features. Cr serves as a robust etch mask for the subsequent plasma etching. The ICP etcher was chosen for its enhanced etch rates and its ability to produce higher aspect ratio features than the standard parallel plate RIE systems. The stack was etched in CF₄ plasma and the etching was stopped in the bottom LTO layer. Etching was then continued in the parallel plate RIE system with CHF₃/O₂ to provide higher selectivity to the silicon substrate. In order to open up a via to the gate electrode a thick resist was used to protect the entire structure. A window was then opened in the resist so that the ICP etcher could be used to etch partially through the stack and stop in the top LTO layer. The resist was then stripped and the structure was dipped in BOE to complete the etching of the via while also removing the dual sacrificial oxide layers. Upon release, the beams were equally likely to buckle to the up or down state due to the highly symmetric design of the structure.

The design for this bistable mechanical memory device permits straightforward fabrication, thus making it possible to scale this structure down further. Having both the beam and plate defined in the same etch allows for a self-aligned process. In addition, the FET channel runs perpendicular to the beam-plate structure, thus permitting self-aligned implants. By incorporating novel and more compliant materials in this structure, device performance can be further improved, making this a highly competitive technology.

References

Ultrasensitive, Magnet-Tipped Cantilevers for Magnetic Resonance Force Microscopy

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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. Figure 1 is of a 50 x 50 x 1000 nm overhanging Co magnet on a test structure. The dotted line outlines the location of the cantilever edges in the final product. Figure 2 shows the entire cantilever and base. The octagonal area on the cantilever is the laser pad for our interferometer based motion detection system.

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...
The work in the past year has focused on process integration. After exhaustive testing, we suspect that cobalt silicide formation is causing the degradation of the magnets during the Unaxis backside etch step. Our current work is in modifying the backside etch process to minimize heating of the magnets, and seeking ways of reducing or eliminating the PECVD oxide layers.

**References**

Magnetic Resonance Force Microscopy
Cantilever Detection by Quantum Tunneling

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Abstract
Magnetic resonance force microscopy is a novel characterization technique, with promising applications to nondestructive three-dimensional sample imaging. Magnetic resonance is detected as force on a magnet-tipped cantilever, with potential for atomic scale magnetic resonance imaging of biomolecules or semiconductors [1]. To achieve such a lofty characterization goal, high-Q nanomechanical magnet-tipped cantilevers are required. Here, we present a fabrication project in progress at the CNF that aims to appease this need; the integration of tunnel-based displacement detectors into the MRFM cantilevers.

Summary of Work
Magnetic resonance force microscopy (MRFM) aims to interrogate nuclear spins locally by setting the Larmour spin resonant condition with high spatial selectivity [1]. This is accomplished by a nanomagnet on the tip of an RF nanoscale singly-clamped cantilever. In an attempt to eliminate coupling between the cantilever and detection interferometer, we are considering a tunnel-based displacement detector. The tunnel sensors themselves consist of a break junction [2] between the beam and a fixed electrode; as the beam oscillates, the junction separation changes, and the tunneling current through the junction varies measureably. Calculations have shown that the measurement sensitivity is approximately $10^{-13} \text{m/}\sqrt{\text{Hz}}$, well within that required for MRFM. More importantly, this technique is viable around ambient temperatures of 300 K, while alternative methods (RF-SET’s, etc.) require cooling. The tunnel sensors are patterned with the JEOL JBX-9300 dedicated electron-beam lithography system, with a feature size of 30 nm. Optical lithography is used to define the interconnects to the probe station. RIE is then used to define the cantilevers and junction underetch.

To date, we have generated a fabrication protocol for tunnel sensors in doubly clamped cantilevers. We are currently characterizing the doubly-clamped beams with the magnetomotive actuation technique. Upon completion of the characterization, we will integrate the tunnel-based displacement detection mechanism with the nanoscale tip magnets (in a singly-clamped beam) to create a new generation of MRFM cantilevers.

References
Figure 1: Here, we see an array of fabricated tunnel sensors.

Figure 2: Up close, one can see the separation between the tunnel sensor and fixed electrode.
**AFM Tip Processing on Hinged Cantilevers**

**CNF Project # 883-00**

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**Abstract**

A method for atomic force microscopy (AFM) tip preparation is described that is amenable for integration onto a hinged cantilever, thereby achieving an exquisitely high-sensitive probe. Undercutting an etch mask with successive reactive ion etching, results in a quasi-pyramidal, high-aspect-ratio tip. Processing steps will be discussed, scanning electron microscopy (SEM) images will be shown, and a short narrative of how the tips are integrated onto the cantilevers will be described, including future directions of the work.

**Summary**

Few modern instruments can lay claim to the level of success enjoyed by the atomic force microscope (AFM). The ability to map surface topography with ~ nm resolution finds usefulness in almost every branch of science.

The original AFM employed a scanning tunneling microscope (STM) to monitor the motion of a diamond-tipped stylus supported on a gold foil cantilever [1].

Today’s AFM typically employs a silicon micromachined cantilever with a sharp tip on one side, and a reflective mirror surface on the other [2, 3]. When the tip interacts with the test surface, its deflection is measured by reflecting a laser beam off the back side of the cantilever onto a 4-segment position-sensitive photodetector (PSPD). Whereas the top and bottom segments of the detector indicate height variations in the position of the tip, the left and right segments can be used to indicate the amount of friction between the tip and sample surface [4]. However, because the same cantilever is used in the vertical bending mode (for topographical imaging) and the lateral bending mode (for frictional force imaging) these motions are inextricably coupled with mechanical crosstalk resulting.

Chui [5] was arguably the first to appreciate decoupling of the lateral and vertical sensitivities in an AFM cantilever. Later, Kageshima [6] was able to mill a trench into a commercial AFM cantilever with a focused ion beam (FIB), thereby enhancing both the lateral and vertical sensitivity.

Taking both of these concepts a step further, Beyder [7] was able to process silicon cantilevers with orthogonal (i.e. gimbaled) Si₃N₄ hinges of arbitrary thickness, thereby decoupling vertical and lateral sensitivities altogether. This allowed the geometry and stiffness to be separately optimized for each sensing direction. With arbitrary thickness hinges, Beyder is able to achieve exquisitely sensitive levers for the analysis of delicate soft-matter samples. Figure 1 exemplifies a dual-axis gimbaled lever with two tips (courtesy of Beyder).
In all of these cantilever arrangements, the tip that interacts with the test surface greatly influences the quality of the AFM image. Whereas the radius of curvature of the tip determines, in part, the imaging resolution of the cantilever, the shape of the tip can be more or less susceptible to image aliasing and artifacts that seriously degrade the final image [8].

At the CNF, silicon AFM tips are defined by a sequence of three reactive ion etch (RIE) processes: (1) isotropic etch to undercut the etch mask (O-Release), (2) anisotropic etch to lengthen the underlying pillar that results (O-Trench), and (3) a second isotropic etch to sharpen the pillar into a final quasi-pyramidal tip (O-Release).

A ~ 5 µm diameter circular etch mask comprising ~ 1.3 µm of S1813 (Shipley) photoresist, 30 nm of LPCVD nitride (low-stress, 800°C, 11 min) and 135 nm of thermal oxide (wet, no-HCL, 900°C, 65 min) is prepared.

For undercutting the etch mask, we utilize the default O-Release recipe in the Unaxis 770 ICP (inductively coupled plasma) tool. The O-Release program uses four etch steps of Ar (50 sccms) with increasing flowrates of SF₆ (20, 40, 60 and 70 sccms, respectively) at 850 watts of RF power. The duration of each of the four etch steps is 2, 2, 2 and 60 sec respectively. The resulting undercut etch mask is shown in Figure 2 with an underlying, partially-obscured pillar supporting the mask. On top of the mask, the resist is deformed into a 4-leaf clover-like formation.

To lengthen the pillar, we use the default O-Trench recipe on the Unaxis ICP tool. Each loop of the O-Trench program consists of a first polymer deposition step (70 sccms of C₄F₈, 2 sccms of SF₆, and 40 sccms of Ar, 5 sec at 850 watts RF-ICP) followed by a first etch step (2 sccms of C₄F₈, 70 sccms of SF₆ and 40 sccms of Ar, 2 sec at 850 watts RF-ICP) and a second etch step (2 sccms of C₄F₈, 100 sccms of SF₆ and 40 sccms of Ar, 5 sec at 850 watts RF-ICP). Five loops of the O-Trench recipe results in the pedestal/cap structure of Figure 3.

A final O-Release etch for 60 sec (in the last etch step) pinches-off the cap, and transforms the underlying pillar into a quasi-pyramidal tip, as shown in Figure 4.

Implementing one or more of these silicon AFM tips onto a single-axis or double-axis cantilever is done by lithographically defining the arms and hinged regions of the cantilever(s) after tip preparation.

Future work may include polarimetric encoding of the reflected optical signals from multiple, reflective regions on the back of a multi-axis, multi-tipped cantilever. Such encoding practices would allow a considerable increase in information content conveyed from the test surface.

The results presented in this work are due in part to the knowledge, expertise and superlative support provided by the CNF staff. This work was supported by the NIH and NSF.

References

Functionalized Micromechanical Resonators with High Quality Factors

CNF Project # 891-00
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Abstract

The dissipation of mechanical energy in 250-nm-thick, megahertz (MHz)-range silicon resonators is found to be strongly dependent on the chemical nature of the surface. As a result, sub-monolayer changes in the termination of 250-nm-thick resonators lead to significant changes in quality factor. The chemical origins of this effect are under investigation. Strategies for the formation of arbitrarily functionalized, high-\(Q\) resonators have been demonstrated.

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, \(Q\). (The \(Q\) of a resonator is proportional to 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.) Therein lies the problem. Although advances in microfabrication have made the production of high frequency resonators almost routine, a general trend of decreasing quality factor with decreasing resonator size has been observed.

We have shown that mechanical energy dissipation in 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 250-nm-thick silicon resonator—less than 0.07% of the total mass—the quality factor of the resonator can be improved dramatically [1]. In contrast, the standard commercial coating, a thin layer of silicon oxide, dissipates at least 75% of the mechanical energy in similarly sized resonators. This result shows that chemical control down to the monolayer level will be necessary for the production of high-performance nanomechanical sensors. We are working to understand and control this effect.

In recent experiments [2], we have shown that silicon oxidation does not inherently lead to increased mechanical energy dissipation. When silicon surfaces are oxidized in a controlled, strain-free manner—such as by the introduction of one-half monolayer of Si-O-R surface groups—increased dissipation is not observed. This
result is important, because it opens the door to a much broader range of functionalization chemistries for high-performance sensor applications.

In related work, we have developed a robust platform chemistry for the production of high quality resonators with essentially arbitrary organic functionality. This process uses Grignard reagents to produce a partially alkene-coated surface followed by a catalyzed olefin methathesis reaction that attaches the desired functionality to the surface alkenes. This strategy is sketched in the accompanying figure. As proof of concept, we have produced ester-functionalized resonators with quality factors near those of our best performing surface coatings.

References


Electromechanical Resonators from Graphene Sheets

CNF Project # 900-00
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Abstract

Nanoelectromechanical systems were fabricated from single and multilayer graphene sheets by mechanically exfoliating thin sheets from graphite over trenches in SiO2. Vibrations with fundamental resonant frequencies in the MHz range are actuated either optically or electrically and detected optically by interferometry. We extract a Young’s modulus of 1 TPa, and find that most suspended sheets are under tension. The quality factors of the suspended graphene sheets are in the range of 20-850 and show no dependence on the thickness. The thinnest resonator consists of a single suspended layer of atoms and represents the ultimate limit of two dimensional nanoelectromechanical systems.

Research Summary

The miniaturization of electromechanical devices promises to be revolutionary in the coming decades as the miniaturization of electronic devices was in the previous ones. Devices ranging from nanoscale resonators, switches, and valves have applications in tasks as diverse as information processing, molecular manipulation, and sensing. The prototypical nanoelectromechanical system (NEMS) is a nanoscale resonator, a beam of material that vibrates in response to an applied external force. The ultimate limit would be a resonator one atom thick, but this puts severe constraints on the material. It should be robust, stiff, and stable as a single layer of atoms.

Graphite consists of stacked layers of graphene sheets separated by 0.3 nm and held together by weak van der Waals forces. It has extremely high strength, stiffness, and thermal conductivity along the basal plane. In addition, graphite can be exfoliated onto an insulating substrate, producing micron-sized graphene sheets with thicknesses down to a single atomic layer. Thus far, research on these thin graphene sheets has focused primarily on their electronic properties. We demonstrate a method of suspending single and multilayer graphene sheets over trenches and show such sheets can be mechanically actuated. This work also makes a detailed study of the mechanical properties of these graphene resonators including resonance frequency, spring constant, built in tension, and quality factor.

Suspended graphene sheets are fabricated using a mechanical peeling process where the graphene sheets are exfoliated off of bulk graphite predefined trenches etched into a silicon oxide surface. Figure 1 shows that these sheets are optically visible under a microscope. The result is a micron-scale doubly clamped beam or cantilever clamped to the silicon oxide surface via van der Waals attraction (Figure 2). Some devices have pre-patterned gold electrodes between the trenches to make electrical contact [1]. Using this method, we have fabricated suspended sheets with thicknesses varying from a single atomic layer to sheets 75 nm thick.

The resonators are actuated using either electrical (Figure 3) or optical modulation. In the case of electrical modulation, a time-varying radio frequency (RF) voltage is applied to the graphene sheet resulting in an electrostatic force...
between the suspended graphene sheet and the substrate. For optical actuation, the intensity of a diode laser focused on the sheet is modulated at the resonant frequency of the graphene sheet, causing a periodic contraction/expansion of the layer that leads to motion. In both cases, the motion is detected by monitoring the reflected light intensity from a second laser using a fast photodiode. Figure 4 shows the measured amplitude versus frequency of a single atomic layer graphene resonator, with a measured resonance frequency of 70.5 MHz and \( Q = 78 \). The resonance frequencies of the fundamental modes of the fabricated sheets vary from 1 MHz to 166 MHz with quality factors, \( Q \) of 20-850.

By comparing the resonance frequency of the suspended sheets to the measured size dimensions, we can deduce a Young’s Modulus of 1 TPa using continuum beam mechanics. However, the frequencies of thinner resonators (under 7 nm) show more scatter with the majority having resonant frequencies significantly higher than predicted by bending alone. A likely explanation for this is that many of the resonators are under tension, which increases the resonance frequency. The tension likely results from the fabrication process, where the friction between the graphite and the oxide surface during mechanical exfoliation stretches the graphene sheets across the trench.

An important measure of any resonator is the normalized width of the resonance peak characterized by the quality factor \( Q = f_o/\Delta f \). A high \( Q \) is essential for most applications, as it increases the sensitivity of the resonator to external perturbation. We measure \( Q \) in the graphene sheets under vacuum of 20 to 850. This contrasts with diamond NEMS (2000-4000), silicon nitride NEMS (up to 400,000 now), and carbon nanotubes (50-100). Preliminary studies on a 20 nm thick resonator found a dramatic increase in \( Q \) with decreasing temperature (\( Q = 100 \) at 300 K to \( Q = 1800 \) at 50 K). This suggests that high \( Q \) operation of graphene resonators should be possible at low temperatures.

The high Young’s modulus, extremely low mass, and large surface area make these resonators ideally suited for use as mass, force, and charge sensors. For a 5 nm suspended sheet where we can detect the thermal vibration, we infer that we should be capable of measurements with a mass sensitivity of 7 zg, a force sensitivity of 0.9 fN/Hz\(^{1/2}\), and a charge sensitivity of 8 \( \times 10^{-4} \) e/Hz\(^{1/2}\). These values are competitive with state of the art silicon NEMS at room temperature, and could get much better at lower temperature with the onset of higher \( Q \).

However, the application of graphene NEMS extends far beyond just mechanical resonators. This robust, conducting, membrane can act as a nanoscale supporting structure or atomically thin membrane separating two disparate environments.

References

Temperature Measurement of Boiling Flow in Microchannels with Reentrant Cavities

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Abstract

Experiments were conducted to investigate flow boiling in 200 µm x 253 µm parallel microchannels with structured reentrant cavities. Flow morphologies and local temperature measurements were obtained and studied.

Introduction

Boiling enhancement technique has been a topic of great interest over the last fifty years [1], and is a rich field of current study. Of particular engineering success are special heat transfer surfaces encompassing re-entrant cavities that promote bubble ebullitions [2, 3]. Inspired by the rapid development of flow boiling in micro domains, research endeavors aimed at exploiting recent advances in microfabrication technologies to form structured surfaces that enhance heat transfer for flow boiling are currently underway [4-6]. The current investigation presents an examination of flow boiling through 223 µm hydraulic diameter microchannels with re-entrant type cavities. Flow boiling patterns and local temperature measurements are presented and discussed.

Device Overview and Experiment Procedures

The silicon microchannel device consists of five parallel 10,000 µm long, 200 µm wide and 253 µm deep microchannels spaced 200 µm apart. Each sidewall encompasses an array of 100 re-entrant cavities spaced 100 µm apart, as shown in Figure 1. An acute angle connects the 7.5 µm mouth to the 25 µm inside diameter reentrant body. In order to minimize ambient heat losses, an air gap is formed on the two ends of the side walls, and an inlet and exit plenum are etched on the thin silicon substrate (~150 µm). A 20 µm wide and 400 µm long orifice is installed at the entrance of each channel inlet to suppress flow instabilities. The microchannel, re-entrant cavities, and orifices were all fabricated from deep reactive ion etch (DRIE) process. At the top of the device, a Pyrex® cover seals the device from the top and allows flow visualization.

Figure 1: SEM image of reentrant cavities.

Figure 2: CAD models of the heater and the thermistors on the back side of the micro device.
Mechanical Devices

Figure 2 depicts a CAD model of the backside of the device. Three aluminum thermistors 10 µm wide and 300 µm long are located 3,400 µm, 6,700 µm and 10,000 µm downstream of the channel inlet together with electrical connecting vias. On top of the thermistors layer, a 1 µm silicon oxide layer is deposited for electrical insulation. An aluminum heater is then formed on top of the oxide layer to deliver the heating power and also serves as a thermistor to measure the average temperature of the entire microchannel area.

The microchannel device is mounted on the fluidic packaging module through o-rings to ensure a complete leak-free system. The fluidic packaging delivers the working fluid (water) and access to the pressure transducers. The heater, which is fabricated on the device backside, is wired (through electric pads) to the power supply. The thermistors are also connected to a data acquisition system. The boiling process in the microchannels is recorded by a high-speed camera mounted over a microscope. During the experiment, voltage was applied in 0.5 volt increments to the test section heater, and the data for the heater and the thermistors were recorded once steady thermal-hydraulic state was reached, at which water flow rate, heat input, and temperature data remained constant. The procedure was repeated for different flow rates.

Results and Discussion

Four prime flow patterns were visualized in the reentrant cavity microchannels prior to the critical heat flux (CHF) conditions: single-phase, bubbly (Figure 3), slug, and annular flow patterns. The extent of the bubbly flow pattern was shown to be dependent on the mass velocity and heat flux. The slug flow was characterized by a large vapor core that occupied the entire microchannel cross-section and oscillated rapidly over several hydraulic diameters. Downstream, a more stable annular flow was developed, which was characterized by a vapor core engulfed by a continuous liquid sublayer attached to the microchannel walls. As the mass quality increased, the liquid layers diminished until dry spots at the exit region were detected indicating the arrival of the critical heat flux conditions.

Figure 4 shows $T_2$ as a function of the effective heat flux ($q''_{eff}$) for four different mass velocities. As expected, at low heat fluxes, single-phase liquid flow prevails, which is depicted by the linear temperature increase with increasing surface heat flux. When the heat flux is further increased, a significant shift in the $T - q''_{eff}$ slope is apparent marking the onset of nucleate boiling. Flow visualization reveals small bubbles emerging from the reentrant cavities near the channel exit. As the heat flux was further increased above a certain value, the average surface temperature abruptly surged with a meager rise of heat flux, indicating the emergence of critical heat flux condition. The CHF was verified by flow visualizations of dry spots at the channel exit region.

References

Directional Microphone Array Fabrication

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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.

Summary of Results
Several new microphone designs and fabrication processes were developed and refined this past year. In collaboration with Prof. Levent Degertekin at Georgia Tech and Prof. Douglas Jones at the University of Illinois Champaign-Urbana, we have developed a prototype packaging scheme for our microphones so that our directional microphone array is small enough to fit on a hearing aid, as shown in Figure 1. The microphone array consists of two differential microphones and a nondirectional microphone located on the same silicon chip. The microphone package contains optoelectronic components to produce electronic outputs from the three microphones.

The microphone diaphragms we are developing are designed to respond to spatial gradients in the incident sound pressure by rotating about hinge supports as shown in Figure 2. An optical detection scheme has been developed to convert the sound-induced motion of the diaphragm into an electronic signal as shown in the figure. This consists of a semiconductor light source (VCSEL), an optical grating on the diaphragm, and photodetectors. We have shown that this optical scheme, along with the novel diaphragm design, has resulted in a high sensitivity and low-noise directional microphone.

The internal noise of miniature directional microphones is a significant design issue because the acoustic sensitivity of the device reduces with the diaphragm size. Figure 3 shows measured sound input-referred thermal noise of the microphone along with the results of an analytical prediction. This small directional microphone, with diaphragm size of 1 mm by 2 mm, has a thermal noise floor of 36 dBA, which is much lower than can be achieved in a directional microphone using conventional technology with a diaphragm of this size.

Figure 1: The prototype microphone package is small enough to fit in a hearing aid.
Figure 2: Schematic showing the optical sensing scheme to detect the sound-induced motion of the diaphragm.

Figure 3: Measured sound input pressure-referred thermal noise of our 2 mm by 1 mm directional microphone diaphragm.
Fabrication of Thin-Walled and High-Aspect-Ratio Nanofluidic Channels

CNF Project # 1176-03
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Abstract

Nanofluidic structures promise to solve the sample preparation problem in various spectroscopy experiments where the thickness of the sample is usually constrained by the penetration depth of the optical probe. We have designed and successfully fabricated a nanofluidic cell which was used in a two-dimensional infrared (2D-IR) spectroscopy experiment to study the nature of the OH bond in water [1, 2]. The fabricated structure employs a system of access holes and channels used for sample delivery and active thickness control.

Summary of Research

The building blocks of the nanofluidic cell are two free standing membranes of low stress silicon nitride back-etched on separate wafers using potassium hydroxide (KOH). A silicon oxide spacer deposited on one wafer was used to define a gap between the silicon nitride membranes when the matching pieces of the structure are bonded together. The etching of the silicon oxide layer to create the actual gap is done with 6:1 buffered oxide etch (BOE). In the first generation cell, the thicknesses of the silicon nitride windows were 800 nm, and the cell was filled from side channels, then sealed, trapping the liquid inside. In such a passive approach, the instability of the thin silicon nitride membranes leads to a poorly defined sample thickness.

In recent designs, input and output access holes were etched into the surface of the structure as shown in Figure 1, then connected to an external pump system. The active control of the gap thickness was realized using the transmitted intensity of an IR beam through the cell as feedback signal. In addition, a 70 µm deep KOH etched channels were used to connect the narrow gap to the access holes therefore restricting the high flow resistance region to the sample area. Lastly, a hydrophilic surface is created by high temperature deposition (LPCVD) of ~ 10 nm silicon oxide [3] in order to enhance the filling of the cell. An assembled cell filled with water is shown in Figure 2. The variation in sample thickness is most apparent from the interference rings.

References


Mechanical Devices

Figure 1: Schematic of nanofluidic device for nonlinear spectroscopy on thin sample liquids.

Figure 2: Nanofluidic cell filled with water. Edge thickness is ~ 1500 nm, center thickness is ~ 400 nm, window size 1 x 1 mm.
Fabrication of Gecko-Inspired Spatula Tips

CNF Project # 1201-04

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Abstract

Insects such as flies and small animals such as geckos employ micro and nano-scale fibers on their feet to achieve excellent attachment capability. However, these animals must also possess the ability to quickly switch between good adhesion and easy detachment, which is essential for locomotion. It is thought that the geometry of the foot fibers (tilted fibers) and that of the fiber tips (asymmetric arrangement) play a central role in achieving such a switching mechanism. In this project, we aim to fabricate a gecko-inspired surface with asymmetric tips, similar to an inverted “L”. Such surfaces are expected to possess interesting anisotropic adhesion and frictional properties of technological interest.

Summary

Following the recent work [1] in demonstrating that the micro and nano-scale architecture of the gecko foot fibers is responsible for the animal’s excellent attachment capability, there have been several investigations to describe the mechanics of insect attachment as well as to create surface engineering strategies to mimic it [2-6]. Some of the successful strategies so far include arrays of fibers with mushroom shaped tips [2-3], film terminated fibrillar arrays [4], surfaces with wrinkled patterns [5], arrays of aligned carbon nanotubes [6], etc. A key feature of gecko and other insect attachment is the ability to quickly switch between strong adhesion and easy detachment, which is essential in locomotion. It is thought that the fiber geometry and the fiber tip geometry play an important role in such a switching mechanism.

One of the goals of this project is to investigate the mechanics of individual mechanisms which contribute to the switching mechanism and develop surface engineering strategies of technological interest.

In order to fabricate polydimethylsiloxane (PDMS) fiber tips with asymmetric tips, we developed a process in which a silicon mold of the corresponding shape is fabricated. We employ photolithography with backside alignment and deep silicon etching to prepare the mold. The mold is then filled with PDMS and cured. The silicon mold is then removed through a combination of wet and dry etching processes. The initial results, as exemplified in Figure 1, demonstrate the feasibility of the fabrication process. The process is currently being refined to achieve, more precise control of surface quality and to minimize the tendency of the fibers to adhere to each other during the release process. Detailed mechanical testing will be reported in the near future.

References

Figure 1: SEM image of PDMS fibers with inverted “L” shape.
High Sensitivity Uncooled Microcantilever Infrared Imaging Arrays

CNF Project # 1202-04
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Abstract
Multispectral Imaging, Inc., is developing and manufacturing infrared focal plane arrays (FPAs) of 160 x 120 pixels, 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 complimentary metal oxide semiconductor (CMOS) integrated circuit, and all microcantilever materials are compatible with standard silicon IC foundry processing.

Summary
Uncooled vanadium oxide (VOₓ) and amorphous silicon (a-Si) microbolometers are presently the technologies of choice for thermally sensing and imaging long wave infrared radiation. However, the performances of these devices have 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, Inc., (MII) has licensed the ORNL technology [4-6] and is pursuing its commercialization using the resources at Cornell NanoScale Science and Technology Facility (CNF).

Figure 1: Schematic diagram showing the operating principle of the bimorph microcantilever IR sensor.

Figure 2: Scanning electron micrograph of a portion of 160 x 120 MEMS imaging array. Inset: Close-up of a pixel in same array. Released height of the sensor structure is controlled using tensile and compressive stresses in the thin film layers used to fabricate the microcantilever.
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 heat sensing bimorph microcantilever structures are fabricated directly onto the CMOS control and amplification electronics to produce a high performance, low cost imager.

MII has fabricated the first batches of fully integrated 160 x 120 FPAs at CNF with typical pixel functionalities ranging from 80% to > 95%. MII has an ongoing development program with Dalsa Semiconductor to commercialize this technology and we expect pixel yields of > 99% when in full production. Positional responsivities of greater than 0.3 µm/K have been modeled and measured for 50 µm pitch pixels, corresponding to a temperature coefficient of capacitance, ΔC/C, (equivalent to TCR for microbolometers) above 30%/K. This responsivity, along with noise-equivalent capacitances in the sub-attofarad range and nominal sensor capacitances of 15 fF, give modeled noise-equivalent temperature differential (NETD) of < 20 mK for these devices.

Preliminary infrared imagery has been obtained with a recently fabricated imaging array, assembled camera and control system with NEDTs in range of 350 mK using f/0.86 optics.

References

Ultra-High Density MEMS-Based Interconnect for Wafer-Level Ultra-Thin Die Stacking Technology

CNF Project # 1260-04
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Abstract

This work describes a novel smart three axis compliant (STAC) interconnect targeted to revolutionize chip-to-chip and chip-to-board high-density three dimensional (3D) integration for ultra-thin Si dies (≤ 75 µm) at the wafer level. The STAC interconnect is a 3D-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 latter type of chip integration is more commonly known as three-dimensional (3D) packaging or chip stacking technology.

STAC interconnects are MEMS-based electrical interconnect that are microstructurally engineered to accommodate the relative displacements between ultra-thin through silicon vias (TSV)-based Si chips and the 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]:

$$ R = \frac{Y'h}{G} $$

where $Y'$ is the biaxial modulus, $h$ is the interconnect film thickness and $G$ is the interconnect film stress range.

STAC interconnects were formed using a titanium tungsten (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 base 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.
Figures 1 and 2 show successfully fabricated STAC interconnects using the microfabrication steps described in the previous paragraph. The total beam length is 150 µm with 25 µm of its length fixed to the substrate (fully covering the Cu pad), and the beam width is 25 µm. Each interconnect is spaced 50 µm (width wise) by 170 µm (length wise) pitch, bringing the total number of STAC interconnects on a 1 cm² chip to 11,200. The total number of I/O exceeds the long term requirement of the International Technology Roadmap for Semiconductors (ITRS) 2005. Since these interconnects were defined with photolithography and dry-etch chemistry, the exact beams shown in Figure 1 and Figure 2 can potentially be spaced at 30 µm (width wise) by 155 µm (length wise) pitch with a new mask, bringing the total number of compliant interconnects on a 1 cm² chip to beyond 21,000.

References
MEMS Fabricated Vapor Cells

CNF Project # 1262-04

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Abstract

Vapor cells for atomic probing/pumping have been fabricated using a double-side polished silicon (Si) wafer. The cavity is formed by a potassium hydroxide (KOH) through-etch in the Si, with windows formed by an anodic bonded Pyrex® wafer and by a low pressure chemical vapor deposition low stress (LPCVD LS) nitride membrane. A glass capillary is sealed to the cavity, then used to pump down and refill the vapor cell.

Summary of Research

Vapor cells for atomic probing were fabricated in bulk Si. Two cavities are required: one to form the probe region, and another to act as the connection to the pump system and the gas. A channel was fabricated between the two cavities. In addition, LPCVD LS nitride was used as the membrane to cap the cavities. Finally a Pyrex wafer was anodic-bonded to finish the process.

The process is shown in Figure 1. First, a 40 µm channel is formed on the top side of a double-side-polished (DSP) Si wafer using a KOH etch with an oxide mask. Next, a micron of LPCVD LS nitride is deposited to act as a mask and, later, as a window membrane. The nitride is patterned with squares with sides of several mm’s, so that the front and back side patterns overlap with the channel. These patterns are KOH etched until it is stopped by the nitride on the other side of the DSP wafer. Finally, a Pyrex wafer is anodic-bonded to the top side of the DSP wafer, enclosing the channel and the cavities.

The cavity formed by the glass and nitride is used to enclose the probe gas (such as xenon or rubidium). The cavity on the backside is separated from the other cavity by the nitride, but is open on the back side. This nitride membrane is broken, then a glass capillary attached using Torr Seal. The glass capillary is attached to a pump system and a cylinder of the probe gas. The nitride membrane is strong enough to withstand pressures down to at least $10^{-5}$ Torr. A picture is shown in Figure 2 of the device, just before anodic bonding to a Pyrex wafer.

Figure 1, top: Process flow.

Figure 2, bottom: Picture of devices just before anodic bonding.
Prototype Cantilevers for AFM Lateral Force Measurement

CNF Project # 1273-04
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Abstract

One of the major difficulties in calibrating the lateral forces measured with optical lever atomic force microscopy is in determining the lateral optical lever sensitivity. Novel cantilevers have been designed and fabricated to simplify this measurement and pave the way for accurate calibrated cantilevers in which normal and lateral forces (e.g. from friction) can be easily measured.

Lateral (Friction) Force Microscopy

Atomic force microscopy (AFM) is widely used today to image surfaces and measure nanoscale forces. The most common form of AFM relies on a laser beam reflecting off the back of a cantilever to provide feedback on interaction of the cantilever tip with the surface of interest. The two most common deflections measured during surface interaction are normal and torsional, as depicted in Figure 1 (a) and (b), respectively. To accurately convert the observed AFM cantilever deflections to normal force and friction using contact AFM requires both accurate cantilever spring constant (stiffness) calibration for both normal and torsional deformation, as well as the determination of the normal and lateral sensitivity of the optical lever. Calibration of the normal spring constant and normal optical lever sensitivity is relatively straightforward using the reference cantilever technique and suitable SI traceable cantilever artifacts [1]. Calibration of the torsional spring constant and torsional optical lever sensitivity, needed for friction measurement, is more difficult.

Lateral Force Microscope Calibration Using a Modified Cantilever

One of the most straightforward methods of applying a known torsion to a cantilever involves fixing a lever to the cantilever and applying a force to the end of the lever arm as

Figure 1: Normal (a) and torsional (b) deflection of an AFM cantilever.

Figure 2: Cross piece attached to an AFM cantilever.
Mechanical Devices

in the technique of Feiler, et al. [2]. More recently, a refined version of this technique was demonstrated by Reitsma [3] in which a cross piece was glued to a cantilever forming a double lever arm (Figure 2). Application of a series of forces at different lever arm distances were performed to more precisely determine the torsional optical lever sensitivity (by a factor of 5 over Feiler) and demonstrate the principles of the new method.

The device was patterned on silicon-on-insulator wafers using optical lithography and etched using deep reactive ion etching (DRIE). Back side alignment was used to match the handle chip to the front side pattern. Final release of the structure was accomplished with buffered oxide etch (HF).

The microfabricated prototypes are currently being evaluated for their ability to measure the torsional optical lever sensitivity precisely. Torsional spring constants, measured using a calibrated instrumented indenter, will also be compared to Euler-Bernoulli theoretical models based on dimensional measurements and material properties for these experimental cantilevers.

References


Prototype Microfabricated Lateral Force Cantilever

A new cantilever has been designed that incorporates the cross piece into a microfabricated cantilever beam. The front side design pattern, shown in the white light interferometric microscope images in Figure 3, provides three important features. The basic handle die (central image) allows the device to be easily utilized in a commercial AFM. Two cantilevers with different normal and torsional spring constants extend over the front edge of the handle die. The cutout at the bottom of the handle die provides the means to calibrate the optical lever sensitivity using a different die. It incorporates a positioning mark and fiducial marks to aid in the placement of the cantilever during the critical torsional optical lever sensitivity measurement. Pressing the cantilever down on the edge of this feature while lined up with the fiducial marks allows known amounts of torque to be applied to the cantilever. A series of these measurements taken at different lever arm lengths then defines the torsional optical lever sensitivity in a precise way.

Figure 3: Microfabricated cantilever prototype incorporating cross piece.
Nanofountain Probes for the Delivery of Molecular Inks

CNF Project # 1296-04
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Abstract

Nanofountain probes (NFPs) are atomic force microscopy (AFM) probes designed to pattern planar substrates with molecular inks in the 50 nm - 1 µm range [1]. They extend the “dip-pen nanolithography” (DPN) [2] mode of patterning, by increasing the writing speed and eliminating the need for disruptive re-inking and subsequent probe realignment. This is accomplished by supplying fluid ink from on-chip reservoirs through microchannels to the probe tips. A volcano-shaped aperture keeps the fluid ink as close as possible to the probe-substrate contact point, without allowing a real flow over the substrate. This is essential to achieving sub-100 nm lines and avoids the formation of outer menisci (droplets) being dragged over the surface, as is the case with nanopipette and apertured probe writing devices.

Summary of Research

The nanofountain probe (NFP) allows parallel writing of molecular inks with a linear array of 12 probes, fed by two reservoirs [3]. It contains silicon nitride cantilevers with embedded microchannels, linking the on-chip reservoirs with volcano-shaped ink delivery tips (Figures 1, 2). The chip is designed to fit standard AFMs. Its fabrication relies on a combination of bulk and surface micromachining, with the release of the chips performed by deep reactive ion etching (DRIE).

A third generation NFP fabricated at the Cornell NanoScale Science and Technology Facility addressed the limitations experienced by the second generation chips. The use of silicon-on-insulator wafers provided better control of the residual stresses in the cantilevers to reduce bending upon release from the substrate, facilitating the patterning process. This also increased the laser signal detected by the AFM, thereby improving the image quality obtained when scanning. The new design features deeper microchannels to allow the delivery of larger molecules as well as stiffer cantilevers. The stiffness of the 520 µm long cantilevers is now 0.46 N/m; previously they were 0.312 N/m. In addition, more robust fabrication processes were used, increasing the device yield to about 75%.

As with the previous fountain probes, the connectivity and sealing of the NFP’s integrated fluidic system was tested with a DI water-diluted fluorescent dye (Texas Red, Dextran). The results showed a good penetration of the dye along the entire path of the channels, from the reservoir to the tips.
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With the application of positive voltage to the reservoir area of the nanofountain probe, it was possible to deliver proteins. 15 µg/ml anti-BSA IgG from rabbit in 10 mM HEPES buffer was deposited on 16-mercaptopentadecanoic acid (MHA)-coated gold surfaces using the fountain probe. As is the case with nanoparticles, the size of patterned spots increased with increasing tip-substrate contact time.

A variety of cleaning methods were attempted to prolong the use of the nanofountain probes for protein deposition. Heating in air at 500°C for 10 minutes was able to partially remove some protein or salt residue at the probe tips. This technique was more useful for making slight adjustments to the curvature of the cantilevers. Immersing used NFPs in piranha solution (7:3 v/v H₂SO₄:H₂O₂) was the most effective method for removing residues from the tip area.

The new NFPs were used to make reproducible patterns of deoxyribonucleic acid (DNA) at room temperature in a relative humidity range of 20-90% (Figure 3). Thiol-modified oligonucleotides were patterned on a gold substrate and then hybridized to complementary nanoparticle-conjugated DNA strands, thereby confirming the biological activity of the patterned molecules. The successful patterning was verified by dark-field optical, AFM and SEM imaging.

In the interest of demonstrating patterning capabilities not easily achieved with other sub-100 nm patterning techniques, citrate-stabilized Au nanoparticles, with an average diameter of 15 nm, were directly patterned on SiO₂ substrates functionalized with 3-aminopropyltriethoxysilane (APTES) [4]. The negatively charged particles were electrostatically attracted to the positive NH₂ groups of APTES-SiO₂ substrates. The tapping mode AFM image in Figure 4 illustrates an array of dots patterned by the NFP. Topographic imaging of the patterns showed that the nanoparticle spot heights ranged from 10-30 nm depending on the density of the nanoparticles within the spots. This is in good agreement with the 15 nm average diameter of the nanoparticles. Typical patterned spots obtained with a tip-substrate contact time of 2s were 600-700 nm in diameter. As expected, spot sizes increased with increasing tip-substrate contact times. Scanning electron microscopy (SEM) imaging also confirmed the direct delivery of the nanoparticles. Computer simulations of fluid flow through the NFP indicated that the fluid remained confined to the core tip area of the probe, producing high-resolution patterns.

**References**


Air-Coupled Acoustic Method for Testing and Evaluating Micro-Scale Structures

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Abstract

Using an air-coupled transducer to externally excite and a Laser Doppler Vibrometer/Interferometer (LDV) to capture transient displacement waveforms, a technique to determine mechanical properties of micro-scale structural elements is created. The resonance frequencies and mechanical properties (Young’s modulus and stiffness) extracted from the transient displacement waveforms have been compared, with good agreement, to computational and simplified analytical models for commercially available micro-cantilever beams and micro-scale rotational oscillators fabricated for this study at CNF. The technique could serve to diagnose stiction problems of micro-scale structures.

Summary of Research

A non-contact, non-destructive testing and mechanical characterization method based on air-coupled acoustic excitation and transient displacement measurement using a Laser Doppler Interferometer/Vibrometer (LDV) for the determination of mechanical properties of micro-scale structures has been created (Figure 1). Using an air-coupled transducer in conjunction with a LDV in room conditions without contacting the micro-scale structure nor its substrate, a structural element is excited and induced vibrations are measured. Modal parameters (resonance frequencies) can be extracted from the transient displacement waveforms and compared with computational predictions such as a finite element (FE) analysis. As functions of material and geometric properties of the micro-scale structure, the modal properties allow the back-calculation of various mechanical properties, including stiffness and Young’s Modulus, from the measured transient displacement waveforms. The resonance frequencies of micro-scale structures can be excited with air-coupled transducers and transient responses can be acquired optically for the determination of mechanical properties such as Young’s modulus and stiffness.

Analytical and computational resonance frequencies of the micro-cantilever beam (Figure 2) and micro-scale rotational oscillator (Figure 3) oscillator are compared with experimentally determined frequencies with good agreement. An experimentally determined fundamental frequency of the micro-cantilever beam was found to be in good agreement with computational predictions. The resonance frequencies of the micro-cantilever beam were measured to be 1.2 kHz and 2.4 kHz for the fundamental and second harmonic, respectively. These frequencies were compared with computational predictions and found to be in good agreement. The technique could serve to diagnose stiction problems of micro-scale structures.

Figure 1: Photo of experimental set-up for non-contact mechanical testing and characterization of micro-scale structures.

Figure 2: Top view of the nanosensors TL-NCL micro-cantilever beam at 20° magnification. (Transducer not shown).
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A resonance frequency of 187.5 kHz for the micro-cantilever beam was calculated from transient displacement data. This approximation was within the 20% dimensional tolerance allotted by the beam’s manufacturer dimensional specifications. An analytical calculation (from $E = 169$ GPa, $\rho = 2,330$ kg/m$^3$, and the reported geometry) showed 215.8 kHz (within 0.6% of the 217 kHz declared by the manufacturer and within 13.2% of the experimentally determined fundamental resonance frequency). The Young’s modulus of the micro-cantilever beam was found to be 127.5 GPa with a stiffness of 42.4 N/m (assuming the specified dimensions) was experimentally determined. The experimentally determined Young’s modulus (127.5 GPa) is significantly lower than the value provided by the manufacturer (169 GPa); assuming no dimensional variations, this deviation (24.5%) may be attributed to material property alterations such as oxidation of silicon and is unaccounted for in the FE model and/or analytical formulas.

For the micro-scale rotational oscillator, a fundamental in-plane resonance frequency of 240.5 kHz and 242.5 kHz was experimentally determined for different devices, within 10% of the 267.0 kHz and 266.4 kHz calculated by the FE analysis and a simplified analytical model (from $E = 175$ GPa, $\rho = 2,330$ kg/m$^3$, and the device geometry), respectively. The resonance frequency of the first out-of-plane mode was identified at 888.0 kHz and 888.7 kHz (Figure 4) for two samples within 2.5% of the 878.8 kHz and 868.8 kHz calculated by the FE and simplified analytical models, respectively. A Young’s modulus of 182.3 GPa and an out-of-plane stiffness of 306.1 N/m were determined from the measured fundamental resonance frequency. The experimentally calculated Young’s modulus (182.3 GPa) for silicon is within 4.2% of the approximated Young’s modulus (175 GPa) and the experimentally determined out-of-plane stiffness is within 4.2% of the expected 293.9 N/m (based on $E = 175$ GPa, $\rho = 2,330$ kg/m$^3$, and the device geometry).

Young’s modulus, and stiffness of micro-scale structures with known geometries can be effectively determined using air-coupled excitation in conjunction with a LDV. In addition, stiction problems for a particular micro-scale structure through the comparison of substrate and structure responses to the air-coupled acoustic field can be diagnosed. The method seems easily adaptable to various micro-scale structures. The proposed excitation and measurement mechanism features: simple set-up, function at room conditions, non-contact and non-destructive operations, and repeatable and rapid turnaround time for the evaluation of modal parameters and mechanical properties of microscale structures. The results could lead to a practical method for the evaluation of modal and mechanical properties of micro-scale structures using this non-contact and non-destructive technique.

Acknowledgements

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This report is extracted from our paper “Air-coupled acoustic method for testing and evaluation of micro-scale structures” from the journal Review of Scientific Instruments (Volume 78, ID Number 055105, May 2007); a full discussion of our method is available there.
Design and Fabrication of Silicon-Pyrex Micromixers for Enhanced Mixing

CNF Project # 1376-05
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Abstract
Silicon microelectromechanical systems (MEMS) technology was used to successfully fabricate a proposed multichannel micromixer and a standard T-junction micromixer for mixing enhancement study. Packaging via anodic bonding of the structured silicon substrates with Pyrex® was performed to facilitate optical and fluidic access to the channels of the micromixers. This packaging produces the desired triple-stacked (Pyrex/silicon/Pyrex) multi-channel micromixer and double-stacked (Pyrex/silicon) single-channel T-junction micromixer. Serving as a foundation for the fabrication of these mixing devices and the complementary experimental work was our prior numerical study carried out to evaluate, characterize, and optimize different model-based microchannel mixers. Our present work focuses on the testing and characterization of these fabricated devices for the ultimate purpose of designing efficient micro-channel mixers for microchemical systems applications.

Summary
Improvement of mixing quality in microchannel mixers/reactors, most especially for liquid/liquid multiphase reactions, has been recognized as a relevant technical issue critical to the development and application of integrated microchemical processing systems. The ineffective mixing in microchannel mixers/reactors, primarily due to the inherently diffusion-dominated laminar flow that characterizes such small-volume space, has become an issue of significant interest to many investigators working in the field of microreaction engineering.

The goal 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 design of our proposed micromixers for mixing enhancement is based on the mechanisms of fluid multilamination and elongational flow. In the mixing configurations designed, mixing is enhanced by placing static or passive mixing structures on the mixer channel floor to reduce the fluid diffusion path while at the same increasing significantly the fluid contact areas via creation of folding as well as local and global re-orientation of fluid interfaces.

The outcome of our prior numerical study using computational fluid dynamics (CFD) approach [1] shows that the proposed mixing configurations exhibit remarkably better mixing performance when compared with the standard T-junction micromixer. However, for our experimental mixing study, we select the standard T-junction micromixer (TjM) and one of the proposed mixing configurations that we refer to as multilaminated/elongational flow micromixer-4 (MEFM-4) based on the set criteria of minimum pressure drop and high mixing performance.

Figure 1: The back-side of MEFM-4 with the critical etched-through holes.
The completion of the fabrication aspect of our work, excluding packaging, has been reported earlier. Utilizing the silicon MEMS microfabrication technology [2], made available by the state-of-the-art equipment at CNF, the two above-mentioned mixing devices (designated as TjM and MEFM-4) were fabricated from silicon with Pyrex as the cover material. The fabrication process was achieved using silicon bulk micromachining techniques, which involved 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 front and back of the wafers.

It is worth mentioning that a three-step lithographic process is required to ensure structural and perfect back-to-front-alignment during the fabrication of the multi-channel MEFM-4 while a two-step lithographic process is 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. As shown in Figure 1, these 500 µm etched-through holes were designed to aid the transport of one fluid from the back-side inlet manifold to meet and mix with the second fluid coming from the front-side inlet manifold.

The packaging aspect of this work was recently concluded with the following processing steps: drilling of holes through Pyrex; anodic bonding of structured silicon with Pyrex; and dicing of the stacked wafers into individual micromixing chips. For fluidic access into MEFM-4, three holes (two inlet ports and one outlet port) were drilled into one of two Pyrex wafers to be bonded on both sides of the structured silicon wafer using a sonic mill. Anodic bonding was performed using 500 µm thick Pyrex glass wafers to obtain a Pyrex/silicon/Pyrex and Pyrex/silicon stacks for MEFM-4 and TjM respectively. The bonded wafers were then diced into individual micromixers of sizes 3.50 cm x 2.60 cm and 6.45 cm x 2.54 cm for MEFM-4 and TjM respectively (see Figures 2 and 3).

These micro-scale mixing devices, shown in Figures 4, are being tested and characterized for their relative mixing performance in our experimental set-up at New Jersey Center for MicroChemical Systems (NJCMCS) specifically designed for that purpose. In essence, our plan is to validate experimentally our numerical study on mixing enhancement using these fabricated devices.

References

Aqueous Transduction of Poly-SiGe Disk Resonators

CNF Project # 1380-05
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Abstract
This paper demonstrates an electrostatic transducer for lateral contour-mode resonators in which the transduction gaps are filled with a liquid dielectric (water) having much higher permittivity than air ($\kappa_{\text{water}} = 80.1$). Aqueous transduction is more efficient than air-gap transduction (lower motional impedance) and has a larger frequency tuning range than solid-dielectric transduction. We demonstrated a 42 megahertz (MHz) poly-SiGe disk resonator with deionized (DI) water confined to the electrode gaps. The resonator has a measured quality factor ($Q$) of 3,800, motional impedance of 3.9 kΩ, and 3% series frequency tuning range.

Summary
Sounart and others have demonstrated that by using an local oscillator (LO) signal that is faster than the response time of a polar fluid, it is possible to prevent electrode polarization and double-layer formation, enabling electrostatic transduction in liquid media [1, 2]. To determine whether the same approach enhances the performance of contour-mode radio frequency micro-electromechanical systems (RF MEMS) resonators, we used a air-gap poly-SiGe disk resonator ($f_0 = 49.2$ MHz, $Q_{\text{air}} = 5,300$, $R_s = 510$ kΩ @ $V_P = 5V$) (Figure 1) [3] and submerged it in a water droplet. However, immersing the resonator in water caused excessive mass-loading and $Q$ losses resulting from viscous drag. To eliminate mass-loading and viscosity effects of the water droplet on the resonator, we coated the resonators with a hydrophobic self-assembled monolayer (SAM). The SAM is non-conformal, coating the top surface of the poly-SiGe disk resonator while leaving the 60 nm transducer gaps hydrophilic.

We placed a drop of DI water on the resonator and then slowly tipped the chip to one side to let the water droplet roll off the structure. The DI water ‘wicked’ the electrostatic transducer gaps (Figure 2a-b) and resonator performance improved to $Q$ of 430 at 36 MHz due to reduced viscous damping (Figure 2c). This low $Q$ resulted from water remaining underneath the

Figure 1: (a) SEM of poly-SiGe disk resonator showing the resonator, electrodes and fill-pattern, and (b) 2-port measurement setup. The superimposed LO signal prevents bi-layer formation [1] and enables aqueous transduction and (c) transmission response in air (after floor cancellation).

Figure 2: (a) Photograph of a water droplet placed on a resonator and then moved to the edge, (b) Schematic illustrating the experiment. The water wicks the 60 nm gap and is left under the structure. (c) Measured transmission response after the water droplet is moved away from the resonator: $Q$ of 430 at 36 MHz.
resonator after the droplet rolled off. Even though the gap under the disk was < 2 µm, water underneath would cause viscous damping and degrade the quality factor. To reduce this effect, we placed the water droplet a few microns away from the resonator and tipped the chip, letting the water droplet roll over the disk resonator (Figure 3a). The short time that the water droplet overlapped the resonator was enough to wick the transduction gaps but greatly reduced the chance of water seeping under the resonator. We were able to repeat the measurement multiple times with similar results. All measurements gave $Q > 3,500$ and $R_x < 4.2 \, \text{kΩ}$ near 42 MHz (Figure 3b). The 100× improvement in motional impedance from air-gap-to-water is comparable to previous solid-dielectric transduced resonators [4, 5]. The experimental $R_x$ improvement is smaller than the theoretical enhancement (~ 3000×) due to the loss tangent of water, which adds to the total series impedance of the resonator.

References

Mechanical Coupling of 2D Resonator Arrays for MEMS Filter Applications

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Abstract

We present a study of mechanical coupling in two dimensional (2D) resonator arrays for filter applications. A robust coupling design for 2D array filters, comprised of weak coupling in one dimension and strong coupling in the second, is demonstrated experimentally and compared with weakly coupled and electrically summed 2D resonator array filters. Effects of inherent disorder in resonator arrays due to fabrication variations are minimized in this mechanical coupling scheme, averaging over resonator mismatch to form a smooth pass-band. The strongly-coupled 2D filter improves insertion loss and ripple without degradation in filter shape factor or stop-band rejection relative to its 1D counterpart.

Introduction

Though two-pole filters currently dominate radio frequency microelectromechanical systems (RF MEMS) filter research, there is an impetus to extend to multi-pole filters. As shown by Wang et al [1], increasing the number of resonators in a 1D filter improves both pass-band shape factor and stop-band rejection. However, spatial decay in the resonators and fabrication variations result in increased insertion loss and distortion in the passband as more resonators are added to the 1D array. This phenomenon has previously been investigated by Castanier and Pierre [2], using classical perturbation theory to model the effects of both dissipation and variations on 1D filters.

To improve this passband distortion, Judge et al. [3] proposed a 2D coupling which averages out the stochastic resonator characteristics. The design strongly couples an array of identical 1D filters, generating a two-dimensional matrix of resonators which are coupled weakly in one direction and strongly in the other (Figure 1).

Filter Design and Fabrication

In this study, four resonator coupling configurations are investigated to determine the effectiveness of the 2D strongly coupled array filter. We construct a 1D 4-pole filter (Figure 2a) as a basis of comparison for all 2D filters in the study. The performance of this 1D filter is compared with a set of four 1D 4-pole filters, electrically summed in parallel (Figure 2b), a 2D 4 × 4 array of resonators, weakly coupled in both directions (Figure 2c), and a 2D 4 × 4 array of resonators, coupled weakly in one direction and strongly in the other (Figure 2d).

The filters are composed of extensional wine glass ring resonators [4] with a fundamental resonance designed for 500 megahertz (MHz). The resonators are driven and sensed with lateral dielectric transduction [5]. The filters are fabricated in a simple silicon-on-insulator (SOI) process shown in Figure 3. First, 100 nm of low pressure chemical vapor deposition (LPCVD) stoichiometric silicon nitride are deposited on a 3 µm thick n"
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Figure 4b presents the 50 $\Omega$ terminated $S_{21}$ transmission of the four electrically summed 1D 4-pole filters (Figure 2b). The increased transduction area of the electrically summed 1D filters improved the insertion loss (IL, defined here at the maximum peak) by 7 dB relative to the single 1D chain. Additionally, the passband flattened to only 1.1 dB ripple, due to the summation of four 4-pole filters, offset in frequency from one another due to fabrication variations. However, these improvements are at the expense of filter shape factor and stop-band rejection. The electrically summed array filter has a 3 dB-8 dB shape factor of 2.19—a 30% degradation from the single 1D chain shape factor of 1.68. Furthermore, the stop-band rejection of the electrically summed filter reduces to 11.1 dB from 16.7 dB in the case of the single chain.

We next inspect the case of the $4 \times 4$ array of resonators, weakly coupled in both directions, shown in Figure 4c. The behavior of this multi-mode 2D array demonstrates that the strong coupling in the next filter is indeed strong enough relative to the weakly coupled direction.

Finally, we observe the effects of coupling a $4 \times 4$ 2D array of resonators weakly in one direction (defining the resonant modes which contribute to the passband) and strongly in the other direction (averaging out fabrication variations). The unterminated frequency response of this filter is presented in Figure 4d. As in the case of the electrically summed filters, the insertion loss improves due to increased transduction area.

The ripple of the 2D strongly coupled filter improves from 5.4 dB to 4.2 dB relative to the 1D 4-pole filter. This corresponds to a 22% improvement in passband ripple. The improvement in passband distortion does not sacrifice filter stop-band rejection and shape factor. The stop-band rejection increases from 16.7 dB to 17.6 dB while the filter shape factor (3 dB–8 dB) decreases from 1.68 to 1.55 for the 2D strongly coupled filter relative to the 1D filter.

Conclusion

In this work, we demonstrated the effectiveness of a 2D mechanical coupling configuration for filters in reducing passband distortion due to micro-fabrication variations. A 2D filter comprised of a $4 \times 4$ array of bulk-mode wine glass ring resonators was demonstrated at 511 MHz. The 2D coupling provided a 22% improvement in unterminated passband ripple relative to its 1D counterpart, without degradation in stop-band rejection or shape factor. The 2D strong mechanical coupling configuration examined in this work can be implemented with any resonators in any fabrication process, providing more reliable and repeatable high-performance MEMS filters.

References

Directed Fluidic Assembly of Microscale Tiles

CNF Project # 1396-05  
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Abstract

The aim of this project is to direct the assembly of micron-scale units (microtiles) into programmable, reconfigurable structures. Here we describe the fabrication of latching silicon microtiles for directed fluidic assembly. We also describe experiments in which fluid flow through a polydimethylsiloxane (PDMS) fluid chamber is controlled in order to direct the manipulation and assembly of various structures. In addition, we present simulation results which predict the further assembly capabilities of this system.

Summary

In order to manufacture increasingly integrated devices with incompatible component fabrication processes, reliable micro-/nano-assembly techniques are required. As size scales decrease, however, traditional pick-and-place assembly with motion planning becomes increasingly intractable. In response to these impediments, many “bottom-up” strategies have been developed, and have achieved the assembly of periodic, random, or specified target structures using pre-programmed components. In many cases however, structures to be assembled are either not known a priori, or would require too many distinct pre-programmed components to be assembled efficiently. Here we present an alternative approach to assemble arbitrarily-shaped microstructures from regular components on the micro scale. We circumvent the difficulties of pick-and-place microassembly by manipulating the components indirectly in a microfluidic chamber and relying on passive alignment and latching mechanisms to complete the assembly. We anticipate this new microassembly approach will form the basis for an alternative microfabrication paradigm and the manufacture of complex, integrated microsystems.

Experiments

Building on previous fluidic assembly experiments [1, 2], we achieved the automated manipulation and assembly of silicon microtiles. This was achieved by controlling the fluid flow conditions within and assembly chamber by regulating inlet and outlet conditions. As a first step, we introduced a single tile into the chamber and marched it along the substrate from one outlet position to the next. The next fundamental step towards the assembly of complex shapes was to create two- and three-tile assemblies (see Figure 1). In order to achieve this, we essentially rolled the tiles back and forth across the substrate together until the conditions were right for assembly. The resulting assembly rates are compared in Figure 2.
Simulations

In order to demonstrate directed assembly of microtiles, we carried out computational fluid dynamics simulations using the commercial software FLUENT. The results of these simulations, showing the assembly of a ‘C’ structure using square building blocks, are shown in Figure 3. Initially, the microtiles float neutrally buoyant in a chamber. The bottom of the chamber contains valves that can be selectively opened or closed and can be used to manipulate tile motion. When a valve is opened, there is fluid motion in that direction, which in turn causes a tile to move to that location. In this manner, tiles can be arbitrarily assembled at the bottom of the chamber, and the assembled tiles can be released back into the chamber by reversing flow at the substrate.

Fabrication

Solid 500 × 500 × 30 µm silicon tiles with patterned sides for self-alignment and passive latches for assembly (Figure 4) were designed using the L-Edit CAD software. Masks were created using the Heidelberg DWL 66 laser pattern generator. The tile patterns where then transferred to Shipley SPR 220-7.0 negative photoresist spun onto a silicon-on-insulator (SOI) wafer using the EV 620 contact aligner. After developing the resist, the exposed wafer was etched through the 30 µm device layer to expose the insulator using the Unaxis 770 Bosch etcher. A 49% HF solution bath was then used to release the etched tiles which were collected in a filter.

The directed assembly microfluidic chamber with on-chip valving was fabricated by multilayer PDMS soft lithography [3]. Soft lithography moulds were created by spinning SU-8 2050 photoresist on silicon wafers at 4000 rpm to obtain a ~ 40 µm thickness film which was patterned with contact alignment and hard baked for durability. Sylgard 184 silicone elastomer base and curing agent were used in a ratio of 20:1 for the thin fluidic layer and 5:1 for the thick pneumatic layer. The fluid layer was spun over its mould at 1250 rpm for a thickness of ~ 60 µm, while the pneumatic layer was poured approximately 1 cm thick. Both were cured at 80°C for one hour, then bonded together (after punching the pneumatic layer ports), and cured for several hours at 80°C. The fluid layer ports were then punched and the entire chamber was cleaned in an oxygen plasma and bonded to a glass slide.

Conclusion

We have presented a novel microfabrication approach which allows the assembly of non-regular microstructures from regular components on the microscale. Experiments have demonstrated the effectiveness of this technique in the assembly of two- and three- component structures. Simulations have further demonstrated the ability of this approach to assemble larger, non-regular structures. Since our assembly mechanism does not rely on unusual properties of the materials used, a wide range of component materials and assembly fluids is possible, along with a range of components sizes. All of this leads to a new microassembly approach for the manufacture of new, integrated Microsystems.

References


Development of Nano Electromechanical Devices for Quantum Behavior Studies

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Abstract

Cooling mechanical resonators is currently a popular topic in many fields of physics including ultra high precision measurement and the study of transition between classical and quantum behavior of mechanical systems. The goal of this project is to fabricate nanomechanical devices coupled to a cavity Quantum ElectroDynamic (cQED) system. The photons from the cavity interact with and cool down the mechanical devices to their quantum ground state.

Summary

In this project we attempt to cool a mechanical device to its ground state using back action from a microwave cavity. We have designed and fabricated nanomechanical-resonator (NR) doubly clamped beams for magnetomotive measurement. The NR beams, fabricated on a 50 nm low stress low pressure chemical vapor deposition (LPCVD) silicon nitride layer, are written by electron beam lithography and are 50 nm wide and separated from a gate electrode by < 100 nm. The beams are released by two consecutive reactive ion etch (RIE) processes, one to vertically etch the nitride layer and the second to isotropically remove the silicon underneath the beams.

In the next phase of the project (work in progress), we will couple the NR beams to a microwave cavity formed from a 2.4 cm long coplanar waveguide. The resulting NR-cQED system will be installed on a 7 mK dilution refrigerator to cool the NR-beams to their quantum ground state. We will investigate ground-state squeezing of these mechanical beams to below the Heisenberg limit on continuous position detection.

References

Figure 1: SEM image of 150 nm wide beams.

Figure 2: SEM image of a suspended 50 nm wide beam.
Feasibility Study on the Microfabrication of MEMS Integrated Antenna

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Abstract

An annular slot antenna has been constructed on microwave laminate. Two radio frequency microelectromechanical system (RF MEMS) switches are placed across the outer slot to connect/disconnect the ground and the inner strip. One switch is placed along the feeding microstrip line on the back side of the antenna and it allows the selectivity to feed either slot. When no switches are activated, the outer slot is fed to radiate at 2.4 gigahertz (GHz); when all the three switches are activated, the inner one is fed to radiate at 5.3 GHz. The frequency selection is realized by controlling the on/off states of the RF MEMS switches.

Summary of Research

Next-generation wireless communications in both commercial and military applications are increasingly moving to small, light-weight, and high-frequency systems with increased functionality and reduced power consumption. A concurrent trend in communication is to develop multi-mode/band systems on a single platform. These communication systems are becoming ever more complex with dozens of standards, a mixture of analog and digital formats and a diversity of spectral utilization. In many cases, these communication systems need to be supported by a number of antennas, which often radiate different frequency bands, polarizations and radiation patterns.

For a multi-mode/band system on a single platform, if one antenna is devoted to each mode/band operation, the limited space quickly becomes littered with antennas, and the use of dedicated antennas is costly, leads to unwanted proximity-coupling and degrades the overall performance. One solution, which keeps the entire system compact and satisfies multi-mode/band operations, is to design a single antenna that can be reconfigured or tuned to deliver the desired frequency bands, polarizations and radiation patterns without sacrificing radiation efficiency.

The radiation behavior of an antenna depends on the distribution (path and electrical length) of the resonant currents over the antenna surface, which is further determined by antenna geometrical parameters, such as size, shape and position of radiating and parasitic elements. Reconfiguring these parameters enables multiple functions to be performed with the same antenna structure. A number of approaches have been proposed for implementing reconfigurable antennas [1-4]. Most of these approaches make use of switches or varactors to change the physical dimensions of an antenna structure or the distribution of the antenna resonant currents.

Reconfiguring antennas can be realized by using semiconductor devices (PINs and FETs) and RF MEMS switches. For semiconductor devices, when the signal frequency becomes greater than 1 GHz, they have large insertion losses and poor isolation. They produce
significant intermodulation distortion as a consequence of their non-linear I-V feature. The antennas made by using these devices will be characteristic of high power consumption. RF MEMS switches are devices that use mechanical movement to achieve an on/off operation. They have very high isolation, very low insertion loss and very high linearity. The RF MEMS switches based on electrostatic actuation have near-zero power consumption. Being essentially broadband devices, RF MEMS switches are far less affected by frequency and are more suitable for high-frequency applications. Most importantly, the MEMS technology enables monolithic integration with antenna radiating elements. RF MEMS switches are now increasingly used to realize reconfigurable or tunable RF devices, components and systems. As RF MEMS switches are becoming mature, it is important to develop novel ideas and practical approaches to make full use of MEMS switches’ remarkable characteristics and to achieve novel, high-performance RF devices, components and systems.

 constructed on a TMM10i ($\varepsilon_r = 9.8, \tan\delta = 0.002$) Rogers microwave laminate. Two switches are placed across the outer slot to connect/disconnect the main ground and the inner metal stripe. Figure 2 shows the double-arm DC contact RF MEMS switch used in this implementation. The dual band behavior is achieved by selectively activating the two concentric radiating slots. One RF MEMS switch is placed along the feeding microstrip line on the back side of the antenna and it allows the selectivity to feed either slot.

In the previous year, we worked on a reconfigurable annular slot antenna. The antenna is designed to primarily realize frequency band selectivity while maintaining radiation pattern. Slot antennas are attractive for many communication and radar applications due to geometric simplicity, efficiency, reliability, and light-weight. It is known that an antenna alters its radiation characteristics when its physical geometry is changed. The change here is realized by RF MEMS switches that are located at the annular slots and along the signal-feeding microstrip line. Figure 1 shows the front side of the annular slot antenna.

When no switches are activated, the outer slot is fed to radiate at a low frequency of 2.4 GHz; when all the three switches are activated, the inner one is fed to radiate at a high frequency of 5.3 GHz. Figures 3 shows the simulated and measured return losses corresponding to the two operation modes, respectively. As shown, the frequency selection is realized by controlling the on/off states of the RF MEMS switches. The measured frequency locations are in good agreement with the simulated results with a very small discrepancy at 5.3 GHz.

References
Fabrication of Ultra-Sharp Diamond AFM Probes

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Abstract

Monolithic, ultra-sharp (R < 10 nm) diamond probes were fabricated from ultra-nanocrystalline diamond (UNCD®). The fabrication sequence developed uses optimized diamond processing with precision lithography, selective etching, anisotropic etching, and precision dicing. The probes showed low wear and stiction as expected, based on the physical properties of diamond, outperforming standard silicon nitride probes in these aspects. These all-diamond probes should open up new avenues of research to explore nanoscale friction and adhesion at the nanoscale, as well as nanofabrication.

Summary of Research

Ultra-sharp atomic force microscopy (AFM) probes with diamond tips are long sought after, but so far, not yet commercially available devices. The roughness of the diamond films and several integration difficulties accounted for this. Our ultrananocrystalline diamond (UNCD®) has been proven [1] to have the properties that make diamond AFM probes desirable: high hardness, low wear, and low stiction to particles, while having as low as 7 nm roughness (Rₐ), low differential stress, and good conformity. This material and its growth process are well-suited for fabricating tips. All-diamond probes are superior to coated silicon tips in terms of sharpness, since in the latter, the tip radius is given by the thickness of the diamond film. A preliminary optimization study showed that the probe fabrication process can be used to obtain tip radii as sharp as 5 nm (Figure 1), on the same order as the best silicon nitride probes on the market. Unfortunately, the as-fabricated tips face the silicon wafer, which makes it necessary to reverse them by attaching/transferring them to another substrate. While this process is standard for silicon nitride tip fabrication [2], difficulties arise when using diamond. The probe fabrication sequence we engineered was optimized for convenient etching of diamond, efficient transfer of the diamond layer and probes by bonding to a Pyrex® wafer, and final release of the structures. Ultra-sharp diamond AFM probes fabricated using this process can be seen in Figure 2.

Probes were produced both for tapping mode imaging (higher resonance frequency and stiffness) and for contact mode techniques (low stiffness, low resonance frequency). The characteristics of the probes are given in Table 1. The layout provided each carrier chip with four probes, two on each side of the chip, for maximization of the number of useful probes for testing.

Figure 1: Ultra-sharp UNCD® tip. Tip radius ~ 5 nm.
The probes were tested in standard AFM equipment (Veeco, DI MultiMode). Wear tests were performed [3] in conjunction with high resolution scanning electron microscopy (SEM) and tunneling electron microscopy (TEM) investigation of the tips, showing a net superior behavior of the diamond tips as compared with standard silicon nitride tips. The tests consisted in scanning/imaging for 100 times a 1 mm × 1 mm area of a UNCD surface with the diamond probes in contact mode with zero net force. While standard silicon nitride probes increased their contact radius from 12 to 75 nm in the given conditions, UNCD probes showed no radius increase outside of typical data scatter (± 5 nm).

Batch processing of ultra-sharp UNCD probes allows for developing new or enhanced scanning probe techniques by integration in complex probe MEMS structures, such as arrays of probes (for parallel imaging [4], data storage [5], parallel dip-pen lithography [6], etc.) or high cost specialized probes such as nanofountain-probes [7] and probes with integrated actuation and sensing [8], making such probes affordable by compensating costs with enhanced life time. Conducting UNCD can also be used to develop probes that use electrochemical contrast that have unparalleled dimensional stability as well as for scanning spreading resistance microscopy [1].

Probes will be sent out to users for beta testing. Interested users are encouraged to contact the authors for receiving evaluation samples.

Work was supported by the National Science Foundation through its SBIR/STTR program and the State of Illinois’ Department of Commerce and Economic Opportunity, and acknowledges fabrication of prototypes in part at the Cornell NanoScale Facility, a member of the National Nanotechnology Infrastructure Network, which is supported by the National Science Foundation (Grant ECS 03-35765).

References

Table 1: Characteristics of the produced probes.
Microfabrication of Flexible Sensing Arrays: Active Nanoparticle Thin Films and Interdigitated Microelectrodes on Plastic

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Abstract

Traditionally most of the electronics fabrication work at CNF is based on rigid silicon and glass substrates. The goal of the Center of Advanced Microelectronic Manufacturing (CAMM) is to develop tooling and processes to use unsupported flexible polymeric substrates. Recently, C. J. Zhong [1] and his group have fabricated gold microelectrode devices on glass substrates to explore the use of thin film coatings of monolayer-capped nanoparticles for chemical sensors (volatile organic compound sensing), medical diagnostics and other microelectronic applications. Here we attempt to fabricate copper microelectrode devices using unsupported poly(ethyleneterephthalate) (PET) substrates for use in low-cost, disposable sensor applications.

Summary of Research

Most of the fabrication processes such as photolithography, etching, and stripping for building the copper interdigitated microelectrodes on PET substrates were performed at CNF. Pieces of 125 µm (5 mil) thick heat stabilized PET polyester (DuPont-Tejin Films Melinex ST507) film was isopropyl alcohol (IPA) and oxygen plasma cleaned followed by sputtering of 50 Å chrome and 3000 Å copper at Endicott Interconnect Technologies (EI). Four inch diameter “mock wafers” were cut from the Cu sputtered pieces of PET. Shipley 1813 photoresist was spin-coated at CNF to a thickness of 1.5 µm. The photolithography mask was designed and fabricated at CNF.

The CNF HTG System III-HR contact aligner with 365 nm wavelength UV light was used to image the microelectrode pattern on the resist coated PET substrate. Four seconds was determined to be the optimal exposure time. The resist was developed using AZ 300 MIF for one minute at room temperature. The line/space (in microns) feature size of the microelectrodes include: 5/5, 10/10, 10/15, and 15/15 µm. Figures 1 and 2 show 15/15 µm and 5/5 µm line/space features in the resist after developing. The CE-200 etchant used at CNF significantly over etched the features. Cupric chloride (concentration [175g/L]) was also used to etch the copper layer. The cupric etching process was done at EI, and over etching was reduced.

After the etching, the AZ-300T at CNF was used to strip the resist at room temperature. Figures 3 and 4 show the resulting flexible
copper microelectrodes on the PET substrate. Figure 3 shows the four inch flexible polyester “mock-wafer” with 1.5 mm Cu circuit lines. These large circuit features connect to the 15 µm sized (over etched) circuit lines shown at high magnification in Figure 4.

This research demonstrates the feasibility of fabricating the flexible copper microelectrodes on flexible unsupported PET substrates using the microfabrication facilities at CNF. Parameters such as etchant concentration, etching time and temperature need to be further improved to prevent overetching the copper layer. PET substrate surface flatness will be improved by use of tooling and a more carefully controlled Cu sputtering process.

The CNF provides education and training for the CAMM students as well as facilities to design and fabricate masks, test various materials, processes and tooling as the CAMM is being established. The long term objective of this project is to develop roll-to-roll microelectronics manufacturing technologies to fabricate large-area flexible chemical sensing arrays at the CAMM. The CAMM was established in 2005 when the United States Display Consortium selected Binghamton University, a global leader in electronics packaging and small scale systems integration, to spearhead development of next generation roll-to-roll electronics manufacturing capabilities [2]. A unique collaborative effort, the CAMM brings together Endicott Interconnect Technologies, Inc., Cornell University and other partners from government, industry and academia to tackle the myriad challenges of this emergent electronics manufacturing technology.

References


Microfluidic Devices for Insect MEMS

CNF Project # 1516-06
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Abstract
This work describes the development of an electroactive microwell based chemical/drug delivery system and its pupae stage implantation within Manduca Sexta moths. This forms one of the critical components in our development of Insect MEMS which aim to fuse nanodevice technology with living systems. The goal of this system is to provide “on-command” chemically induced immobilization and subsequent reanimation of the insect. In this talk we will present the results of our adult survivability data for late and early pupae stage microdevice insertion experiments, our initial toxin injection experiments, and a comprehensive numerical/experimental study of our electroactive drug delivery system.

Introduction
Modern microsystems technology has enabled the development of a large number of biomedical devices which allow us to monitor biological systems and biomolecular events with extreme precision. While such technology is proving extremely successful, rarely has the extension been made to exerting active control over a living system. In this work we present our initial results on the development of implanted microfluidic devices which enable such control over flying insects, namely Manduca Sexta Moths. One potential application of such a hybrid-insect system is in the development of microscale air vehicles, which exploit the highly evolved aerodynamics of insects with recent advances in microdevice engineering. An overview illustrating the system integration is presented in Figure 1.

Experimental Results
As shown in Figure 2, the microdevice platform is partially implanted within the insect at the dorsal thorax of the pupae. To find the optimal location (which minimizes its physiological footprint, maximizes survivability, and favorably disperses the toxin), both the platform size and implantation location were varied. Figure 1 shows the results of a successful emergence of an insect with fully developed wings.

Figure 3 illustrates an abbreviated version of the steps involved in our injection experiments used to determine the dosage effects on the degree and length of insect paralysis. For example, injection of 5 µL of a 5.839M
solution of L-Glutamic acid potassium salt monohydrate, which comprises one of the major components of spider venom, into the thorax, the insect was immobilized for approximately 5 hrs, after which the insect regained its pre-injection activity level. The electroactive microwell drug delivery system developed here is based on the fusion of previous implantable drug delivery technologies with our recently developed electrokinetically active microwells.

Briefly, the drug is stored within a well, etched into a silicon substrate (similar to illustrated in Figure 1b) which is sealed from the exterior using a thin gold membrane on PDMS (polydimethylsiloxane). When a dosage command is issued, a voltage is applied to electrochemically dissolve the gold membrane, exposing the toxin. Unlike previous approaches, which rely on diffusive transport of the drug, we incorporate an electrokinetic injector whereby a potential field is applied between the top and bottom of the well and the drug is electroosmotically rejected from the well. A detailed, finite element based, model of the local fluid and transport dynamics involved in the drug ejection procedure will be presented along with our initial experimental results (see Figure 4). As will be demonstrated, this approach reduces the drug injection time from approximately 30 minutes for diffusive transport to less than 1s.
Capacitive Drive and Detection of MEMS Resonant Motion: Electrical Integration of MEMS

CNF Project # 1520-07

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Abstract

Incorporation of microelectromechanical system (MEMS) oscillators into devices has been limited by the lack of an easily-integrated motional detection scheme. A capacitive detection scheme enabled by impedance matching techniques has been demonstrated for nanowires. However, no measurement had been demonstrated in micron-scale oscillators of interest in sensor and electrical filter applications. Through our work at the CNF, we have demonstrated entirely electrical drive and detection of micron-scale dome oscillators.

Summary

An easily-integrated readout mechanism would enable MEMS/NEMS oscillators to be incorporated to electrical devices, whose applications could include electrical filtering, ultra-sensitive inertial mass sensing [1], and any mechanical operations requiring electrical feedback. A purely electrical readout scheme, in place of the effective-but-bulky interferometric or magnetomotive techniques commonly employed, naturally lends itself to device integration. We are developing a technique that detects the fluctuations in capacitance between the oscillating MEMS/NEMS structure and a charged gate in its vicinity, as demonstrated for nanowires [2]. The tiny resulting signals can be effectively isolated and amplified through the use of an impedance matching circuit to match the oscillator to conventional 50Ω-electronics (Figure 1). Ultimately this approach could be integrated into complementary metal oxide semiconductor (CMOS) processes and devices, allowing far wider application to MEMS/NEMS technology.

The electrical readout technique is demonstrated using concave polysilicon domes. The single crystal silicon substrate and upper polysilicon device layer are insulated by a layer of thermal oxide. The oxide is removed by buffered oxide etch through photolithographically defined etch holes opened in the polysilicon in a reactive ion etch, resulting in a radially symmetric drum, dome, or bowl, depending on film stresses, whose upper and lower surfaces represent the plates of a capacitor (Figure 2). The dome oscillator and a neighboring gold bond pad are surrounded by a...
Mechanical Devices

Trench through the polysilicon into the underlying oxide, electrically insulating the structure from the rest of the wafer and limiting the gate capacitance. Resulting devices are wire bonded to a ceramic DIP and connected to a printed circuit board containing the impedance matching circuitry. The ensemble is placed in vacuum for measurement.

Readout of MEMS/NEMS oscillators examines the vibratory modes of the structure. We are able to identify identical modes with both laser interferometric and an entirely electrical readout (Figure 3 & 4). The electrical readout analyzes a reflected RF signal from the device driven by a network analyzer. At resonance, the fluctuations of the oscillator create an effective impedance, upsetting the impedance match. The electrical signal can be used to drive of the device as well as detect the motion of the oscillator.

References


Figure 3, top: Mechanical resonance peak of dome oscillator, driven and detected optically.

Figure 4, bottom: The same resonance peak, driven and detected with fully electric readout technique.
Arrays of MEMS Intertial Sensors with Optical Readout

CNF Project # 1538-07
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Abstract

Microelectromechanical system (MEMS)-based inertial sensors have many advantages over traditional discrete-component-based inertial sensor designs. This is due to their smaller size, batch fabrication, lower cost and higher robustness. Although MEMS-based inertial sensors can withstand high-g accelerations, they are prone to bias instability. This limits their current use to less demanding applications [1]. The goal of our project was to explore alternative technological approaches to MEMS-based gyroscopes, with potential of achieving the navigation grade performance.

Summary of Research

We have focused on devices that consist of double-tine resonators arranged into a tuning fork shape. Each device features two driving tines and two sensing tines (Figure 1). The driving (lower) tines are to be actuated optically with an amplitude of up to several micrometers. The Coriolis force is exerted on the upper tines when the system is being rotated around the main axis. This force can be detected by observing oscillation of the sensing tines, hence detecting the rotation of the system. The deflection is probed using the optical lever readout similar to the one used in atomic force microscopes [2] and which can detect sub-angstrom displacements.

We have fabricated several batches of devices with different geometries, all featuring single-crystal silicon (Si) as a structural material for the driving tines. Single-crystal Si was chosen as it is characterized by low mechanical losses. Sensing tines were fabricated using either Si (a device layer in SOI wafers) or low stress LPCVD silicon nitride (SiNₓ) as a structural material. In order to fabricate our targeted devices, we have implemented a three-mask process flow that relies entirely on dry etching. The driving tines and the Si sensing tines were patterned using the deep reactive ion etching (DRIE, Bosch process), while SiNₓ sensing tines were patterned using the regular fluorine-based RIE.

Our preliminary characterization of the fabricated devices indicated resonant frequencies of driving and sensing resonators in the ranges of 8-25 kHz and 2-4 kHz, respectively. The resonators in vacuum exhibited typical $Q$-factors above $10^4$.

References

Figure 1: Photograph of one of the structures. Driving and sensing tines oscillate in perpendicular planes.

Figure 2: Array of inertial sensors.
**Fabrication of Nanomechanical Resonators Using a Sequential Exposure of Opposite Tone Electron Beam Resists**

CNF Project # STAFF  
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**Abstract**

We report a method for fabricating nanomechanical structures from a hydrogen silsesquioxane (HSQ)-based negative tone electron beam lithography (EBL) resist. This technique utilizes a sequential exposure of poly(methyl methacrylate) (PMMA) and HSQ to produce self supporting nanomechanical resonators. The first exposure is performed in the PMMA layer. This pattern is developed, HSQ is applied to the substrate over the PMMA, and a second exposure is performed registered to the first. The patterned PMMA film serves as a structural template for patterns written in the HSQ layer. HSQ exposed in patterned regions of the PMMA film becomes mechanically connected to the substrate. HSQ patterns connected to the substrate at multiple points can be released by removing the PMMA film using an oxygen plasma. Nanomechanical oscillators of different lengths were produced using this technique and the resonant frequency spectrum of these structures was examined using an optical excitation and detection technique. Resonant frequencies from 6 to 24 megahertz (MHz) with quality (Q) factors between 300 and 1500 were measured and found to be a strong function of oscillator length. The dynamic response of the system was modeled using the Euler-Bernoulli formalism and found to agree with the experimentally measured results.

**Introduction**

In the past decade, research on nanomechanical structures [1,2] has illuminated fundamental physical phenomena, enabled the fabrication of mass sensors with unprecedented sensitivity, and yielded a host of other unique structures. In 2001, Tanenbaum, et al., presented a simple process for forming nanomechanical resonators composed entirely of a hydrogen silsesquioxane (HSQ)-based negative tone electron beam lithography (EBL) resist [3]. These structures were created by performing a double exposure of the HSQ layer using two patterns exposed at different beam energies: 30 keV and 1 keV. The 1 keV electrons do not penetrate the entire HSQ film; they are confined to the near surface region. In contrast, 30 keV electrons completely penetrate the HSQ film and enter the substrate. Tanenbaum capitalized on this difference in beam penetration depths and used the 1 keV beam to expose patterns of nanoscale resonators. Portions of the pattern were selectively re-exposed using a 30 keV beam. During the HSQ develop process, the regions patterned at 30 keV remained rigidly fixed to the substrate while the patterns exposed at the lower energy were undercut by the developer releasing them.

Like Tanenbaum’s work, this process uses a double exposure. However, the first exposure is performed in a layer of poly(methyl methacrylate) (PMMA). Once this pattern is developed, HSQ is applied to the substrate over
the PMMA and a second exposure is performed registered to the first. The PMMA film acts as a structural template for patterns written in the HSQ layer. HSQ that is exposed in patterned regions of the PMMA film becomes “anchored” to the substrate forming a robust mechanical connection. HSQ patterns connecting multiple “anchor” points can be released by removing the PMMA creating self supporting structures.

Summary

An overview of the fabrication process is shown in Figure 1. 100 mm diameter Si (100) n-type wafers were used as the substrates for this work. An array of alignment mark structures was patterned onto these wafers using standard photolithography and dry etching techniques. These patterns consisted of an array of 10 µm wide squares etched 500 nm into the Si substrate. 500 nm of PMMA was spin-coated onto the wafers and baked at 170°C for 15 min (Figure 1-a). The substrates were exposed using a Leica VB6HR operated at 100 keV with a 5 nA beam current. The locations of the anchor points were exposed in the PMMA film using a dose of 1000 µC/cm$^2$. These patterns were developed in a solution of methyl isobutyl ketone (MIBK) and isopropyl alcohol, 1:3 (Figure 1-b). After inspecting the developed patterns, the substrates were coated in 4% HSQ in MIBK (Dow Corning, XR1541). This film has a nominal thickness of 120 nm when coated at 3000 rpm. The HSQ was applied to the PMMA using a static dispense process and allowed to puddle for 3 seconds before spinning. This puddle process facilitated coverage of the PMMA topography. Early experiments revealed that features in the PMMA layer less than or equal to 200 nm were almost completely planarized using this approach (Figure 1-c). After aligning to the predefined registration mark patterns, the locations of the anchor points were re-exposed. Patterns for structures that would ultimately become released or undercut were exposed such that they were connected to the anchor points. A dose of 2200 µC/cm$^2$ was used for these exposures. The HSQ film was developed using a commercially available 0.26 M aqueous solution of TMAH (AZ, 300 MIF). A 4 min immersion develop with no agitation followed by a deionized water rinse was used for this process (Figure 1-d). Following inspection of the developed patterns, the PMMA film was removed from the substrates using isotropic O$_2$ plasma etching (Figure 1-e).

An oblique angle scanning electron microscope (SEM) image of a fully released bridge is shown in Figure 2-a. Optionally, completed structures could be coated with a layer of Al using electron beam evaporation (Figure 1-f). Beyond changing the optical properties of the released structures, this coating provides a way to produce electrical interconnects and probing pads that are connected to the released nanomechanical structures and isolated from the substrate. An example of a completed Al-coated HSQ nanomechanical structure fabricated using this process is shown in Figure 2-b. Figure 3 shows nanomechanical bug-like structures that illustrate the compressive stress state residing within the HSQ layer.

References

Electrospun Light-Emitting Nanofibers

CNF Project # 762-99, 775-99
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Abstract
We have fabricated sub-wavelength light sources based on electrospun nanofibers deposited on microfabricated interdigitated electrodes. The nanofibers contained transition metal complexes embedded in a polymer carrier which emitted light when a bias was applied across the electrodes. The fibers showed light emission at low operating voltages, with turn-on voltages approaching the band gap limit of the organic semiconductor. Because of the fiber diameter and the operation mechanism of the transition metal complex, emission from light-emitting nanofibers was confined to sub-wavelength dimensions, an attractive feature for sensing applications and lab-on-a-chip integration, where highly localized excitation of molecules is required.

Summary of Research
Micropatterned interdigitated gold electrodes were fabricated on silicon oxide surfaces by patterning a photoresist layer, followed by gold evaporation and lift-off. Nanofibers were then deposited on the micropatterned interdigitated electrodes by electrospinning a solution containing a mixture of ruthenium(II)tris(bipyridine)/polyethylene oxide ([Ru(bpy)₃]²⁺(PF₆⁻)/PEO) in acetonitrile. Electro-spinning is a well developed electro-hydrodynamic method used to produce micro- and nanofibers from a variety of dissolved materials without the need of expensive fabrication methods [1-3]. In our electrospinning setup, high voltage was applied between a droplet of the solution that rested on a sharp conducting tip and a grounded substrate (Figure 1). As a result of molecular ionization and charge redistribution, a jet of the solution was extracted, accelerated by the electric field, and collected on the substrate [4]. The volatile solvent used for the electrospinning solution evaporated in flight, yielding solid fibers on the substrate containing the micropatterned electrodes.

The nanofibers were lit up by applying a dc bias across an array of interdigitated electrodes in a nitrogen environment. Because each fiber could span more than one inter-electrode gap, a single fiber could produce more than one emission zone (although in practice not all of them did, Figure 2). The size of the emission zone for a particular fiber segment spanning an inter-electrode gap is determined in the axial dimension by the operation mechanism of the transition metal complex, and in the transverse dimensions by the fiber cross-section. We imaged the lit up fibers through an inverted microscope and captured the images with a CCD camera. Figure 3 shows the highly-confined electroluminescence from an 800 nm diameter fiber lit at 4 V. Image analysis of the intensity of the emitted light revealed an emission area with dimensions of 240 nm along the axis and 325 nm in the transverse dimension. In reality,
this emission could be even smaller, as the resolution of the measurement is restricted by the diffraction limit of optical microscopy.

In all experiments performed, the current passing through the device and the lumiance from the fibers was monitored as the voltage was ramped up/down. While the current monitored was the total current flowing through all the fibers in each device, only a few lit junctions could be imaged in each experiment. Thus, the lumiance measured in each experiment was that of a section of an array of emitters from a single fiber. Multiple measurements from different fibers were performed and averaged to obtain the characteristic behavior for the device as a whole. In this way, lumiance versus voltage and current versus voltage plots were generated (Figure 4). The turn-on voltage for the nanofibers on ramp-up was 3.2 V, with a subsequent exponential increase of the lumiance at higher voltages. Interestingly, the current showed a two-step ramping behavior, which corresponds to monopolar and bipolar injection regimes [5]. Upon application of the voltage, PF$_6^-$ ions redistribute, leading to the onset of monopolar injection at 1.7 V. As the bias is increased, the monopolar current continues to increase in an exponential fashion until sufficient PF$_6^-$ motion has occurred to establish the second carrier injection at 3.2 V, initiating light emission.

In summary, this research shows that light-emitting nanofibers made of [Ru(bpy)$_3$]PF$_6$/PEO mixtures can be easily produced via the electrospinning method. The fibers were successfully lit on devices containing interdigitated electrodes with 500 nm gaps. Light emission was readily detectable with a CCD camera at voltages as low as 3.2 V and visible to the naked eye at 4 V, approaching the band gap limit for the organic semiconductor [6]. Emission from the fibers was found to be highly confined, with imaged emission areas small enough to be diffraction limited.

References

Optically Decoupled Dual-Cavity VCSEL-Modulator: Materials, Components and Integration Technologies for Optical Interconnects

CNF Project # 780-99
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Abstract

The project is focused primarily on materials, technologies and components for the III-V optoelectronic devices integrated with silicon platform. 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 2006/07, we have proposed and demonstrated the principle of optical decoupling in a loss-modulated VCSEL using intracavity electroabsorption modulator [1] and detuned dual-cavity device [2]. The proposed optical scheme allows feedback-free transmission modulation of the output since the reflectivity is kept constant. Through this modulation method, we have demonstrated a flat (± 3 db) response exceeding 20 GHz [2].

Summary of Research

Fabry-Pérot cavity with electro-absorption modulator (Figure 1) is used as a top mirror for VCSEL and spectrally detuned from the active region cavity so that lasing wavelength coincides with one of the cross-over spots at which reflectivity is almost independent of absorption value. A fabricated device with 8 micrometer optical aperture is shown in Figure 2. The proposed approach allows the VCSEL to be modulated by changing transmission T of the Fabry-Pérot reflector without changing the photon density N in the active region. Initial spectral alignment is controlled through growth (Figure 3), but applied bias at the multiple quantum well (MQW) modulator section allows adjustment of detuning between cavities by changing the top cavity resonance wavelength. The high frequency modulation characteristics can be tuned in this manner to show little or no sign of resonance (Figure 4), in which case the high frequency roll-off of the modulation response is entirely determined by parasitics of the modulator section. If the detuning of the cavities is not optimal, the relaxation resonance appears in the modulation characteristics due to feedback-associated variation of photon density ΔN.

References

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Figure 1: Schematic diagram of high frequency VCSEL-Modulator emitter.

Figure 2: Top view of the fabricated dual-cavity VCSEL-Modulator.

Figure 3: Focused ion beam cross-section of the dual-cavity VCSEL-Modulator.

Figure 4: High frequency response of the device as a function of modulator DC bias.
Preparation of Silicon-Based Photonic Materials

CNF Project # 810-99
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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. Additionally, we have fabricated silicon waveguides in SOI in order to investigate the dispersion and nonlinear optical properties of such waveguides. To achieve these goals, various thin-film deposition, etching, annealing, and lithographic processes were performed at CNF. Equipment used in this project includes the MOS dry oxide and nitride LPCVD furnace, 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, GCA PG3600F optical pattern generator, CVC 4500 E-gun evaporation system, PlasmaTherm 720 aluminum etcher, GSI PECVD nitride deposition, FilMetrics Film Measurement systems, as well as CAD tools and the wet chemistry hoods.

2D Photonic Crystal Resonator for Electro-Optical Switching

By carefully tuning the local lattice constant in the center region of a 2D photonic crystal, we have made a resonator with the measured $Q$ factor of the order of 10,000. If we can change the refractive index of part of the materials—for example, fill the air holes with EO materials like liquid crystal or EO polymer—and change its refractive index by thermal effect or electric field, it is possible to have very high on/off extinction ratio transmission signal for some particular wavelengths. Figure 1 shows an SEM image of the device. A second type of 2D photonic crystal resonator was also made, this time with aluminum electrodes fabricated near the resonator [1]. By filling the photonic crystal with liquid crystals, we were able to red- or blue-shift the resonance by applying an electric field across the electrodes, as seen in Figure 2 [2].

Figure 1: SEM image of the 2D photonic crystal resonator with $Q$ value on the order of 10,000.

Figure 2: Graph showing the redshift and blueshift of the resonance upon the application of a voltage to the liquid-crystal-containing photonic crystal resonator.
Group Velocity Tailoring for Si Waveguides and Optical Soliton

The tight mode confinement in SOI waveguides helps introduce significant waveguide dispersion and thus allows one to obtain a dramatically large anomalous GVD by designing the SOI waveguide appropriately. Figure 3 shows the calculated GVD curves for our waveguide, which is fabricated by photolithography (autostepper) and Cl₂ RIE etching on the [100] silicon device layer of a SOI wafer. For dimensions of 860 × 400 nm² and an etching depth of 300 nm (see inset), the fundamental TM mode exhibits a GVD of -2.26 ps²/m at 1500 nm [3]. This value is more than 100 times larger than that of standard silica fibers (< 0.02 ps²/m). In our experiments, we employ ultra-short pulses from an optical parametric amplifier (OPA). Our SOI waveguide has a zero-dispersion wavelength (ZDWL) near 1282 nm for the fundamental TM mode. Moreover, it has a small effective area of a_eff = 0.13 µm², which enhances the nonlinear parameter dramatically and thus enables a nonlinear length of millimeter long with a fairly small input power. We have observed the formation of optical solitons inside a short SOI waveguide (only 5 mm long), as shown in Figure 4. The extremely low pulse energy requirement exhibits the great potential of SOI waveguide for low power nonlinear signal processing. We observed a significant spectral narrowing in the anomalous-dispersion regime, in contrast to all previous reported experiments. The extent of spectral narrowing depended on the carrier wavelength of input pulses because of changes in group-velocity dispersion. Our demonstration should enable the transfer of many soliton-based signal processing techniques directly to silicon devices on the chip scale.

References

Zero-Mode Waveguides for Single Molecule Studies

CNF Project # 917-00
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Abstract

Zero-mode waveguides (ZMWs) are sub-wavelength optical nanostructures designed to provide optical confinement for biophysical investigations [1]. The structures have demonstrated single molecule resolution at fluorophore concentrations exceeding 100 µM. Applications include investigations of kinetics and binding equilibriums in chemical and biological systems, especially interactions between complex enzymatic systems and their substrates.

Summary of Research

Pacific Biosciences has been working to improve our fabrication process on several fronts. Our work has focused on quantifying sources of process variability that impact device yield and developing procedures that reduce defect rates. In addition to improving throughput and yield, we are investigating new metrology tools that will address needs for high throughput and cost effective screening of optical nanostructures.

References


Figure 1: SEM of a zero mode waveguide.
Etched Facet Technology for Blue-Violet Lasers

CNF Project # 924-01
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Abstract
A continuous wave (cw) blue-violet (405 nm) emitting laser was fabricated using etched facet technology on blue laser epitaxy on free-standing GaN substrate. A 100 µm cavity laser with cw laser performance had a threshold current as low as 10 mA.

Research Summary
The motivation behind this work is to fabricate blue lasers using BinOptics’ etched facet technology (EFT). EFT has been shown to drastically reduce the cost of processing and testing InP-based lasers, and is anticipated to have similar benefits for GaN-based lasers. In addition, EFT allows the formation of shorter cavities than conventional facet cleaving—making possible a much larger number of laser devices from each wafer.

Last year, we reported the pulsed operation of blue lasers formed with etched facets on a GaN-based epitaxial material grown on a sapphire substrate. Through improved etched-facet quality and higher reflectivity facet coatings (Figure 1), we were able to increase the duty cycle of these etched facet devices. Ultimately, although the duty cycle was increased through these improvements, we were unable to achievecw operation. Analysis revealed that poor thermal conductivity of the sapphire substrate was most likely the primary reason.

This year, with access to GaN-based epitaxial laser structures grown on free-standing GaN substrates, we were able to build a cw laser with a cavity length of 100 µm and threshold current of only 10 mA. We believe the main factor behind this achievement is the high thermal conductivity of the GaN substrate.

GaN-based blue lasers currently used commercially for HD-DVD and Blu-ray players are typically limited to cavity lengths of 500 to 600 µm due to fabrication limitations imposed by conventional facet cleaving. In addition to limiting the number of devices that can be obtained from a high priced free-standing GaN wafer after epitaxy and processing, the yield and reliability of these long cavity devices may be lower because of the high defect-density of currently available materials.

The vast majority of HD-DVD and Blu-ray players are used for the read function. This application requires less than 10 to 20 mW of power compared to 200 mW or more for the write function. EFT-fabricated short cavity lasers are able to provide this power level. We estimate that we can increase the number of devices obtained from a wafer by a factor of 3 to 6 when the cavity length is reduced to 100-200 µm compared to the longer cavities currently used (Figure 2). This advance is expected to significantly contribute to lower production costs for blue lasers.

Our current work is focused on reducing the operating voltage of the EFT-based GaN lasers and qualifying reliable sources of GaN materials in anticipation of higher volume production.

Acknowledgements
Alfred Schremer, Cristian Stagarescu, Malcolm Green, Alan Morrow and the rest of the BinOptics technical team contributed to this work.
Figure 1: SEM image of a CAIBE etched laser mirror after deposition of a 4-pair high reflectivity (HR) mirror.

Figure 2: On-wafer density obtained from devices fabricated with cleaved vs. etched facets.
Optical Disk Resonators for Capture and Detection of Biothreat Agents

CNF Project # 946-01

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Abstract

We have developed an integrated-optical disk-resonator device for use as an evanescent-field biosensor. The resonator structures were patterned using conventional photolithography and the features were defined with a reactive ion etch. We also developed a procedure to immobilize antibodies on the device surface to enable capture of target pathogens, and integrated a microfluidic flow cell with the chip. By monitoring the shift in resonance wavelength as a fluid sample is sent through the cell, we are able to demonstrate reproducible capture and detection of E. coli 0157-H7 in real time.

Summary

Integrated-optical evanescent-field sensors are a class of devices that have the potential to compete with the prolific technology of surface plasmon resonance in providing sensitive detection of biological agents in real time. We have constructed disk resonator devices, also known as whispering-gallery-mode resonators (WGMR). In these devices, light sent through a straight bus waveguide evanescently couples to a disk structure in which it can circulate and interfere with light coupling into the disk at later times. If this interference is constructive for a given input wavelength, the optical field strength will build up in the cavity and the device is said to be on resonance at that wavelength. If the optical field extends above the surface of the chip, the resonance wavelength will be highly dependent on the optical properties at the surface, allowing such devices to operate as effective sensors [1].

The vertical structure of our devices was grown on a silicon substrate via plasma-enhanced chemical vapor deposition (PECVD) and consisted of a 350 nm silicon oxy-nitride guiding layer with a 1.81 index of refraction, over a 3.4 µm spacer layer of silicon dioxide. The top 1.2 µm of glass was grown using a low deposition rate recipe to improve the optical quality. The wafers were then processed by optical lithography using a GCA Autostep, and a chrome-on-quartz mask. The mask itself was fabricated by e-beam lithography at Photronics Inc., and its pattern included disk and ring resonators of several different sizes so that many different possible devices could be tested. After a post-exposure bake of the resist, the resist was developed, and the resonator features were defined by a 120 nm deep reactive ion etch. Finally, a 30 nm layer of PECVD SiO₂ was added to the surface to facilitate the attachment of antibodies. Figures 1 and 2 show features of the finished devices.

Figure 1, top: Optical microscope image of a 150 µm radius disk coupled to a bus waveguide.
Figure 2, bottom: Scanning electron microscope close-up of the coupling region between a bus waveguide and a ring resonator.
After polishing the edges to provide smooth surfaces for input and output coupling of light, we tested the optical properties of our devices using a micropositioning stage that aligned the waveguides to a single-mode fiber. By collecting the light at the output with another fiber and sweeping the wavelength of the input source, we could observe the resonance properties of the devices. Figure 3 shows the transmission spectrum for the 150 µm disk. The finesse is observed to be about 10, indicating some significant propagation loss. The disks of 200 and 500 µm radii had comparably fine resonances. In an effort to reduce these losses, we tried another fabrication procedure where the silica spacer layer was thermally grown oxide, and low pressure chemical vapor deposition was used to deposit a silicon nitride guiding layer. It was hoped that the higher optical quality of these films would reduce material losses. However, we found the losses in these waveguides to be comparable to those in the previous batch of silicon oxynitride guides, suggesting strongly that edge roughness scattering is the main source of loss.

A surface preparation was used to attach antibodies for *E. coli* O157:H7 and for *B. subtilis* endospores, and its effectiveness was verified by fluorescence microscopy.

To enable efficient and reliable delivery of pathogen samples to the device surface, we added a microfluidic cell fabricated in polydimethylsiloxane (PDMS) to the chip. The flow cell was bonded covalently to the glass surface with an oxygen plasma treatment.

While rapidly sweeping the wavelength of the tunable diode laser light source, we can closely observe the position of a single resonance and track its shift in real time. We monitored the resonance shift as a solution of heat-killed *E. coli* O157:H7 was introduced into the flow cell. A significant resonance shift of 0.3 nm was observed while the bacteria was flowing. After the cell was filled with bacteria, it was washed with PBS, and only a slight movement in the resonance position was noticed, indicating that the majority of the resonance shift was due to bacteria bound to the surface. Similar results were obtained when the experiments were repeated with different chips. Sample results are shown in Figure 4.

**Reference**

Compact Bandwidth Tunable Micro-Ring Resonators

CNF Project # 980-01
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Abstract

Optical resonators have vast applications in optical systems. For integrated optics, resonators of various geometries such as micro-ring, micro-disk, Fabry-Perot and photonic crystal resonators have been analyzed extensively. One important characteristic of an optical resonator is its bandwidth, i.e., the range of wavelength it can operate on, which is inverse to its photon lifetime and its quality factor. For some applications, such as dynamically configurable filters and optical storage devices, a resonator of tunable bandwidth is desired. Here we demonstrate a compact micro-ring resonator where the bandwidth can be tuned over a broad range using the thermo-optical effect.

Summary of Research

The resonance bandwidth of a cavity depends on the intrinsic loss inside the cavity and its coupling with the outside. For a micro-ring resonator side-coupled to two bus waveguides (Figure 1a), it scales linearly with the total loss into all three channels, \( \kappa + \kappa' + \alpha \), where \( \kappa \) and \( \kappa' \) are the power coupling coefficients to the input and output waveguides, and \( \alpha \) is the intrinsic round trip power loss coefficient inside the ring. High extinction ratio can be obtained only with “critical coupling” \( (\kappa = \kappa' + \alpha) \). In this case, the bandwidth measured in full-width at half-maximum (FWHM) can be analytically written as:

\[
\Delta \lambda = \frac{\lambda_0^2}{n_g L} \ln \left( \frac{1}{1 - \kappa} \right)
\]

where \( \lambda_0 \) and \( n_g \) are the central resonant wavelength and the corresponding group index, and \( L = 2\pi R \) is the physical length of the ring. For resonators of high quality factor \((\alpha, \kappa, \kappa' << 1)\), it reduces to:

\[
\Delta \lambda = \frac{\lambda_0^2 \kappa}{n_g L}
\]

Therefore, by tuning the coupling \( \kappa \) and \( \kappa' \) independently and ensuring \( \kappa = \kappa' + \alpha \), one can tune the bandwidth while maintaining a high extinction ratio.

A tunable interferometer can be incorporated to tune the coupling. As shown in Figure 1b, the original waveguide-ring side-coupling is replaced by two interferometric couplers, each consisting of two side-coupling points and the two segments in between as the interfering arms. Now the effective coupling coefficient \( \kappa \) depends on not only the individual coupling coefficient \( \kappa_0 \), but also the optical path length difference between the two arms, which can be tuned by either thermo-optical or electro-optical effect. Assuming the amplitude and phase response as \( t_b \phi_b \) (bus) and \( t_r \phi_r \) (ring), we can write \( \kappa \) as \( \kappa = \kappa_0 (1 - \kappa_0) (t_b^2 + t_r^2 + 2t_b t_r \cos(\phi_b - \phi_r)) \).

As the phase difference \( \Delta \phi = \phi_b - \phi_r \) scans over the \( (0, \pi) \) range, the resulted \( \kappa \) dramatically varies from \( \kappa_0 (1 - \kappa_0)(t_b - t_r)^2 \) to \( \kappa_0 (1 + \kappa_0)(t_b + t_r)^2 \), and the bandwidth \( \Delta \lambda \) varies correspondingly. The lower limit of \( \Delta \lambda \) is determined by the intrinsic loss of the ring where \( \kappa = \alpha, \kappa' = 0 \), equivalent to the case where a micro-ring is critically coupled to a single bus waveguide. The upper limit of \( \Delta \lambda \) is achieved at \( \kappa = \kappa_0 (1 - \kappa) (t_b + t_r)^2, \kappa = \kappa - \alpha \), and can be simply adjusted by design of \( \kappa_0 \).

We fabricated our devices on a Unibond silicon-on-insulator (SOI) substrate, with 3 \( \mu \)m buried oxide and 240 nm silicon layer. The waveguide and micro-ring are 520 nm wide and are defined by electron-beam lithography followed by reactive ion etch. The ring has a radius of 10 \( \mu \)m and the bus waveguide has a bend radius of 5.78 \( \mu \)m. The spacing between the waveguide and ring is 50 nm to ensure strong coupling. From calculation, the power coupling coefficient at each point is about 5.3% for TE-like mode at wavelength 1.55 \( \mu \)m. After patterning the silicon layer,
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an oxide top cladding is first deposited via plasma-enhanced chemical vapor deposition (PECVD) and then planarized with chemical mechanical polishing (CMP), leaving a final thickness of ~ 1 µm. Thermal heaters are then patterned on top of the bus waveguides with electron-beam lithography, evaporation and lift-off process. The heaters consist of 4 nm Ti adhesion layer and 100 nm Ni, with sheet resistance measured to be 1.6 Ohms. As a last step, electrical contact pads consisting of 5 nm Ti adhesion layer and 150 nm Au are then fabricated with a photolithography, evaporation and lift-off process. The measured total resistance for each heater is ~ 160 Ohms.

We tested the devices with a TE-like polarized light from a wavelength tunable laser. Nanotapers are used at both ends of the silicon wire waveguide to ensure low-loss coupling to the optical fiber. The transmission spectrum of one device with both heaters off is shown in Figure 3. The resonance wavelengths are mostly determined by the ring, and the free spectral range (FSR) is measured to be ~ 9 nm, agreeing well with theory. Due to its interference nature, the effective coupling oscillates with wavelength and therefore both the bandwidth and extinction ratio are obviously different for different resonances. As an eye guide, we sketch the curve of effective coupling as the dash line in Figure 3. At low coupling level, the resonances have narrower bandwidth and smaller extinction ratio, both of which increase as the effective coupling increases.

The bandwidth and extinction ratio can be tuned as we drive the micro-heaters to tune the effective coupling. As an example, Figure 4 shows the resonance at 1557 nm at different heater powers. With both heaters off, the resonance has a bandwidth less than 0.1 nm and an extinction ratio less than 3 dB, and it is in the “under coupling” regime. As we turn on the two heaters to increase the coupling at the input side and reduce the coupling at the output side, we increase the extinction ratio to > 15 dB and obtain a bandwidth about 0.1 nm. As we increase the input and output coupling further and maintain the critical coupling, the resonance bandwidth can be increased up to 0.6 nm with high extinction ratio. Theoretically, with appropriate design, the bandwidth can be tuned from < 0.02 nm (with reduced propagation loss, $Q ~ 80 000$) to > 10 nm (with increased coupling $\kappa$). Such a broad tuning range can be useful for integrated dynamically configurable filters and optical storage devices.
All-Optical Compact Silicon Comb Switch

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Abstract

In this project, we demonstrate a 1 × 2 all-optical comb switch using a 200 µm diameter silicon ring resonator with a switching time of less than 1 ns. The switch overcomes the small bandwidth of the traditional ring resonator, and works for wavelength division multiplexing applications. The device has a footprint of ~ 0.04 mm² and enables switching of a large number (~ 40) of channels spaced by ~ 0.83 nm.

Summary of Research

Optical switches for dense wavelength division multiplexing (DWDM) are key components for all optical networks and optical interconnects on chip [1]. Silicon photonics has attained much attention in recent years owing to the maturity of silicon in the electronic industry and its possibility to combine both photonic and electronic devices all on one chip [2]. However due to the small index change of the silicon that can be attained by carrier dispersion effect, silicon as active devices remains a challenge. High Q devices can alleviate this limitation [3]. For example, our group has demonstrated 12.5 GHz electro-optical modulators by using 10 µm diameter ring resonators [4]. High Q devices, on the other hand, operate by sacrificing the optical bandwidth, and therefore are usually not suitable for DWDM and broadband applications. Although cascaded ring resonators can be used for multiple wavelengths, the resonance of each resonator is difficult to control due to fabrication and temperature variations [5]. A silicon optical switch for DWDM applications is still missing at present.

In this project, we propose and demonstrate a technique of comb switching by using a single ring resonator for DWDM applications. The ring resonator’s free spectral range (FSR) corresponds to the wavelength spacing in DWDM, and therefore can simultaneously switch all of the channels in DWDM applications. Therefore, the high Q advantage of the ring resonator is kept without suffering from the traditional small bandwidth limitation. We demonstrate a device with a working area of 200 µm × 200 µm and a switching time of less than 1 ns.

We fabricated a silicon-on-insulator ring resonator with a 200 µm diameter and a 450-nm-wide by 250-nm-high rectangular cross section by electron beam lithography and subsequent plasma reactive ion etching (see Figure 1). The ring is coupled to two straight waveguides with the same cross section, one acting as an input port and through port, and the other acting as a drop port. The minimum distance between waveguides and ring outer edges is 400 nm.
The quasi-TM transmission spectrum shows that the FSR of this ring is 0.83 nm at a wavelength around 1530 nm, and a quality factor $Q = 18,500$. The transmission to the through and drop ports is highly wavelength sensitive. On resonance, the light is coupled into the ring and collected by the drop waveguide. If the refractive index of the ring waveguide is changed, the light, initially on resonance, is redirected to the through port. Note that all of the resonance wavelengths will be shifted simultaneously, which enables switching of multiple wavelengths using a single ring resonator. In Figure 2, we show 40 channels, with the transmission drop of the through port about 15 dB and the transmission rise of the drop port about 15 dB on resonance.

To demonstrate the concept of comb switching, we use two continuous-wave tunable lasers, both on resonance with the ring resonator. We use a $fs$ pulse laser centered at a wavelength 400 nm to generate free carriers by shining it on the top of the ring. At this wavelength the laser is strongly absorbed by the silicon layer and free electron-hole pairs are excited in less than 1 ps. The generated electron-hole pairs modify the refractive index and the absorption of silicon, and therefore tune the cavity resonance. Figure 3 shows the time dependence of the probe signal transmission. The amount of modulation depth is $\sim 90\%$ for both the transmission and drop ports. The switching time is measured to be 100 ps, limited by our detector. The switch-off time is determined by free-carrier lifetime of the photo-excited carriers and is measured to be $\sim 500$ ps [3].

The probe resonance is shifted by $\Delta \lambda = -0.5$ nm, which corresponds to an effective index change of $-6.5 \times 10^{-4}$ induced by a carrier concentration of $\Delta N = \Delta P = 2.2 \times 10^{17}$ cm$^{-3}$ [6]. We estimate that the actual power absorbed by the ring resonator to excite this carrier concentration is only 8 pJ [3].

In conclusion, we propose and demonstrate an all optical silicon switch by using a ring resonator on a silicon chip. We have demonstrated a 1 × 2 all optical switch with a wavelength spacing of $\sim 0.83$ nm, a switch time of $\sim 1$ ns and a small area of 200 $\mu$m × 200 $\mu$m.

References
Polysilicon-on-Insulator Photonic Devices

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Abstract
Integrated optical interconnects based on silicon photonic devices appear promising to replace metallic interconnects, which increasingly are the limiting factor in high-performance microelectronics. Most of the major advances in silicon photonics have been based on single-crystalline silicon-on-insulator (SOI), whose material properties are well-understood due to extensive investment by the microelectronics industry. However, large-scale integration of photonics and microelectronics will require multiple vertical layers of electrically-active material. We demonstrate optical microresonators in complementary metal oxide semiconductor (CMOS)-compatible polycrystalline silicon with intrinsic quality factors of 20,000. The resonators can be coupled in-plane to polysilicon waveguides, or coupled vertically to low-loss crystalline silicon waveguides for dense integration on chip.

Summary of Research
Polycrystalline silicon may be a flexible solution for rapid integration and immediate introduction of active photonics into standard CMOS chips [1]. Due to its relatively poor optical properties, polysilicon has largely been ignored by the photonics community, with a few notable exceptions: a three-dimensional photonic crystal with a band gap at infrared wavelengths demonstrated by Lin et al. [2], passive all-polysilicon ring resonators demonstrated by Maki et al. [3], and a MOS-based electro-optic modulator by Liu et al. [4], which included a polysilicon gate built into a crystalline silicon waveguide. If low-loss waveguides (e.g., using amorphous silicon, crystalline silicon, or silicon nitride) could be utilized to efficiently guide light to and from resonant polysilicon devices, then the quality factor $Q$ of polysilicon resonators becomes our main figure of merit instead of total losses. Loaded $Q$ factors of 10,000 are achievable even with high optical losses of 35 dB/cm, and $Q$ values around 5,000-10,000 are sufficient to build state-of-the-art active devices such as electro-optic modulators [5].

To fabricate optical devices in polysilicon, a 2 µm silicon dioxide layer is thermally grown on a 4-inch silicon substrate and a 250 nm thin film of amorphous silicon is deposited by low-pressure chemical vapor deposition (LPCVD) at 550°C. The r.m.s. surface roughness of this film is measured by atomic force microscopy (AFM) to be ~0.3 nm. The sample is annealed in N₂ at 600°C and 1100°C.

The initial 600°C anneal crystallizes the sample into polysilicon, while the additional 1100°C anneal serves to maximize the crystallized fraction and remove defects from the crystalline regions [6]. The final r.m.s. surface roughness of the film is measured by AFM to be ~0.7 nm.

From this point on, the samples are treated just like commercial SOI wafers. E-beam resist is spun on and patterned by e-beam lithography, and the pattern is transferred by reactive ion etching using a standard
chlorine-based shallow silicon etch recipe. SiO$_2$ cladding is deposited by plasma-enhanced chemical vapor deposition (PECVD). An example polysilicon ring resonator laterally-coupled to a polysilicon bus waveguide is shown in Figure 1. The spectrum of this device is shown in Figure 2, with loaded $Q$ values over 10,000, intrinsic $Q$ values around 20,000, and high extinction ratios over 15 dB. To our knowledge, these are the highest values of $Q$ demonstrated in polysilicon optical resonators.

We have also shown loaded $Q$ values of 4,000 in polysilicon films annealed at only 600°C, demonstrating the range of $Q$ values that can be obtained with different thermal budgets.

Additionally, we show polysilicon ring resonators vertically coupled to low-loss crystalline silicon waveguides. We start with a commercial SOI wafer containing a 3 µm buried oxide (BOX) layer and a 250 nm-thick silicon layer. Waveguides are patterned in the crystalline silicon layer by e-beam lithography and reactive ion etching, and a 350 nm film of oxide is deposited by PECVD from a tetraethoxysilane (TEOS) precursor. Then the fabrication steps of the previous paragraph are repeated on the new substrate to build polysilicon resonators. Figure 3 shows an SEM of a polysilicon ring resonator and a buried silicon waveguide before the final oxide cladding. Figure 4 shows a spectrum of such a device with loaded $Q$ values over 4,000.

To our knowledge, this is the first demonstration of this type of mixed-silicon optical system.

**References**


Sub-100-nm Light Confinement in Transparent Photonic Wires

CNF Project # 980-01

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Abstract

We directly measure the field confined in an 85 nm gap in a silicon slotted waveguide using a nanoscale probe to locally perturb the phase velocity of the light. We detect this perturbation interferometrically on-chip.

Summary of Research

Trapping light to regions much smaller than its wavelength greatly enhances interaction between light and matter [1]. This enhanced light matter interaction forms the basis for many photonic devices including lasers, sensors, and modulators. Typically light confinement is limited to a half wavelength in the propagation medium (\(\lambda/2n\)). Surface plasmon modes in metals provide the means to achieve light confinement in regions of cross sectional dimensions less than \(\lambda/2n\), however this usually comes at the cost of increased optical loss due to absorption by the metal [2, 3].

Recently it was theoretically shown that a nanoscale slot in a silicon waveguide could, in principle, achieve loss-less light confinement of less than \(\lambda/15\) in air [1, 4] by utilizing the electric field discontinuities at the boundaries of high-index-contrast waveguides. An example of such a structure with an 85 nm slot is shown in Figure 1a. Figure 1b plots the fundamental TE mode showing clear confinement of light to the slot region. While evidence of sub-100 nanometer light confinement has been reported from light emission of materials embedded in the slot [5], direct measurement has remained beyond the ability of near field measurement techniques: aperture near field scanning optical microscope (NSOM) probes are larger than the nanoscale slot, and apertureless NSOM techniques are primarily sensitive to the component of the field not confined in the slot region [6, 7].

To overcome the limitations of current near field measurement techniques we employ a novel transmission-based near field scanning optical microscopy (TraNSOM) technique which is based on changes in transmission through a photonic structure induced by near field perturbation by a nm-scale atomic force microscope (AFM) probe [7]. The probe used in our experiment is a Veeco high aspect ratio silicon shown in Figure 2. To ensure that the measurement is polarization independent, we measure the waveguide in an unbalanced Mach-Zender interferometer (MZI) configuration where we detect changes in transmission resulting from the probe-induced change in phase velocity.

Figure 1: (a) SEM of a slot waveguide; (b) calculated fundamental TE optical mode.

Figure 2: High aspect ratio Si AFM probe used to map the optical field.
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(which is polarization insensitive [6]). The MZI used in this experiment was fabricated in 250 nm thick silicon on insulator using electron beam lithography and reactive ion etching. The device is approximately 250 μm long consisting of one arm with a 300 nm wide reference waveguide, and another arm with a 450 nm wide waveguide with a 85 nm slot (measured at the waveguide center using a scanning electron microscope (SEM)). To measure the change in phase velocity we measure the power transmitted through the MZI as a function of probe position. At the wavelength chosen for this experiment (1524.5 nm) this change in transmission is proportional to the local magnitude of |E|^2 [6], therefore we construct a 2D profile of the optical mode.

Figure 3a shows the topography of the slot waveguide as measured by the AFM and Figure 3b shows the simultaneously recorded transmitted power. The large increase in transmission when the probe is in the slot is a direct measurement of the strong electric field confined in this region. Note that we have chosen a wavelength where the phase in the slot waveguide leads that of the reference waveguide by π/2. Therefore, decreasing the phase velocity in the slot waveguide with the silicon probe increases the total transmittivity.

Choosing this wavelength, where transmission increases under the influence of the probe, ensures that the dominant effect of the probe is indeed from a phase shift and not increased loss (which would result in a decrease in transmission at this wavelength). The solid line in Figure 4 shows a cross section of the TraNSOM measurement. Notice the sharp peak of the field in the slot has 20 nm full width half-maximum which agrees with the theoretical phase shift (dashed line) computed by convolving the probe profile with the fundamental TE mode shown in Figure 1b.

This TraNSOM measurement represents, to our knowledge, the strongest light confinement measured in dielectric photonic structures. Devices based on this strong light confinement will benefit greatly by the increased light-matter interaction in the slot region.

References

Ultra-Low Power Frequency Conversion in Silicon Micro-Ring Resonators

CNF Project # 980-01
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Abstract
We demonstrate ultra-low-power frequency conversion in a micrometer-sized photonic silicon structure via microcavity-enhanced four-wave mixing. Using continuous wave pump powers of 5 mW, we achieve frequency conversion across the C-band in a compact 50 µm radius ring.

Summary of Research
As photonic components scale down in size and the demand for high bandwidth on a chip increases, schemes commonly used in telecommunications, such as wavelength division multiplexing (WDM), are being considered for on chip optical interconnect architectures. In such architectures, the ability to perform wavelength conversion is needed and to date has only been demonstrated on a silicon chip using schemes that are either fundamentally narrow band [1], require large devices or require large powers [2-9], making their integration on a microelectronic chip challenging. Recently, we have demonstrated using the efficient nonlinear optical process of four-wave mixing in silicon waveguides for large bandwidths of hundreds of nanometers of all-optical wavelength conversion [9]. By increasing the total length of the waveguide to maximize the efficiency, efficient wavelength conversion has been realized using pump powers on the order of hundreds of milliwatts [2-9], however for chip applications, ultra-low power and compact devices with micrometer dimensions are needed.

Here we demonstrate parametric wavelength conversion using ultra-low powers in compact micrometer structures using microcavities for enhancing the four-wave mixing in silicon. We show wavelength conversion at 16 different resonances across the C-band using ultra-low coupled input pump and signal powers of 5 mW and 100 µW, respectively. The enhancement of the non-linear process is achieved using a microcavity with Q = 20,000.

The resonator used here (Figure 1) is a 50 µm radius ring resonator fabricated on a silicon-on-insulator substrate consisting of a silicon waveguide with a height of 300 nm and a width of 400 nm. The cross-sectional dimensions of our waveguide provide us with a large anomalous value of 1500 ps/(nm·km) near a pump wavelength of 1550 nm, allowing phase matching over a bandwidth of more than 50 nm [9]. A tapered lens fiber with a fiber polarization controller to optimize the TE-like mode is aligned to the inverse taper mode converter in the silicon waveguide, allowing for coupling into the waveguide. The pump is
centered on a resonance of the ring at 1549.84 nm and the signal is scanned across the C-band. The output of the waveguide is sent to an optical spectrum analyzer (OSA). The bus waveguide loss is 3 dB/cm, due to stitching errors from the electron beam lithography, scattering losses due to roughness and bending losses through the waveguide. The transmission of the structure is shown in Figure 2. As is shown in the plot, the extinction ratio of this resonance is greater than 15 dB, indicating that we are operating close to critical coupling.

An example of a measured four-wave mixing spectrum is shown in Figure 3. The pump is tuned to the resonance at 1549.84 nm while the signal is tuned to a different resonance at about 1544 nm. The converted signals can only be measured when both the pump and the signal are tuned into resonances of the ring, indicating that the conversion is indeed occurring in the ring. The coupled pump and signal powers are 5 mW and 100 µW before the ring, respectively. The converted signal coupled back into the bus waveguide from the ring is 300 nW, indicating a net conversion efficiency from the input signal to converted output of -25 dB. Our conversion efficiency is currently limited by nonlinear loss mechanisms arising from free carrier absorption. Integrating a diode-structure across the device and reverse biasing for sweeping out carriers would reduce the nonlinear loss mechanisms [10].

To demonstrate the wide bandwidth tunability of this device, we scan the input signal across the C-band and measure the converted output (see Figure 4, lower curve). The transmission of TE-like polarized amplified spontaneous emission (ASE) through the device is shown as the upper curve. The free spectral range (FSR) of 2 nm is evident in both curves in Figure 4. As shown by the lower curve, the conversion is enhanced by 15 dB each of the 16 times the signal enters a resonance. One can see the large bandwidth conversion of over 30 nm limited only by our experimental setup. These measurements of ultra-low power on a micrometer-size device would enable the integration of non-linear components on-chip for all-optical functions on chip such as wavelength conversion, signal regeneration, amplification, and switching.

References
Silicon Nanophotonics

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Abstract

Nanofabricated silicon waveguides provide a remarkable system for developing massively integrated ultrafast nonlinear optics. Because of their extremely high mode field concentrations and the ability to evanescently couple the modes to highly-active nonlinear optical cladding materials, they provide an ideal system for both second- and third-order nonlinear optical devices. In our recent work, we have built a variety of silicon nanofabricated waveguide based devices, including recent work on optical detectors, electro-optic modulators, and all-optical modulators.

Summary of Research

Over the past several months, we have made use of the world-class electron beam lithography capabilities at the Cornell NanoScale Facility to make a number of novel integrated optical devices using standard silicon-on-insulator wafers. Silicon provides a remarkable system for integrated optics—it is transparent in the critical telecommunications bands, is ideal for the fabrication of nanoscale structures, and it provides an extremely high index of refraction. Taking advantage of these properties, it is possible to construct waveguides with extremely small mode volumes, which concentrate light into submicron areas. Because of this high mode field concentration, it is possible to create systems with extremely strong optical nonlinearity.

Our recent work has focused on the development of silicon:polymer hybrid devices. By utilizing both the high mode field concentration in the silicon waveguides and the ability to create nanostructured waveguides, we are able to concentrate the optical fields into polymer layers surrounding our optical waveguides. These structures can then be used to create low-voltage hybrid modulators, novel optical detectors, wavelength converters, and all-optical modulators. Publications are forthcoming in particular on novel waveguide structures and silicon detectors in the near infrared.
Figure 1: Slot waveguide and slot waveguide resonator.

Figure 2: Electrical contact structures to an optical waveguide.

Figure 3: SEM image of a lateral electrical contact structure.

Figure 4: Optical plan view of several ring resonators and electrical contacts.
Fabrication of Nanochannels in Glass

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Abstract

The 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 it contains eight isolated from each other flow-cells. 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 a 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 a project for single viruses recognition and classification using light scattering.

Fabrication Procedure

A borosilicate glass wafer (Schott Glass, Germany) was precleaned 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 photo-resist (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 the CNF 10X stepper (GCA Corp., Andover, MA), and the micro-sized features were patterned using the EV620 contact aligner (Electronic Visions, Phoenix, AZ). The channels were etched using 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 the 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 of Research

The fabrication procedure has been developed for repeatable and reliable fabrication of nanometer-sized channels, sealed between two glass wafers.

References

Figure 1: Optical micrograph of the fabricated glass nanochannels.

Figure 2: AFM topography of a nanochannel.
**High Power Monolithic Passively Modelocked Semiconductor Laser at 1550 nm Wavelength**

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### Abstract

In recent years, there has been a surging interest in generating ultrashort pulses from a monolithic, electrically pumped semiconductor laser. Modelocking the laser diodes usually produces pulses whose widths are on the order of a few pico-seconds. The drawback of a monolithic semiconductor laser is that it produces pulses whose energies do not usually exceed a few pico-joules. Using a slab coupled optical waveguide geometry laser, we have managed to increase the limit by an order of magnitude. Our lasers directly produce pulses with energies exceeding 40 pJ.

### Summary of Research

Our group is investigating the generation of high energy ultrashort optical pulses from a semiconductor diode laser. The geometry that we are interested in is the slab couple optical waveguide laser. The structure makes it possible to increase the saturation energy which allows us to generate pulses whose energies are tens of pico-joules. The added advantage of this structure is that the laser produces a single transverse mode beam with large modal area ($\approx$ 20 $\mu$m$^2$).

Using MOCVD, epitaxial layers of InGaAsP are grown on an n-InP substrate. A 4 $\mu$m thick layer of InGaAsP serves as the mode guiding section. The active region which comprises of five 8 nm InGaAsP quantum wells is grown on top of this mode guiding layer. The active region is then covered by a 1 $\mu$m thick p-InP which allows us to make ohmic contacts for the laser on the top of the ridge (Figures 1, 2).

![Figure 1: Schematic of the cross-section of the laser.](image1.png)

![Figure 2: SEM of one of the facets of the fabricated lasers. The region marked by InGaAsP is the wave-guiding section which is 4 m thick. The section marked MQW region consists of five periods of InGaAsP based quantum wells and barriers.](image2.png)
The wafer was covered with a 300 nm SiO$_2$ film using the IPE PECVD tool. Waveguides were defined using standard optical lithography techniques. We used SPR 220-3.0 resist and exposed it in the GCA AutoStep 200. The pattern was transferred to the underlying oxide layer and after stripping the resist, the patterned oxide was used as etch mask for etching the semiconductor. Using a Cl$_2$/H$_2$/Ar recipe, the semiconductor was etched in an ICP tool. The ridge height of the waveguides was around 1.9 µm. The etch mask was removed and a fresh 280 nm layer of PECVD oxide was deposited. Again using optical lithography, we defined openings on the top of the waveguides from which we etched away the oxide using a CHF$_3$/O$_2$ RIE recipe. The Ti/Pt/Au ohmic contact was evaporated on top of the waveguides. The ohmic contact was defined using a bi-layer photoresist lift-off procedure. Once the ohmic contact was defined, the substrate was thinned down to a thickness of about 150 µm. A Ni/Ge/Au n-ohmic contact was evaporated on the back side. The sample was annealed at 400°C for 30 seconds.

The laser bars were cleaved to a length of about 9.0 mm. The bare semiconductor/air interface had 30% reflectivity. This was good enough to obtain lasing, but to increase the output power from one of the facets we evaporated three periods of Al$_2$O$_3$/Si to serve as a high reflection coating. The output facet is also coated with just one layer of Al$_2$O$_3$ in order to reduce the reflectivity to about 10%.

The laser bars were mounted on gold plated copper chuck using indium solder. To modelock the laser, the top ohmic contact was segmented and electrically isolated into two parts. The longer section served as the gain section and was forward biased. The shorter section was reverse biased and it is this section, which acts as the saturable absorber, which is the main pulse shaping agent. About 1.5 Amp of current was passed through the gain section. The reverse bias across the saturable absorber section was then gradually increased. At a reverse bias voltage between 1.5 V and 2.0 V (depending on the forward bias current), the laser modelocked. Increasing the reverse bias voltage beyond this point results in a decrease in the width of the pulse (Figure 3). The shortest pulse width measured via autocorrelation was just over 5 ps (Figure 4). The highest pulse energies though, were observed right around the reverse bias voltage where the laser modelocks. Increasing the reverse bias voltage leads to a decrease in the pulse energy. The highest pulse energy observed thus far was over 40 pJ.

References
Visible Three-Dimensional Metallic Photonic Crystal

CNF Project # 1305-04
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Abstract

We report experimental realization of a 5-layer three-dimensional (3D) metallic photonic crystal structure that exhibits characteristics of a 3D complete bandgap extending from near-infrared down to visible wavelength at around 650 nm. The structure also exhibits a new kind of non-localized passband mode in the infrared far beyond its metallic waveguide cutoff. This new passband mode is drastically different from the well-known defect mode due to point or line defects.

Summary of Research

Photonic crystals offer unique properties and promising prospects for intriguing applications. To fully exploit these possibilities, a complete bandgap from a three-dimensional (3D) structure is desirable. Yet, complete bandgap at visible wavelengths has so far been elusive due to the technological challenges in fabricating high-quality 3D crystals with sub-wavelength-scale features. Here, we report experimental demonstration of a 3D metallic woodpile-like structure with the shortest pitch to date (300 nm) over a large area (5 mm × 5 mm) that shows an omnidirectional bandgap extending from near-infrared down to visible wavelength at around 650 nm. Furthermore, a new kind of passband mode, far beyond the metallic waveguide cutoff of the structure, is first experimentally observed, opening up possibilities of new phenomena and applications.

The structure is fabricated layer-by-layer using electron beam (e-beam) lithography [1]. Figures 1 and 2 show the cross-sectional scanning electron microscopy (SEM) images of a 5-layer gold-HSQ woodpile-like 3D photonic crystal with a pitch of 300 nm. The average rod width is 105 nm. The thickness of each gold rod layer is 85 nm. Each gold rod layer is separated from its adjacent layers by a spin-on HSQ spacer with a thickness of 85 nm.

To investigate the effect of the incident angle on the optical characteristics of the 3D metallic structure, variable-angle FTIR spectroscopy is performed in reflectance mode. Figure 3 shows the measured tilt-angle reflectance of the sample depicted in Figure 1 at incident angles from 20° to 60°, at 10° increments. The tilt-angle geometry is as
shown in the inset. The data shows a band-edge fixed at a visible wavelength of around 650 nm, indicating a 3D complete bandgap exists above this wavelength. It also shows consistent high-reflectance extending to beyond 3 µm in the infrared. Within the high-reflectance region, the “double-dip” in reflectance clearly shifts to a shorter wavelength (“blue-shifts”) with an incident angle and without a change in overall spectral shape. The total spectral shift is about 200 nm for the measurement range. The data shows that, in striking contrast to defect modes induced by a 3D cavity resulting from point defect, the observed passband mode [2] is angular dependent and does not exhibit the characteristic of a 3D cavity.

In summary, an omnidirectional photonic bandgap down to the visible regime at wavelength of $\lambda = 650$ nm has been demonstrated using a metallic woodpile-like structure with the shortest pitch (300 nm) to date, which may extend the exploration of applications of full photonic bandgap into the visible. It is also shown experimentally for the first time that if dielectric spacer layers are incorporated into a metallic photonic crystal, it is possible to generate non-localized passband modes far beyond the waveguide cutoff of the structure. This result has great potential to facilitate new ways to tailor and engineer photonic bandgaps, and possibly opens up new exciting prospects for 3D photonic crystals in many applications such as light-emitting devices.

![Figure 3: FTIR-measured tilt-angle reflectance of the sample.](image)

**References**


Integrated Terahertz Waveguides and Microcavity Resonators

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Abstract

In the last few years, terahertz (THz) technologies have been extensively explored for applications in chemical and biological sensing, spectroscopy, medicine, and imaging [1, 2]. Integrated THz platforms are highly desirable for chemical and biological detection because they can provide field confinement and enhancement to increase sensitivity [2]. Some such platforms, including one dimensional (1D) parallel-plate waveguides with embedded photonic bandgap structures [3-6] and integrated microstrip resonators [7], have already been experimentally realized. Our research is focused on the development and measurement of integrated THz photonic devices. In particular, we are studying air-core metallic microcavity resonators coupled to rectangular metal waveguides [8] and 1D Bragg gratings embedded in rectangular metal waveguides. Such hollow-core THz microphotonic structures can be ideal for narrow-band sensing applications.

Summary of Research

Figure 1 shows a schematic of a typical device as well as a scanning electron microscopy (SEM) image of a waveguide-coupled microresonator. The THz electric field is polarized vertically with respect to the waveguide walls and propagates in the fundamental TEM-like TE10 mode. The THz time-domain spectrometer (TDS) used in this work was based on a Si-GaAs photoconductive emitter and a silicon-on-sapphire photoconductive detector pumped by a 90 fs Ti:sapphire ultrafast laser (780 nm, 10 mW) [6]. The TDS had a bandwidth of 3 THz and an amplitude spectral dynamic range exceeding 3000. Terahertz pulses from the spectrometer were coupled into and out of the photonic devices using hyperhemispherical Si lenses.

The integrated air-core THz waveguides and resonators were fabricated from highly doped 3 in Si wafers. Devices were patterned into the wafers using standard contact photolithography and deep-RIE Bosch etching. Next, the wafers were oxidized and metallized conformally with Ti/Au (50 nm/400 nm). Finally, two wafers, one with the patterned devices and one blank, were bonded at a temperature of 350°C and a pressure of 1.5 MPa for 45 minutes [9], then diced and polished. A photograph of the facet of a completed 2D rectangular metal waveguide is shown in Figure 1. The waveguides are generally 200 µm wide and 150 µm tall.

We have also fabricated rectangle metal waveguides with embedded 1D Bragg gratings. These gratings exhibit strong photonic bandgaps and can be combined with defect cavities to form high Q-factor resonators. The fabrication of these gratings is similar to the side-coupled cavities mentioned above with the exception that it requires one additional photolithography step and one additional etch. Initially, 25 µm deep grating teeth are defined and etched in a 3 in Si wafer. Next, the waveguide pattern is aligned to the grating teeth and the whole structure is etched an additional 100 µm in the deep-RIE Bosch etcher. The resulting structure is a 125 µm deep waveguide with a 25 µm tall grating embedded in its bottom. An SEM is shown in Figure 2.

Figure 3 displays a typical time-domain scan of the THz electric field and the accompanying power spectrum of a pulse from the spectrometer after propagation through a
waveguide coupled to a 125 µm x 125 µm square cavity. The waveform displays long ringing due to waveguide dispersion as well as the frequency-dependent transmission of the resonator (the secondary pulse at 50 ps is due to reflection off of the waveguide facets). A dip in the power spectrum at 1.33 THz is observed due to excitation of the resonator.

Square metallic cavities of dimensions 175 µm, 150 µm, and 125 µm wide have already been studied. The resonators were coupled to the waveguides via apertures. The size of the coupling aperture of each cavity was varied between 100 µm, 90 µm, and 80 µm (see Figure 1). Figure 4 shows the extracted experimental resonances for all three cavity sizes. The power transmission, \( T \), of a resonator has a Lorentzian shape given by [10]:

\[
T = \left| 1 - \frac{Q_C}{Q_C + \frac{Q_L}{Q_L - 2j(1 - f)}} \right|^2
\]

where \( f_0 \) is the center frequency of the resonance and \( Q_C \) and \( Q_L \) are the cavity Q-factors due to waveguide coupling and loss, respectively. This model was used to fit to the experimental data and the results are shown in Figure 4. The resonance frequency increases with decreasing cavity size and decreasing aperture size, which is in good agreement with the trends predicted by 2D finite-difference time-domain (FDTD) simulations. From Figure 4, we conclude that the total Q-factor of these integrated metallic cavities is less than 50 and is limited by cavity loss. Cavity losses are expected to be dominated by ohmic losses in the metal sidewalls.

References

An Ultra Compact Optical Mode Order Converter

CNF Project # 1366-05
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Abstract

We describe the design and fabrication of an ultra-compact optical mode order converter employing sub-micron waveguides. The mode converter can realize optical mode conversion between 0 order and 1st order optical modes with a device footprint less than 18 µm by 3 µm. The optical mode converter could be employed in various types of photonic integrated circuits where optical mode order conversion is needed.

Summary of Research

Photonic integrated circuits (PIC) employing nanophotonic devices have attracted increasing interest in various optical communication applications because of their advantages in high device density and low operating power. In PICs, it will be desirable if a compact optical mode order converter exists so an additional dimension of freedom can be obtained in device design [1], as different orders of optical mode have different effective refractive indexes. For example, in the case of a directional coupler with waveguides supporting both 0 order and 1st order modes, a different coupling length can be obtained from the same coupler for the two different mode orders. In the past, mode order conversion has been achieved using both bulk optics [2] and waveguide devices, such as multimode waveguide [1].

We propose a new type of optical mode converter based on an ultra-compact interferometric structure formed by nanowaveguides. The two arms of the interferometer consist of single mode nanowaveguide structures with an optical path difference of π. In a 0 to 1st order mode conversion, the input end is a single mode waveguide supporting only 0 order mode, while the output end is a multimode optical waveguide, which can support both 0 and 1st order mode. After the beam enters the input waveguide, it is split 50/50 by a Y-splitter to the two arms of the interferometer and a phase difference of π is introduced between the two beams. The two beams are then recombined at another enlarged Y-junction at the output waveguide end designed to produce a 1st order mode. The structure can also be used in reverse as a 1st to 0 order mode converter by reversing the propagation direction.

The nano-scale waveguides have strong refractive index contrast between the core and cladding. As a result, large angle Y-splitters and sharp bending are possible, making the resulting device ultra compact.

An initial fabrication and measurement results for the mode order converter shows very low loss (0.4 dB per conversion) and shows an ultra-compact footprint of less than 18 µm by 3 µm.

References

Figure 1: SEM image of the fabricated mode order converter before InP etch.

Figure 2: SEM picture of the fabricated mode order converter after InP etch.
**Surface Plasmon Enhanced Silicon-on-Insulator Metal-Semiconductor-Metal Photodetectors**

CNF Project # 1368-05

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**Abstract**

Surface plasmons and optical resonant modes were studied in complex grating structures. It was shown that surface plasmons are not responsible for anomalously large transmission and that instead, waveguide modes are responsible. Polarization independent transmission in grating structures is described along with optoelectronic and photonic device applications.

**Summary of Research**

During the last year, several significant accomplishments were made in this project. While accomplishments in device fabrication were made, the most important advancement was made during our studies of surface plasmon and optical modes in complex lamellar grating structures. It was found that what had previously been thought as responsible for the anomalously large transmission in periodically perforated structures, namely surface plasmons [1-6], was not responsible and that the phenomenon is simply caused by another type of mode; waveguide modes [7, 8]. Because waveguide modes produce this phenomenon, both polarizations and randomly polarized light can experience anomalously large transmission.

This result is important not just on a theoretical basis but especially important when it comes to using this phenomenon to develop optoelectronic and photonic devices because light incident on a photodetector is generally unpolarized. Our research last year fully and accurately described the mechanisms for anomalously large transmission, and how the energies at which this transmission occur for each wavelength can be tuned by adjusting various parameters of the device.

The three cases, shown in Figure 1, of anomalously large transmission, were considered. Case 1 was the commonly observed case of TM polarized light being entirely transmitted while TE light is reflected. Case 2 was the opposite where TE light is transmitted and TM light is reflected. Finally, Case 3, which had not been observed, was where both TE and TM polarized light can simultaneously be anomalously transmitted [8].

The ability to have both polarizations transmitted is a significant advancement and opens up this area of research for the development of numerous device applications. Currently in this project, we are fabricating the structure shown in Case 3 at the CNF to experimentally verify or theoretical results. We are also collaborating with research groups at the University of Exeter to demonstrate these same phenomena in the microwave spectral region.

**References**


Figure 1: The three different cases of anomalously large transmission in 1-D metal gratings described in this paper. Note that the cross-sections of the gratings are shown.
Focal Volume Confinement of Fluorescent Particles in Nanometer-Sized Channels

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Abstract

Using conventional photolithography, we have fabricated 500 nm wide square fluidic channels in optical grade fused silica wafers. The channel walls act as a physical barrier confining a dilute sample of fluorescently labeled species allowing for sensitive single molecule fluorescence measurements. Additionally we have fabricated fluidic channels that can be used for fluorescence image calibration and for determining distortion due to deep tissue imaging.

Summary of Research

Fluorescence detection allows for sensitive visualization and quantification of molecules in solution and microfluidic devices have developed as a platform for analyzing, separating, and investigating small samples. Additionally, the sensitivity of single molecule fluorescence is enhanced in nano-structures due to focal volume confinement. Previous work from our laboratory has demonstrated high signal-to-noise ratio (SNR) fluorescence measurements in nanofluidic devices [1, 2].

In our device, a dilute sample of fluorescently labeled particles is driven by voltage from the inlet to the outlet passing through the focal volume of the excitation beam one molecule at a time. The focal volume confinement region of the channels is a square channel that is 500 nm wide and deep and 10 µm long. The device pattern is transferred into a positive tone resist using the 5x Autostep 200 i-line wafer stepper. The device pattern is then etched into fused silica wafers using the patterned resist as a mask. Finally, inlet and outlet holes are blasted in the wafer and it is sealed using a fused silica cover-slip wafer.

In addition to the focal volume confinement project, we are also working on the fabrication of chips for aberration testing for fluorescence imaging application. Aberrations of the point spread function are often a problem in deep tissue multiphoton microscopy. We have fabricated channels of standard dimensions—from 2-50 µm wide—which can be used for both image calibration and evaluation of aberrations that occur while imaging through tissues such as skin and cartilage. Fused silica wafers are regularly patterned using standard soft contact lithography. The mask pattern is transferred to the resist which is then used to pattern a layer of chromium. Ultimately, the patterned chromium is used as a hard mask for etching on the Oxford PlasmaLab 100.

References

Feasibility Study for Making Phase Plates by Electron-Beam Lithography for Extension of the Depth of Field for Optical Microscopy

CNF Project # 1470-06
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Abstract

In biological microscopy, the depth of focus is often too short to adequate view the specimen with sufficient contrast or visibility. A solution [1,2,3] is to extend the depth of field using wavefront coding. A phase plate is made that broadens the range of adequate focus when combined with computer image processing. We made one specifically for the objective we are using and the desired depth of field.

Summary of Research

We had a particular biological application for our optics project in mind in which we needed a larger depth of focus. Our goal was to reduce error due to defocusing of the out-of-plane motions of beating cilia. In Chlamydomonas, a single celled alga that swims with a pair of cilia, the “planar” beating of its pair of cilia extends to ~ 4 µm depth at the tips, greater than the 0.8 µm depth of focus (DOF) of our microscope objective. The situation is even worse for helical ciliary beating in which the beating extends in depth to more than 10 µm and the broadening of the out-of-focus image decreases contrast too much to see the motion. A DOF increase to 8 µm would be a big improvement.

Using electron beam lithography, we made our DOF extending phase plate specifically for a Nikon 60X 1.0 NA objective lens. Electron beam lithography has the advantage of being able to make higher quality phase plates for wavefront coding than those manufactured to date. We used a well known design [4], namely a cubic phase profile with $\rho = \alpha(x^3 + y^3)$, where $\alpha = 20\pi$ radians was selected for implementation.

The profile was plotted and adjusted to modulo $2\pi$ to physically limit the depth of the phase plate. The total depth was then quantized to 32 discrete amplitude zones, corresponding to 32 different depths (Figure 1A. CAD design, 1D. AFM profile). The dimensions of the phase plate were scaled to 7 mm to cover the back aperture of our microscopes objective. The procedure involves generating the CAD layout, programming the electron beam doses to give grayscale images that are exposed onto a photoresist methyl methacrylate (PMMA) (Figure 1B. photograph of developed photoresist) that was then developed leaving the embossed pattern measured with an atomic force microscope (Figure 1C. AFM of photoresist, 1D. AFM profile), and then converted to a more permanent quartz phase plate by plasma ion etching.

We were successful on the first try, so we can study Chlamydomonas and other cells with cilia even when the cilia are not oriented in the imaging plane.

References

X-Ray Transmission Mirrors

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Abstract

We are using CNF to build thin-film x-ray transmission mirrors. These mirrors would be a new x-ray optic for tailoring the properties of x-ray beams at synchrotron sources. The transmission x-ray mirror, in conjunction with the traditional thicker x-ray mirror, can allow us to have a broad and tunable band-pass in x-ray energies on the synchrotron beam-line. The transmission mirrors are constructed of large silicon nitride windows, 0.6 mm wide by 85 mm long and 300 to 400 nm thick.

Summary of Research

We have fabricated potential x-ray transmission mirrors at the CNF, which are presently being tested at the Cornell High Energy Synchrotron Source (CHESS) for their optical properties. The transmission x-ray mirrors will be used in conjunction with traditional reflection x-ray mirrors to allow us to have a broad and tunable band-pass in x-ray energies on the synchrotron beam-line. We are trying to achieve a roughly 30% energy bandwidth over the energy range of 10 keV to 13 keV. By comparison, multilayer x-ray optics which are frequently used at synchrotrons, typically have a bandwidth below 2%. The upper limit of bandwidth achieved with multilayer optics has been 10%. We hope the x-ray transmission mirror will allow us to get a higher width in the energy spectrum, and allow us to tune the bandwidth of the x-ray beam for Laue x-ray diffraction experiments.

Many x-ray optics are reflecting optics in order to avoid significant x-ray absorption. For x-rays, all materials have an index of refraction slightly less then 1. The index of refraction is often expressed as \( n = 1 - \delta \), with \( \delta \) having a value on the order of \( 10^{-5} \). At grazing incidence (less than 1°) from a surface, there is total external reflection, because the index of refraction in a material is slightly less than the index of refraction of vacuum (\( n = 1 \)). High quality x-ray reflection optics need to be very smooth and flat to be effective, with a surface that has less then a 10 arc second (50 microrad) variation. They also tend to have relatively large surfaces because of the small angles of the reflections.

A thick (regular) x-ray mirror sets the high limit on the photon energy. The higher the angle of reflection, the lower the x-ray energy cutoff is of the reflected beam (see the Rhodium mirror reflection curve in Figure 1). The x-rays with a photon energy lower then the energy cut off for total
external reflection are reflected by the mirror surface and the higher energies are absorbed by the mirror. By adjusting the angle of an x-ray mirror, you are able to adjust the high photon energy limit of the reflected beam from the very wide spectrum of x-rays that the synchrotron produces.

The x-ray transmission mirror works exactly like the reflection mirror, but instead of using the beam that is reflected from the surface, we use the beam transmitted through the mirror. This switches the mirror from being a high energy filter to a low energy filter. Figure 1 gives a simple schematic of the set up and a graph of the effect that each optic will have on the energy spectrum of the beam. In order for the transmission mirror to work, it has to be very thin. X-ray transmission mirrors have been demonstrated with large soap bubble films 1000 to 30 nm thick in an 8 × 20 cm frame by B.M. Lairson and D. Bilderback [1]. Our aim is to construct similar optics that are very thin with large window openings that are made of something which lasts longer than a bubble.

At CNF we have fabricated some large silicon nitride windows which are 85 mm long, 0.6 mm wide and 300-400 nm thick. At the target angle of 0.22°, this will allow a 0.4 by 0.4 mm x-ray beam to pass through the transmission mirror. We have fabricated many widows of this size and thickness, which are now being tested at CHESS.

The first successful tests of the transmission mirrors were on June 21, 2007, at the B1 white beam station at CHESS. Figure 2 shows the transmission for a silicon nitride window at two different angles and the predicted transmission for the same angles. The transmission of the mirrors is in good agreement with these predictions.

References
2D Sub-Wavelength Metallic Hole-Array

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Abstract
A set of two-dimensional metallic hole-array samples was fabricated and investigated. The zero-order transmission spectra were measured and sharp transmission peaks were observed at sub-wavelengths. The average field strength through two-dimensional (2D) holes, defined as the ratio of measured transmission to the fraction of area occupied by air-holes, has shown larger than 100% enhancement and the physical origin of extraordinary transmission has also been studied.

Summary
2D metallic hole-arrays were first demonstrated experimentally and studied by Ebbesen et al. [1] in 1998. The optical output through these periodic arrays displayed an extraordinary transmission enhancement at sub-wavelengths which was larger than the prediction by standard aperture theory [2]. In general, the physical mechanism responsible for the extraordinary transmission was attributed to the coupling of photons to the surface plasmon polaritons (SPPs). The extraordinary transmission at sub-wavelengths has a number of potential applications in near-field microscopy, subwavelength lithography, etc. In our experiment, 2D hexagonal hole-arrays with different lattices and thickness were fabricated and studied. The transmission result showed a > 100% transmission enhancement at sub-wavelengths when normalized to the area of air-holes.

The 2D hole-arrays with various geometrical factors perforated in optically thin metallic film were defined by conventional photolithography and fabricated by lift-off process. The SPR resist was spun onto silicon (Si) substrates and pre-baked. The resist-coated samples were then exposed by using the GCA AutoStep 200. The samples were baked with ammonia vapor in the YES image reversal oven after a post-exposure bake. After the oven, the samples were flood exposed with the EV620 contact aligner. The samples were then developed in MF321 developer which left the resist cylinders on the substrates. Au film with 50 to 150 nm thick was evaporated on the samples using e-beam evaporators. The resist was finally lifted-off by 1165 resist remover.

References
Optofluidic Propulsion Using Nanophotonic Structures

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Abstract

The goal of this research is to develop a new class of micro-/nanofluidic device which exploits the intense electromagnetic fields present in micro- and nanophotonic structures as the primary transport mechanism. This mechanism exploits the near-field optical gradients (which serve to confine particles through a Lorenz force) and concentrated optical energy (resulting in intense scattering and absorption forces for propulsion through photon momentum transfer) present in these devices to perform a series of particle handling operations including transport, concentration and separation. We present our experimental results using waveguides fabricated from SU-8 cross-linked polymer integrated with polydimethylsiloxane (PDMS) microfluidics demonstrating the dynamic trapping of flowing particles and subsequent radiation pressure propulsion. Figure 1 shows a schematic of our system along with a mode field calculation illustrating the force vectors applied to the particle. These devices have the potential to develop into sophisticated optical train tracks, allowing for a new paradigm in particle manipulation.

Summary of Research

The ability to perform controlled trapping and concentration of nanoscale objects is becoming an important part of the development of high sensitivity, low limit of detection nanosensor devices. Essential to the development of integrated microfluidic devices incorporating such functionality is the ability to fuse active target handling components with electrical and/or optical sensor elements. Traditional optical trapping mechanisms allow for a great deal of control in two dimensions, but exhibit a fundamental limitation. It is well known that electromagnetic forces applied to a particle are proportional to the intensity of the incident light. This intensity is equal to optical power divided by the cross sectional area (spot size) of the trapping laser. To achieve the necessary intensity for trapping smaller particles, smaller spot sizes obtained using high numerical aperture lenses are needed. This necessarily results in a decrease in the interaction length between the optical force field and the transported particle, limiting the distance over which it can travel.

Waveguide structures confine light within microscale structures through total internal reflection over extremely long distances. While the majority of the optical energy is confined within the solid core of a waveguide, there exists a near-field non-propagating component called the evanescent field. This field extends from the waveguide surface and exponentially decays into the surrounding medium over a distance of a few hundred nanometers. This rapid decay in optical intensity results in a strong trapping field. Such confinement also enables radiation pressure propulsion of particles along the length of a waveguide.
Experimental Setup

Our experimental setup is illustrated in Figure 2. We use a syringe pump to flow our particle solution through the channels. The particles used are polystyrene beads of various diameters which are impregnated with green or red fluorescent dyes sensitive to specific wavelengths. Dichromatic filters and a mercury lamp provide the excitation light, and filter out unwanted wavelengths (excitation and laser). Our particle solution is made using a 100 mM 7.0 pH phosphate buffer solution in a 100:1 ratio with our particle suspension.

Experimental Results

We were able to achieve trapping of 3 µm polystyrene spheres on our waveguides with bulk particle speeds of up to 10 µm/s, and at an input laser power of 80 mW. Once trapped, particles exhibited propulsion due to radiation pressure, as seen in Figure 3. The image frames are explained as follows: (a) Position of particles pre-trapping, white line indicates (b) Top particle (red) reaches waveguide structure (c) Propulsion of red particle along waveguide structure. Blue particle nears waveguide (d) Red particle no longer trapped on waveguide. Propulsion of blue particle along waveguide (e) Continued propulsion of blue particle (f) Both particles no longer interact with waveguide.

The propulsion speed was considerably faster, almost double the bulk particle speed. This resulted in a considerable shift in particle position before and after trapping, even with the relatively short trapping time. The untrapping of particles in this particular case were due to collision with the sidewall of the microchannel (upper particle), and a minor defect in the waveguide (lower particle). As scattered light from the waveguide is filtered out during our experiments, we believe this effect may have been due to two-photon excitation, as our particles are excited at 542 nm, while the laser wavelength is at 980 nm.

Fabrication

The chosen waveguide material is SU-8 which is an excellent waveguide material with high transparency in the wavelength range of interest (850-1100 nm) for trapping applications. SU-8 is also compatible with many substrates, although for this experiment fused silica was used as the test substrate. The fused silica has a refractive index of 1.453, while the exposed SU-8 film has a measured refractive index of 1.554 at λ = 975 nm, which along with the water cladding with refractive index of 1.33 provides for significant refractive-index contrast for high confinement and strong evanescent field gradients. The waveguide dimensions were chosen to be a height of 560 nm and a width of 2.8 µm. An SEM of the SU-8 waveguide is shown in Figure 4.
HSQ-Based Fabrication of Magnetic Nanopillars

CNF Project # 111-80
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Abstract

Hydrogen silsesquioxane (HSQ) has been used as e-beam resist and ion milling mask in the fabrication of spin valve devices. The excellent performance of HSQ in patterning nanometer size features and corresponding procedures such as chemical mechanical polishing (CMP) make the HSQ-based process a reliable and economic way to make nanomagnetic devices.

Summary

The recent discovery of spin current induced magnetization switch [1] and oscillation [2] has opened up a new way to manipulate small magnetic structures. Compared with the traditional magnetic field driven dynamics, spin transfer torque has advantages in the preciseness of control and efficiency, and therefore has a large potential in information storage and as microwave devices. Because of the rich science and the promising applications of these phenomena, they have stimulated a great interest in both academia and industry.

Size plays an important role in determining the dynamics of a nanomagnet. Therefore, in order to get better performance out of magnetic structures, it is crucial to be able to fabricate nanometer size devices in a reliable and efficient way. The present process which utilizes poly(methyl methacrylate) (PMMA) as e-beam resist in our lab has been proven to be an effective way in patterning magnetic pillars of nanometer size. However, because of the relative small etch resistance of PMMA under ion milling, extra steps are required that involve the deposition of extra layers that assist the patterning process and the subsequent removal of these layers in order to get effective selectivity in the patterning. Those steps increase the cost and time of the fabrication and more importantly, reduce the yield and make it prohibitive to make complex structures. As a novel e-beam resist, hydrogen silsesquioxane (HSQ) has many advantages over PMMA, including smaller linewidth fluctuations, which provides a better solution for whole wafer processing [3]. Moreover, since HSQ has a much stronger etch resistance in ion milling and reactive-ion-etching, it is especially suitable for the application of magnetic structure patterning [4].

In our experiment, metal multilayers are deposited using magnetron sputtering. An HSQ thin film is then coated onto the metal surface and processed to drive the hydrogen out of the HSQ, forming a rigid Si-O network. In this process development effort, we have been using photolithography
to pattern micro-size ellipses rather than e-beam lithography in order to reduce the cost and for the convenience of characterization. Ion milling is used to define the magnetic micropillars. After proper encapsulation with oxide, a brief CMP is utilized to remove the HSQ mask. The top part of metal multilayer is therefore exposed and provides self-alignment contact with the top leads. Experiment shows that CMP provides a clean surface for top lead deposition.

In summary, HSQ has been utilized in making spin-valve structures to study the effect of spin transfer. Compared with present process, the HSQ based process is more efficient and provides ample room for the improvement of performance. In our future study, e-beam lithography will be used to make practical spin valve or tunnel junction devices.

References


![Figure 3: AFM image of the top part of a magnetic pillar after CMP, showing a shallow hole.](image-url)
Magnetic Vortex Dynamics Driven by DC Spin-Polarized Current

CNF Project # 111-80
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Abstract
We fabricated nanopillar spin-valves with a thick magnetic layer that facilitates the nucleation of a vortex. In the presence of a spin-polarized current, we observe persistent microwave oscillations, which correspond to precession of a magnetic vortex core in the thick magnetic layer, coupled to oscillations of the thinner layer, as indicated by micromagnetic simulations. The frequency varies weakly with the applied field strength and direct current (DC) current amplitude and have also been observed in near-zero applied magnetic field. These self-oscillations exhibit frequency to linewidth ratios of up to ~4000 and linewidths down to ~280 kHz at room temperature.

Summary of Research
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 [6], we use STT to drive oscillations of a non-uniform magnetic structure, a magnetic vortex formed in a nanopillar spin valve.

The samples (Figure 1) were fabricated by depositing (Ta (5 nm)/Cu (2 nm)/CuOₓ (20 nm)) x2/Ta (20 nm)/permalloy (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 × 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 thicker magnetic layer results in a lower self-magnetostatic energy in the vortex state than in the uniform state, thus facilitating the formation of a magnetic vortex. The second magnetic layer is considerably thinner and therefore remains largely uniform. The resulting oscillations of the magnetizations of the Py layers produce a time-varying voltage via the giant magneto-resistance (GMR) effect (Figure 2). Simulations indicate that these oscillations are consistent with the precession of a magnetic vortex in the thick Py layer (gyrotropic vortex mode). The simulations also show that the vortex core undergoes periodic
distortions as it oscillates (Figure 3). We find that the frequency (f) of these oscillations generally increases with increasing perpendicular-to-plane applied magnetic fields and decreases with increasing in-plane fields applied along the major axis of the ellipse. The vortex oscillations can also be observed in near zero applied magnetic field. This differs from oscillations in uniform magnetization spin valves, which require an applied magnetic field of several hundred Gauss or more. The oscillation linewidths (∆f) are typically less than 1 MHz at room temperature. The minimum observed value (∆f~280 kHz, corresponding to f/∆f~4000) is the narrowest linewidth reported to date for a spin torque driven oscillator (Figure 2). Such narrow linewidths suggest that the vortex oscillator is particularly insensitive to noise. This may be due to the larger magnetic volume of the vortex when compared with typical vortex-free devices used in previous experiments, such as Reference 4. Additionally, the vortex might exhibit a weaker coupling to other modes, resulting in less perturbation and hence a more stable frequency. Conversely, a narrow linewidth implies that the vortex oscillator is a sensitive indicator of inhomogeneities in the nanostructure, since small changes in linewidth or frequency can be readily detected. While the integrated power for vortex oscillators is smaller than for the vortex-free spin valves due to the nature of the trajectory, power densities can be as high as 800 times the Johnson noise power densities at room temperature. As the vortex oscillator devices are cooled the integrated power increases and the linewidth decreases to values that can be below 120 kHz.

References


Torsional Oscillator for the Study of the Two Dimensional Superfluid $^3\text{He}$

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Abstract

One of the most important characteristics of the superfluid helium, $^3\text{He}$, is the existence of two phases; the A (axial) and B (isotropic) phases. They have similar free energy and are separated by a surface of first order transition in the space of pressure, temperature and magnetic field. Theoretical work predicts unexpected phase transitions for the two dimensional superfluid $^3\text{He}$ as compared to the transitions in the bulk superfluid $^3\text{He}$ [1, 2]. In order to study the phase transitions in the two dimensional $^3\text{He}$, we have developed a torsional oscillator in which the superfluid helium is constrained to a 500 nm thick layer.

Summary of Research

The superfluid helium is constrained to a thin film in a doughnut shaped cell with a diameter of 1 cm and a depth of 500 nm. In order to create the cell, a 3 µm thermal oxide is grown on a 3 mm thick silicon wafer followed by patterning and etching the oxide. A second thermal oxidation consumes different amounts of silicon in the etched and non-etched regions. The cell is then ultrasonically drilled to create the helium fill-channel and the mounting point for the torsional rod. The wafer is then diced into square pieces with the fill-line in the center, followed by removal of the remaining oxide layer (Figure 1). The final step is to anodically bond the silicon to a 3 mm thick SD2 Hoya glass with the same size as the diced silicon pieces.

The cell is then epoxied to a torsional rod made of coin silver with magnesium wings. The torsional rod has been drilled through in advance to create the fill-line for the cell. The wings are capacitively coupled to the driving and detection electrodes. In order to achieve better thermal conductivity for the assembly, a 2 µm silver film is sputtered on the cell and the epoxy joint. The final assembly of the oscillator is shown on Figure 2.
The quality of the bond between the silicon and the glass has been tested by cooling the whole oscillator to liquid nitrogen temperature and pressurizing it to 20 psi with helium while looking with a leak detector for escaping helium gas. A separate cell with a different geometry (rectangle with width 7 mm and length 10 mm) has been tested at liquid helium temperatures for the quality of the bond. In both cases the cells were leak tight.

Since the distinction between the A and B phases of the superfluid $^3$He is done by measuring the ratio between normal and superfluid fractions in the respective phases, we need to know precisely how much of the normal fraction is coupled to the oscillator. Casey, et al [3], have observed the slip of thin slabs of normal $^3$He in a torsional oscillator. Prior to the measurement of the A and B transitions, we will characterize the slip amount in our cell for different temperatures and pressures.

Acknowledgements

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References

**Weak Coupling of Superfluid Helium Confined to Zero Dimensions**

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**Abstract**

Measurements of the specific heat of liquid helium confined in all three spatial dimensions shows an anomalous behavior that suggests a coupling of individual regions of superfluid. This coupling is thought to occur through the shallow channels used to fill the boxes used as confinement structures. This may be evidence of a proximity effect where helium in the larger boxes modifies the behavior of the helium in the smaller channels and vice versa. Thus, the behavior of both regions is modified beyond typical finite-size effects.

**Summary of Research**

Our group studies finite-size effects in liquid helium near the superfluid transition temperature where critical behavior dominates thermodynamic responses such as the heat capacity [1]. These effects are manifest when an intrinsic length scale in helium, the length over which fluctuations in the system are correlated, approaches the spatial extent of the system. This correlation-length diverges as the temperature approaches the superfluid transition temperature and becomes macroscopic very close to the transition. By confining liquid helium to small homogeneous structures, one can induce finite-size effects in a controllable manner. We use structures in silicon dioxide thermally grown on silicon to confine helium in one, two, or all three spatial dimensions [2]. Scaling theories predict that if one performs measurements on different sized confinements, all with the same geometry, the data will collapse onto a universal curve that is a function of the ratio of the spatial size to the correlation-length.

While seven different measurements of helium confined to a planar geometry [1, 3, 4] where the difference in the small dimension varies by more than a factor of 1200 show remarkable scaling, the data of two different zero-dimensional confinements, where the difference in size of the boxes on each cell is a factor of two, show a lack of scaling [5]. We attribute this behavior to a coupling of the individual boxes of helium through the shallow channels that are used to fill the nearly 100 million boxes that constitute a confinement cell. It is believed the cell with the smaller boxes, where the boxes are spaced closer together, shows a stronger coupling. By performing new experiments we will systematically influence the coupling between individual boxes filled with helium to gain insight into this behavior.

Figure 1 shows a scanning electron microscope image of the boxes used to confine helium in the new experiment.

Figure 1: Boxes used to confine liquid helium in three spatial dimensions.
of tens of nanometers, filling the space above the boxes. The height of these films will be controlled by patterning a series of posts and a border ring in a thin oxide grown upon the second wafer that closes the cell using direct-wafer bonding. A cartoon of this second pattern is seen in Figure 2.

References

Spin-Torque Excitations in Magnetic Nanopillars with an Exchange-Biased Fixed Layer

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Abstract

We have fabricated and are studying iridium manganese (IrMn) / permalloy (Py) / Cu / Py spin-valve nanopillars. We use exchange-bias coupling between IrMn layer and Py to strongly pin the magnetic moment of the lower Py layer and keep its orientation approximately 40° from the easy axis of the other permalloy layer. We have used these samples to measure the bias dependence of the spin transfer torque vector using spin-transfer-driven ferromagnetic resonance (ST-FMR), and found that the torque stays in the plane defined by the two magnetic moments in the bias range $|I| < 2$ mA. We are also studying resonant magnetic switching using the spin-transfer torque from microwave-frequency pulses.

Summary of Research

When current passes perpendicularly through the layers of a spin-valve structure (ferromagnet / metal / ferromagnet), electrons polarized by one magnetic layer (the “fixed layer”) can transfer their spin angular momenta to the other magnetic layer (the “free layer”) and hence exert a torque on the free layer \cite{1}. This effect provides a new way to manipulate small magnets by electrical current rather than magnetic field, and has potential applications in developing non-volatile magnetic memories. For many types of experiments it is convenient to fabricate samples with an offset angle between the magnetic moments of the two magnetic layers in the absence of any magnetic field. This can be achieved by using an anti-ferromagnetic layer to exchange-bias the fixed layer, and pin its moment in the desired direction \cite{2}.

We have fabricated exchange-biased spin valve nanopillars of the structure (in nm): Py 4 / Cu 80 / IrMn 8 / Py 4 / Cu 8 / Py 4 / Cu 2 / Pt 30. The multi-layers are first deposited in a sputtering system and then annealed in a magnetic field to induce an exchange bias between the IrMn and Py layers. Then electron-beam lithography and ion milling are used to define pillars with elliptical cross sections having an aspect ratio $\approx 3:1$ and a short axis diameter between 30 nm and 100 nm. The long axis of the ellipse is oriented so that the easy magnetic axis of the free layer is offset approximately 40° from the exchange-bias direction. The contact leads are made by photolithography, and SiO\textsubscript{2} is deposited to provide electrical insulation between the top and bottom leads.

The samples are initially characterized by measuring the differential resistance, $dV/dI$, as a function of magnetic field at different field angles. By fitting these curves to a macrospin Stoner-Wolfarth model, we can measure the exchange bias strength to be typically 400 G at room temperature, oriented approximately 40° from the easy axis of the magnetic free layer, in agreement with the sample
design. We then used the samples to perform the recently-developed technique of spin-transfer-driven ferromagnetic resonance (ST-FMR), which requires an initial offset angle between the moments in the two magnetic layers. Based on the magnitude and peak shape of the resonance signals, ST-FMR enables measurements of the bias dependences of the strength and direction of the spin-transfer torque on the free layer due to the spin-polarized current. We found that the FMR resonance peaks were symmetric in the bias range of $|I| < 2$ mA, which indicates that the spin-transfer torque is confined strictly in the plane defined by the magnetic moments in the two layers. This is in contrast to the recent observation of an out-of-plane component of torque in MgO magnetic tunnel junctions under finite DC bias [3].

These same samples can also be used in RF-enhanced switching experiments in which an applied microwave signal may resonantly excite the free layer and reduce the critical current for switching. Experiments to explore this effect are underway.

References

Signatures of Spin Transitions in an Endofullerene-Based Single-Molecule Transistor

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Abstract

A single-molecule transistor (SMT) is a three-terminal device which has as its active element a single organic molecule [1]. SMTs allow us to study the quantum-mechanical transport of electrons though single molecules as small as 1 nm. Our previous research on single-molecule transistors has involved work on the coupling of molecular vibrations to electron transport [2], as well as studies of Mn$_{12}$ (acetate) molecules having anisotropic magnetism [3]. In the experiment presented here, we examine SMTs based on a high-spin endofullerene molecule. We then manipulate the electron transport through these devices using an externally-applied magnetic field.

Summary of Research:

We have made single-molecule transistors based on the high-spin endofullerene, N@C$_{60}$, which consists of a magnetic nitrogen atom inside the cage of a C$_{60}$ molecule. We observe two uniquely-magnetic signatures in the electron tunneling spectra of the molecule. First, there is a change in slope of the ground-state conductance peaks when plotted against bias voltage and magnetic field (see Figure 1), which indicates a low-spin-to-high-spin transition in the ground state of the molecule as a function of applied magnetic field. This change in slope is not observed in the tunneling spectra of simple C$_{60}$ molecules (see Figure 2). Second, some excited-state conductance peaks terminate in other excited-state peaks (see Figure 3), as opposed to terminating in ground-state peaks as is observed in non-magnetic spectra.
This behavior is a signature of transitions originating from non-equilibrium spin states of the molecule. While similar changes in spin state are commonly observed in larger quantum-dot devices, the electronic energy-level spacing in small molecules is normally so large as to preclude spin changes at experimentally accessible fields. The spin changes observed in N@C\(_{60}\) are made possible by the combination of the high symmetry of the molecule and the exchange interaction between the electrons on the nitrogen and those on the C\(_{60}\) cage.

We prepared the single-molecule transistors following techniques developed previously in the CNF [2, 3]. We began by fabricating an Al gate electrode 16 nm thick and 2 \(\mu\)m wide and then exposing the Al to air to form a thin insulating oxide. On top of the gate electrode, we fabricated continuous Pt wires with widths of approximately 150 nm and thicknesses of 10 nm. The chips were cleaned with an oxygen plasma and immediately covered with 25 \(\mu\)L of either a 0.1 mM solution of N@C\(_{60}\) for 2.5 minutes or a 0.5 mM solution of C\(_{60}\) in toluene for 1 minute. Then the excess solution was blown off the chip with nitrogen, and the deposition process was repeated. This technique produced a convenient yield of single-molecule devices in the control C\(_{60}\) samples. After the molecules were deposited, we cooled to cryogenic temperatures and broke the wires using electromigration, forming nanometer-scale gaps in which a molecule was sometimes trapped (see Figure 4).

The success rate for observing Coulomb blockade transport characteristics due to the presence of a molecule was 9/19 for N@C\(_{60}\) devices and 17/59 for C\(_{60}\) devices, while 0/39 control devices prepared using pure toluene instead of a fullerene solution showed Coulomb blockade.

References
Polarizability of Quantum-Dot States within a Suspended Carbon Nanotube

CNF Project # 598-96

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Abstract

We have fabricated single-electron-transistor devices made from an individual carbon nanotube suspended between two independent side gates (Figure 1). The nanotube acts as a clean quantum dot, isolated from the disordered substrate. By applying gate voltages, we can populate the dot controllably with any number of electrons, from zero to many tens. By applying different voltages to the two side gates, we measure for the first time the electric polarizability of individual few-electron quantum states in a nanotube, and we observe the effect of electron-electron interactions on this polarizability.

Summary of Research

In the past, a variety of transistor geometries have been developed to study electronic and optical properties of carbon nanotubes. In addition to top gates [1] and buried gates [2], suspended nanotube devices [3, 4] have attracted interest because they eliminate artifacts arising from contact with a disordered substrate and because they enable studies of a nanotube’s mechanical degrees of freedom. Here, we describe the development of a new fabrication scheme which allows complex gating of a suspended nanotube, and its use to measure the electric polarizability of few-electron quantum states.

Fabrication starts with a highly doped silicon-on-insulator substrate. Dry etching of the device layer is employed to pattern electrically isolated gate electrodes spaced 500 nm apart, which are later individually contacted using wire bonds. After isolating the gate electrodes with 100 nm of thermal oxide, all metal electrodes, including bonding pads, are deposited using liftoff techniques before growing the nanotubes. This avoids damage or contamination of the nanotubes by post-growth processing, but requires metals which are compatible with the high temperature chemical vapor deposition (CVD) process. For this reason we contact the gate electrodes using photolithography and titanium/
When measured at zero bias, the curvature of the \(n^{th}\) Coulomb oscillation is a measure of the electric polarizability of the \(n^{th}\) electron groundstate. Unlike single particle levels in a harmonic potential (which all shift in space by an equal amount when applying a constant electric field), we find that an energy level in our quantum dot is less polarizable if other electrons are present on the dot. This indicates the importance of electron-electron interactions in suspended carbon nanotubes, and that screening takes place despite the one-dimensional confinement.

References


Tuning the Kondo Effect with a Mechanically Controllable Break Junction

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Abstract
We have fabricated C$_{60}$-based single-molecule devices with the ability to mechanically adjust the spacing between the source and drain electrodes. We use this device geometry to study the Kondo effect, a many-body phenomenon that can arise from the coupling between a localized spin and a sea of conduction electrons. By varying the electrode spacing, we are able to change both the width and height of the Kondo resonance, indicating modification of the Kondo temperature and the relative strength of coupling to the two electrodes. We are also able to tune finite-bias Kondo features which appear at the energy of the first C$_{60}$ intracage vibrational mode.

Summary
Our device fabrication begins with a growth of 200 nm SiO$_2$ on 200 µm-thick degenerately doped silicon wafers, which are sufficiently flexible to allow for some mechanical bending. We then strip the oxide within a 75 µm × 75 µm square window and grow a thinner 40 nm-thick oxide there. Using a series of photolithography and electron beam lithography steps, we define 32 nm-thick Au lines with a 50 nm constriction centered in the window, connected to thicker Au bonding pads lying on the thicker oxide. Using a combination of dry and wet etches, we then strip the 40 nm oxide from under the Au wires to leave Au bridges suspended 40 nm above the silicon substrate.

To incorporate molecules in our devices, we deposit 25 µL of a 100 µM solution of C$_{60}$ in toluene onto a chip with unbroken wires, wait one minute and blow dry. We then cool the chip to 1.6 K. We use electromigration [1] to create a molecular-scale break in the gold wires before beginning studies as a function of mechanical motion. After electromigration, we find that one or a few molecules can sometimes be found bridging the gap between the electrodes, as inferred from the existence of a Coulomb blockade characteristic in the $I$-$V$ curve. We choose to

Figure 1: Scanning electron micrograph of an Au bridge suspended 40 nm above a Si substrate.

Figure 2: Differential conductance traces for a C$_{60}$ device at various temperatures. Inset: A fit to theory yields a Kondo temperature of 28.2 ± 0.3 K.
study C$_{60}$ molecules because they are sufficiently durable to survive high temperatures present during electromigration and because previous work on single-molecule C$_{60}$ devices has observed the Kondo effect [2, 3].

Measurements on individual C$_{60}$ molecules in our device geometry show signatures of the Kondo effect in a quantum dot, namely a zero-bias peak in $dI/dV$ which is suppressed as a function of increasing temperature. By varying the electrode spacing, we are able to tune both the Kondo temperature and the magnitude of the zero-bias conductance signal associated with the Kondo resonance. These changes allow a determination of how the motion modifies the relative coupling of the molecule to the two electrodes. The normalized linear conductance exhibits scaling behavior as a function of temperature, as predicted by theory.

In addition to a zero-bias peak in $dI/dV$, we have also observed peaks in $dI/dV$ at symmetric values of $V$ near $\pm 33$ mV. The energy of 33 meV is known to correspond to the lowest intracage vibrational mode of isolated C$_{60}$ in which the molecule oscillates between a sphere and a prolate ellipsoid shape [4]. As the electrodes are pulled apart, the positions of the inelastic features increase in $|V|$, suggesting that the mechanical motion increases the energy of the active vibrational mode.

References

Temperature Dependence of Anisotropic Magnetoresistance and Atomic Motion in Ferromagnetic Break Junctions

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Abstract
We fabricate ferromagnetic nanocontacts with cross-sections a few atomic-diameters wide using electromigration. These devices exhibit a large anisotropic magnetoresistance (AMR) signal as a function of the angle of an applied magnetic field. We measure a strong temperature dependence of this effect at cryogenic temperatures, in quantitative agreement with the expectation for a quantum-interference mechanism. In the course of making these measurements, we also observed two-level resistance fluctuations as a function of time, associated with reconfigurations of the atomic structure, which depend sensitively on the magnetic-field angle.

Summary
Recent experiments [1] have found that the anisotropic magnetoresistance (AMR) of nanometer-scale ferromagnetic contacts at low temperature can be much larger than that of bulk samples, and can exhibit more complicated variations as a function of sample bias and the angle of an applied magnetic field than in the bulk case. Here we test a proposal that quantum interference of electrons may explain these results, by measuring the temperature dependence of the AMR signals in nanometer-scale contacts made from permalloy, nickel, and cobalt.

We fabricate our devices by electron beam lithography to define a 100-nm-wide, 30-nm-thick ferromagnetic wire connected to gold contact pads. By using electromigration [2] with active feedback, we form a constriction in the ferromagnetic wire and reduce the contact cross section at 4.2 K while monitoring the resistance. We can reliably achieve nanometer-scale contacts within approximately 10% of a desired resistance value. We then rotate the angle of a large magnetic field in the plane of the sample, and use a lock-in amplifier to measure the differential resistance. We control the measurement temperature by using a resistive heater inside our cryostat.

When any of our devices is narrowed to the point that the resistance is greater than about 1 kΩ, we observe enhanced AMR variations at $T = 4.2$ K as a function of field angle and bias, in agreement with our previous study. When we increase the device temperature from 4.2 K, the AMR variations decrease significantly in amplitude and variations as a function of bias voltage smooth out (Figure 1). The temperature dependence fits well to the convolution of the low-temperature conductance with the derivative of the

Figure 1: AMR of a Ni nanocontact as a function of magnetic field angle. Inset: SEM image of the Ni wire before electromigration.
Fermi distribution at finite temperature (Figure 2). This is the type of strong temperature dependence that is expected for a quantum-interference effect.

In the course of exploring the temperature-dependent transport properties of the ferromagnetic contacts, we also observed abrupt changes in the conductance at particular angles of magnetic field in about 10% of samples (Figure 3). When we set the field angle to values close to the abrupt steps, we observed two-level conductance fluctuations as a function of time due to atomic motion. The duty cycle of the fluctuations changed from 0 to 100% within a small interval of angle (Figure 4). The observation of these time-dependent fluctuations at fixed magnetic-field angle demonstrates that the abrupt jumps in Figure 3 are due to mechanical instabilities, rather than being an intrinsic electronic effect as has been claimed by other groups. We find that mechanical instabilities and two-level conductance fluctuations become increasingly common in all of our contacts above a few 10’s of degrees Kelvin, and in a few samples are present even at 4.2 K. This suggests that magnetic nanocontacts measured at even higher temperatures are likely to be highly dynamic, unstable structures.

References


Linewidths of Spin-Torque-Driven Nanomagnetic Oscillators as a Function of Field Angle

CNF Project # 598-96

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Abstract

In a magnetic multilayer, spin-transfer torque from a spin-polarized DC current can drive magnetic layers into steady-state precessional oscillations [1]. We have investigated how the linewidths of spin-transfer-driven precessional modes depend on the angle of an applied magnetic field. We find that the linewidths can decrease dramatically, by almost a factor of 50, as the field is rotated away from the magnetic easy axis. Spin-transfer-driven ferromagnetic resonance measurements confirm that the same free layer normal mode is excited at different field angles.

Summary of Research

Spin torque from a DC current can generate steady-state magnetic precession in magnetic multilayers, a phenomenon of interest for making tunable nanoscale microwave sources and resonators [1]. We report measurements looking into how the linewidths of these excitations depend on the angle between the applied magnetic field and the magnetic easy axis of the layers (Figure 1). We find that the most-commonly studied field orientation, parallel to the easy axis, produces the broadest linewidths, corresponding to the least-coherent precession (Figure 2). The linewidths can be improved dramatically, decreasing in some cases by approximately a factor of 50, as the field is rotated toward the in-plane hard axis (Figure 3).

Figure 1: Schematic and SEM of a magnetic nanopillar with an elliptical cross-section.

Figure 2: Spectral peaks for DC-current driven resistance oscillations at different field angles.
We have measured two types of multilayer samples: (i) a thin permalloy (Py) free layer with an exchange-biased Py pinned layer (in nm, IrMn 8/Py 4/Cu 6/Py 4) and (ii) a Py free layer with a much thicker Py fixed layer (in nm, Py 20/Cu 6/Py 4). The magnetic layers are 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 70-100 nm. We use photo-lithography to pattern bottom leads and to make top contacts.

Using spin-transfer-driven ferromagnetic resonance (FMR) measurements [2, 3], we are able to identify the DC-driven excitations as due to primarily to motion of the magnetic free-layer (Figure 4). As the field is rotated away from the easy axis, we find that the DC-driven linewidths decrease by a much larger factor in the exchange-biased fixed layer devices compared to the thicker fixed layer devices. Since the free layer in both kinds of devices is made from the same material and has the same thickness, we conclude that the configuration and dynamics of the fixed layer play an important role in the degree of coherence for the free layer. We are investigating the physical origin of this effect using both macrospin models, in which each magnetic layer is approximated as a single magnetic domain, and also micromagnetic simulations which should capture the full dynamics of both layers.

References
Direct Patterning of Polymer Brushes by E-Beam Lithography

CNF Project # 640-97
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Abstract

We have studied patterning polymer brushes directly using electron beam lithography. Usually patterned polymer brushes are fabricated by patterning an initiator layer, followed by the surface initiated polymerization of the desired monomer. However, contamination or loss of activity of the initiator is always a concern. We have grown poly(methyl methacrylate) (PMMA) and poly(2-hydroxyethyl methacrylate) (PHEMA) brushes via atom transfer radical polymerization (ATRP) and successfully patterned them using a 1 nA beam current with doses ranging from 10 to 1500 µC/cm². We have shown that feature sizes close to 20 nm (for PMMA brushes) and close to 50 nm (for PHEMA brushes) can be achieved using e-beam lithography. The sensitivity curves of these brushes have also been studied.

Summary

Patterned polymer brushes have attracted great interest because of the need for systems in the fields of molecular-scale electronics, magnetic storage, biosensing and other areas of nanotechnology [1, 2]. We have studied the possibility of patterning polymer brushes directly using electron beam lithography. Conventionally, patterning of polymer brushes is done by first patterning an initiator layer on the substrate, followed by surface initiated polymerization of a suitable monomer [3, 4]. However, it would be favorable to make this a single step process as this would reduce the possibility of surface contamination. Poly(methyl methacrylate) and poly(2-hydroxyethyl methacrylate) have been used as positive e-beam resists [5]. Hence PMMA and PHEMA brushes were grown via ATRP and then directly patterned using e-beam lithography to create nano-patterned polymer surfaces.

The brushes were patterned using 0.5 nA to 1 nA beam current, with doses ranging from 10 to 1500 µC/cm². Development was done using 1:3 ratio of methyl isobutyl ketone and isopropyl alcohol mixture for 90 seconds. Figure 1 shows an SEM image of a patterned PMMA brush. AFM images of the e-beam patterned PMMA and PHEMA brushes are shown in Figure 2. “Direct” patterning of PHEMA brushes by e-beam lithography was carried out after pre-baking at 160°C for 5 minutes. A 0.5 nA beam current was used with doses ranging from 10 to 120 µC/cm². The patterned brushes were developed in 0.9 NTMAH solution for 60 seconds. We also investigated the sensitivity of these polymer brushes.
Figure 3 shows the sensitivity curves for a 45.3 nm thick PMMA brush and a 50 nm thick PHEMA brush. Current work involves polymerization of a second monomer on these patterned brushes to combine top-down lithography and bottom-up self-assembly to investigate any synergic effects that may lead to periodic structures with features beyond the limits of current lithography.

References

Infrared Systems Progress at CNF; Tunable and Static Infrared Frequency Selective Surfaces

CNF Project # 659-97
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Abstract

Periodic metallic microstructures were fabricated on Kapton sheets and on polyimide membranes. Techniques were developed to maintain flatness of the Kapton during processing while still allowing for flexibility in the final structure. For proper functionality of the structures, it was necessary to first fabricate a continuous metallic film and a continuous dielectric layer on top of the flexible substrate, which underlaid the periodic microstructure. Flexibility of the overall structure was maintained by using a polymer as the dielectric layer, which was constrained to have high optical transmission over the infrared wavelength range of 6 to 14 µm.

Summary

Metallic microstructures have been fabricated for use in infrared (IR) devices and typically have critical dimensions ranging from 0.2 µm to 1.5 µm. In the case of larger critical dimensions, frequency selective surfaces are ideally fabricated using photolithography techniques.

In this project a 10 cm (4 inch) diameter flexible Kapton substrate was fully populated with periodic microstructures. A technique was developed to use an optical adhesive to fix the Kapton to a Si wafer so that it could be held in a flat position during lithography processing. It was necessary for the Kapton to remain flat during all processing steps, so the substrate was re-glued to the Si wafer following each thermal step. Since a typical IR device involves the use of a groundplane and a dielectric layer stacked beneath the microstructured elements, the polymer benzocyclobutene (BCB) was used as this flexible layer. BCB also has the useful property of being transparent in the mid-IR. An alternative method was also developed using liquid polyimide in place of Kapton. Publications related to this research are cited below. A portion of the research was conducted at the CNF with aid from the staff.

References

Figure 1: Polyimide substrate fully populated with metallic periodic microstructures.

Figure 2: Metallic microstructures fabricated on polyimide substrate.
Graphene Nanoribbons from Carbon Nanotubes

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Abstract

We make thin graphene nanoribbons from carbon nanotubes. We do this by cutting open carbon nanotubes with an oxygen plasma. We use the nanotube as a self shadow mask for a protective etch mask layer along one side of the nanotube, and remove the other side. The remaining strip of the carbon nanotube is a ribbon of graphene a few nanometers wide. This fabrication technique lets us study graphene nanoribbons and junctions of graphene nanoribbons with carbon nanotubes.

Summary

Carbon nanotubes, discovered more than a decade ago [1, 2], are straws a few nanometers in diameter made up of carbon in an sp² hybridization. Conceptually, one can think of carbon nanotubes as graphene, individual sheets of graphite, rolled on to itself to close the edges. In practice, nanotubes are grown from catalyst particles as tubes [3]. Recently, there has been an explosion of interest in graphene [4-7]. Our research is focused on opening sections of nanotubes to get back to the graphene sheet, making graphene nanoribbons from carbon nanotubes. See Figure 1. This research also provides a way to make graphene nanoribbon-nanotube junctions.

We start with a doped silicon/silicon dioxide wafer and lithographically define catalyst pads. We deposit iron nanoparticles from solution, and use the rapid heating method [3] to grow our nanotubes from a C₂H₄ and CH₄ feed gas. Electrical contacts of palladium are lithographically defined and deposited at both ends of the nanotube, followed by larger gold contact pads. We then do an initial electrical characterization of the nanotube’s conductance versus back gate voltage.

After the electrical characterization, we cover the nanotube and electrical contacts with photoresist. We expose and develop a few microns of resist along the nanotube between the electrical contacts. A thin layer of aluminum (~ 2 nm) is deposited at an angle (~ 60°) over the entire chip. The nanotube casts a deposition shadow, leaving an exposed leeward part of the tube; see Figure 2. The aluminum is oxidized, and the wafer transferred to a plasma cleaner. A short (~ 6 s) burst of low intensity oxygen plasma (~ 200 pW/µm²) is used to burn away the exposed section of nanotube. The aluminum oxide and photoresist are then

Figure 1: A schematic of a carbon nanotube with localized damage, resulting in a small strip of graphene.

Figure 2: A schematic of a carbon nanotube (black circle) partially covered with aluminum oxide (grey) on a silicon/silicon dioxide wafer (hatched).
removed, and the devices are electrically characterized again. We can tune the parameters on different tubes to see from no change in the conductance to zero conductance (severing the tube completely).

After the localized damage to the nanotube, we use a variety of scan probe techniques to investigate the damaged tubes. Atomic force microscopy shows clear height changes in the section of the device damaged by plasma. In Figure 3, the height goes from 1.6 nm in the undamaged section on the left, to 0.2 nm in the damaged section on the right. We believe this is an example of a graphene nanoribbon fabricated from a carbon nanotube. Electric force microscopy shows that the voltage drop along the device comes in the area where the tube was damaged by plasma. Scan gate microscopy shows the device is most sensitive to a local gate voltage in the region damaged by the plasma. Having developed a method to fabricate graphene nanoribbons, we can now start to study their properties and the properties of nanoribbon-nanotube junctions.

**References**

Optoelectronics and Terahertz Electronic Transport in Individual Carbon Nanotube Devices

CNF Project # 900-00
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Abstract

The excellent electronic and optical properties of carbon nanotubes (NTs) strongly motivate the use of these quasi-one-dimensional (1D) materials in optoelectronic [1] and high frequency electronic [2] applications. By studying individual NT devices, we aim to uncover novel physical phenomena and establish a foundation for future applications in carbon nanoelectronics. Here, we review our recent work that investigates NTs as the active element in photovoltaic devices and microwave mixers in the terahertz frequency regime. We demonstrate that nanotube gated p-n junctions behave as sensitive nanoscale photodetectors and show strong evidence of carrier impact ionization and multiplication. Also, we show that NTs in field effect transistor (FET) geometries can function as gated mixers up to the terahertz frequency regime, and moreover, can detect ultra-short pulses with pico-second width. By studying NTs in gated p-n junction and FET geometries, we demonstrate the practical capabilities of nanotube devices and reveal new physics in these 1D materials.

Summary of Research

Figure 1 shows a schematic of a nanotube gated p-n junction device being illuminated by a focused laser beam. Molybdenum electrodes, deposited on a highly doped silicon substrate with a layer of thermal oxide, form the split gates above which an additional 100 nm Si oxide layer is deposited. Iron nano-particle catalyst sites are defined and NTs (with diameters of 1-3 nm) are grown by chemical vapor deposition. Finally, metallic electrodes (5 nm Cr, 50 nm Au) form the contacts for the NT. After fabrication, the devices are wire bonded and placed in an optical cryostat. Electronic characteristics are monitored while the device is illuminated under focused laser illumination at temperatures down to 4 K.

Current-voltage characteristics are measured while the p-n junction is illuminated at low temperatures. Figure 2 shows I-V_{SD} data at 4 K under illumination at λ = 1560 nm for a NT with 2.0 nm diameter. For the data in Figure 2, each trace was taken with increasing optical power density.

Figure 1: Schematic of gated p-n junction NT device under illumination.

Figure 2: I-V_{SD} characteristics under illumination at 4K.
In forward bias (positive $V_{SD}$), the characteristics intersect at 0.3 V. The reverse bias characteristics demonstrate strong photo-response as the optical power increases. Also, reverse bias conductance ($dI/dV$) (Figure 2 inset) shows peaks occurring in regular intervals, corresponding to the step-like features in the $I-V_{SD}$ data.

We relate the intersection point of the forward bias photocurrent to the flat band condition in the device and infer the band gap, which is in excellent agreement with the band gap determined by thermal activation [3] and diameter measurements. The striking photocurrent steps in reverse bias occur at intervals roughly equal to the band gap. We attribute these steps to impact ionization and carrier multiplication in the junction region of the device. As electrons (holes) gain energy in the built-in electric field, they collide with valence (conduction) band carriers and produce additional carriers, which are subsequently collected at the device contacts. This process results in a photocurrent enhancement when carriers undergo multiplication. Future work will investigate the photon energy dependence of the carrier multiplication and impact ionization processes.

The NT mixing current response to terahertz excitation is shown in Figure 4, which compares the mixing current $I_{mix}$ and the transconductance vs. gate voltage. The mixing current was obtained by measuring the NT device response under terahertz pulse excitation, while the transconductance vs. gate voltage was obtained under DC voltage bias. The two curves show similar behavior over the gate voltage region measured. In Figure 4 (bottom), the NT device current was measured as a function of two-pulse delay time. A large peak with a full width at half maximum of several picoseconds is observed centered at zero pulse delay. The large peak corresponds to the nonlinear mixing current response when two pulses are incident on the device.

The coincidence of the mixing current and transconductance curves in Figure 4 (top) demonstrate that NT FET devices act as mixers up to the terahertz frequency regime. We interpret the large peak in Figure 4 (bottom) as the nonlinear NT response to simultaneous voltage pulses. The narrow peak width (several picoseconds) suggests that NT mixers can detect and resolve ultra-short pulsed excitations. Future studies will investigate coherent excitations in single-walled carbon nanotubes from terahertz pulses.

**References**


Fabrication of Tungsten Step-Wedge Filters for Use in X-Ray Plasma Diagnostics

CNF Project # 1049-02
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Abstract
Calibrated density and structure measurements of tungsten (W) plasmas that develop around exploding wires have been obtained through the use of x-ray radiography and a set of thin W layers that were deposited onto 12.5 µm titanium (Ti) foils. The deposited W varied in thickness in discrete steps ranging from 150 Å to 1.1 µm, creating x-ray filter “step wedges.” The Ti foils with W step-wedges were placed immediately in front of film exposed to a burst of x-ray radiation that passed through the W exploding-wire plasmas. From the exposure level of the film as a function of position, the plasma density profile in two dimensions was obtained by comparing the x-ray attenuation through the exploding wire plasmas with the attenuation through the steps of the step-wedges. An example of such a film is shown in Figure 1, with the images of step-wedges located along the left and right edges of the film and the image of the plasma produced by 10 exploding wires occupying most of the rest of the film.

Summary of Research
Density and structure of dense (> 10¹⁸/cm³), hot (10⁴ - 10⁵ K) plasmas are challenging to measure directly, but are necessary pieces of information in studies of high energy density plasmas. One method of obtaining density profile measurements is through the use of images obtained using a point source of x-ray radiation to expose a piece of film after passing it through the plasma to be measured. In order to be able to convert the x-ray absorption as a function of position on the film into a calibrated plasma density, an adjacent part of the film needs to be exposed to x-rays that have passed through step-absorbers having known thickness and made from the same material as the plasma [1].

In these experiments, carried out in Cornell’s Laboratory of Plasma Studies, a 1MA, 100 ns rise time, 200 ns duration current pulse was used to explode a cylindrical array of ten W wires, each with a diameter of 25 µm. The development of plasma around the exploding wire cores, and the self-magnetic-field-driven implosion of that plasma to the cylindrical axis of the array was studied. The backlighting radiation, taking into account Ti filter and the step-wedges, consisted of 2.5-10 keV photons produced by a molybdenum wire X-pinach [2]. An example radiograph is shown in Figure 1, and a density profile obtained using the step-wedges to calibrate it is shown in Figure 2.

The W step-wedges were created using the CVC Sputter Deposition System at the Cornell NanoScale Science and Technology Facility. Seven layers of thin W were deposited onto 12.5 µm Ti foils. The total deposited W thickness was varied in discrete steps over the range 150 Å to 1.1 µm with variations not exceeding a few percent. Steel masks were employed to ensure deposition at the desired position on the foils.

The quantitative density profiles obtained using the step-wedges are a major portion of the Ph.D. thesis of Jonathan Douglass, who is expected to complete his dissertation during the Fall of 2007 [3].

References
Figure 1: Radiograph of the plasma generated by exploding wires in a wire-array z-pinch experiment initiated from ten 25 µm diameter, 8 mm long W wires in a 12 mm diameter circular configuration. The image, which includes one step-wedge on each side of the array (on the right and left edges of the film), was obtained 140 ns after the start of a 1 MA, 100 ns (zero-to-peak) current pulse that is equally divided among the ten wires. The dark rectangle indicates the location that the density profile shown in Figure 2 was taken from the radiograph.

Figure 2: Calibrated ion density profile from Figure 1. (Notice that absorption of x-rays before reaching the film is proportional to the density of the plasma integrated along the path of the x-rays. As such, the profile is in number per cm$^2$ rather than number per cm$^3$. )
Lack of Superconductivity in Individual End-Bonded Multiwalled Carbon Nanotubes

CNF Project # 1192-04
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Abstract

In 2005, Takesue et al., claimed the finding of high Tc (up to 18K) superconductivity in entirely end-bonded multi-walled carbon nanotubes (MWNTs), which they attributed to the intershell effects of individual MWNTs. To test this claim, we took a novel approach, and fabricated samples with individual end-bonded MWNTs.

Research

The MWNTs we used were grown by a catalyst-free arc-discharged method. High resolution transmission electron microscope (TEM) images showed that typical MWNTs had an outer diameter of 10~15 nm, an inner diameter ~ 2 nm, with few or no visual defects.

Using scanning electron microscope (SEM), we were able to verify the telescoping-antenna-shaped ends of the MWNTs before they were contacted by metal electrodes, proving true end-bonding. Simple transport tests also revealed that the MWNTs were indeed end-bonded to the electrodes.

The temperature-dependent resistance measurement showed that, down to 0.25 K, no superconductivity was found in individual end-bonded MWNTs. To this date, we tested more than six end-bonded MWNTs. So far, none exhibit any sign of superconductivity.

Figure 1: SEM image of a single MWNT end-bonded by 2 electrodes. Originally the MWNT is contacted by 4 side-bonding electrodes (50 nm Ti/Pt). After fabrication, the ends of the MWNT are opened, all walls of the MWNT are contacted by a new layer of metal (30 nm Ti/Pt), and the four side-bonding electrodes are combined into two new end-bonding ones.
**Fabrication of Nanoscale Josephson Junctions for Quantum Coherent Superconducting Circuits**

**CNF Project # 1314-05**

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**Abstract**  
We fabricate nanoscale superconductor tunnel junctions for experiments involving quantum coherent superconducting circuits. 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 that allows for the placement of many qubits on a chip.

**Summary of Research**  
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 × 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). In addition, the SQUID can provide a controllable interaction for entangling two qubits together [3], a necessary ingredient for a quantum computer.

We pattern these circuits at the CNF with 100 nm-scale structures defined with electron-beam lithography integrated with photolithographically-defined large-scale features with the goal to lead to scalable qubit architectures. Our investigations of these circuits 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 [4], 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-AlO$_x$-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 for achieving a temperature of 20 mK.
Physics & Nano-Structure Physics

References


Figure 1, top: 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, middle: SEM image of completed Al-AlO$_x$-Al tunnel junction.

Figure 3, bottom: AFM image of completed Al-AlO$_x$-Al tunnel junction.
Probing Vortex Dynamics in Supercondutors with Nanoscale Weak-Pinning Channels

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Abstract

We are fabricating nanoscale superconducting structures for guiding the motion of vortices. Vortices are quantized bundles of magnetic flux that thread many different superconductors over a particular range of applied magnetic field. By etching asymmetric patterns for the channel walls, we are able to produce a vortex ratchet, which results in the directed motion of vortices when driven with an oscillatory force with zero mean. In addition, we are developing channel structures that may allow us to probe fundamental properties of vortices in superconductors, such as their effective mass and intrinsic damping, and to search for the quantum interference and tunneling of a vortex.

Figure 1: Channel fabrication process with weak-pinning amorphous NbGe layer (200 nm thick) and strong-pinning NbN layer (50 nm thick).

Summary of Research

For technological applications of superconductors in large magnetic fields, controlling the dynamics of magnetic flux vortices that 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. By nanofabricating pinning structures with intentional asymmetries, it is possible to produce classical vortex ratchets that exhibit directed motion of vortices when driven with an oscillatory force with zero mean [1]. Such ratchets may provide controllable model systems for investigating the dynamics of certain molecular motors. 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 vortex using nanofabricated structures, would guide the 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, such as vortex mode locking [2]. Much of the pioneering work was performed at Leiden University in the research group of Peter Kes [3] 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 allows easy motion of vortices in the weak-pinning channels, while the vortices in the strong-pinning banks remain immobilized. In our research program, we are fabricating similar weak-pinning channels but with various asymmetric configurations of the channel walls for producing ratchet potentials. Our measurements of the vortex response indicate a significant rectification of an oscillatory drive due to the ratchet effect [4]. In addition, we are developing channels, combined with novel readout techniques, for guiding vortices around various paths at low temperatures to explore quantum coherent effects [5].

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. We measure these circuits in cryogenic systems that we have constructed in our lab at Syracuse University.

References


Shearing Confined Colloidal Suspensions Using Microfabricated Templates

CNF Project # 1361-05
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Abstract

Microfabricated poly(methyl methacrylate) (PMMA) templates prepared using photolithography and reactive ion etching techniques are used to structure surfaces that shear 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 real-time using confocal microscopy. By applying 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 super-cooled confined colloidal suspensions. The effects of shear on glassy suspensions is monitored in real-time on the particle scale using confocal microscopy.

One defining characteristic of glassy systems is the observation of non-exponential correlation functions using relaxation techniques. There are two dominant models for this behaviour. 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.
Physics & Nano-Structure Physics

References


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

Figure 2: One layer of an FCC colloidal crystal of 1 µm diameter particles.

Figure 3: A random distribution of 1 µm diameter circular holes etched into PMMA.
Josephson Junction Quantum Computing

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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

Porous Thin Films for Supersolid Hydrodynamic Devices

CNF Project # 1458-06
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Abstract

After many years of unsuccessful searches for the self-organized supersolid phase, Kim and Chan [1] reported in 2004 evidence for a new quantum phase of matter in solid helium-4, which is consistent with supersolidity. Recent experiments indicate that disorder in the solid crystal strongly affects the flow: Reppy’s group at Cornell demonstrated the ability to quench the signal by annealing the helium [2], while the Balibar group found that grain boundaries appear to be necessary for flow [3]. Despite these findings, the fundamental nature of the flowing solid, as well as the hydrodynamic details, remain largely unknown.

Summary of Research

We are developing patterned thin porous oxide film devices to study superflow through solid helium microcavities and address these issues. Our design relies on a curious property of superfluid helium in a porous material: it takes extra pressure to solidify. This allows superfluid reservoirs to coexist with solid helium crystals—a situation not found otherwise in stable equilibrium. We will probe helium supercurrents directly, as they interact hydrodynamically with micron-scale solid crystals. This allows us the freedom to investigate directly whether a macroscopic quantum order parameter penetrates solid helium, and its dependence on crystal size and/or boundary defects.

A simple circuit schematic of our device is shown in Figure 1. There are two porous superfluid reservoirs connected via a bulk solid (non-porous) helium crystal. Each porous reservoir also acts as the dielectric region of a parallel-plate capacitor. Due to the polarizability of He-4, the density of fluid on each side of the solid crystal can be driven (or detected) by means of an applied (or measured) electric field. If there exists a hydrodynamic path through the solid helium crystal, then a helium supercurrent can be forced from one reservoir to the other and detected as a density change.

To fabricate the thin porous films, we have integrated a known recipe [4] for creating thin porous dielectrics with electrode metallization and cavity photolithography to develop functional superfluid devices. SEM images of a prototype porous film are shown in Figure 2, indicating roughly 15 nm pores.

Figure 1: Simple circuit schematic of device.
Figure 2: SEM images of a prototype porous film.
Though early devices will employ simple rectangular cavities in which the solid crystal is formed, one of the advantages of thin-film fabrication is that we can control the size and shape of the crystal through photolithography. Future devices can be designed to investigate boundary-induced defects or finite-size hydrodynamics. Figure 3 shows the edge of a prototype (unsealed) solid cavity boundary, where it meets the superfluid detector reservoir.

We are currently testing a calibration device on board our dilution refrigerator, which includes a porous dielectric superfluid reservoir but no patterned solid crystal. We have detected the bulk helium flow into the device and are currently searching for superfluid sound modes, with the goal of probing their interaction with the bulk solid bath to find the suppressed solidification curve.

References

A Tunable DNA Spring in a Nanochannel

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Abstract
Nanofluidic devices based on nanochannels are being developed for deoxyribonucleic acid (DNA) analysis. In order to optimize design strategies for active plumbing elements, we have investigated the behavior of nanoconfined DNA under a.c. electric fields, and found that a phase transition can be induced, through which the elastic properties of the confinement-stretched polymer change radically.

Summary of Research
In recent years, the fabrication of large-area nanofluidic systems for the analysis of DNA and DNA-protein complexes has become technologically feasible through continued improvement of electron-beam lithography systems [1, 2]. A particularly striking transition in the behavior of DNA can be achieved by confining single molecules to channels that have a cross-section that is comparable or smaller than twice the persistence length of double stranded DNA (dsDNA), and are hundreds of microns long. In those channels, DNA becomes linearized, and stretched out to a constant fraction of its contour length, thus delivering a direct connection between a physical measurement and the abstract picture of the linear genetic information that can be found in any molecular biology textbook [1].

For complex, multi-step analyses, we are developing integrated nanofluidic elements that resemble classical plumbing items such as mixers, valves, switches etc. (Figures 1, 2). These will not function via liquid manipulation using moving parts or deformable materials, as in microfluidics, but will be based on selective transport of polyelectrolytes (such as DNA) in electric fields. The new functional elements rely on carefully tailored nanofluidic volumes and a.c. electric fields. The basic function has recently been demonstrated in the form of a nanofluidic meta-material whose transport properties can be switched by applying an a.c. electric field [3]. The central idea is the balancing of entropic, mechanical, and dielectrophoretic contributions to the energy landscape.

Figure 1: Example of nanofluidic channel network in fused silica.

Figure 2: Cross-section through a junction of two nanochannels.
In order to design such structures successfully, we have investigated the basic behavior of polyelectrolytes, and in particular DNA, under strong a.c. fields, and have surprisingly found giant electrostriction of single, nanochannel-stretched molecules. The basic experiment is shown in Figure 3. On the left of the figure are individual images of DNA in the nanochannel, visualized by fluorescence microscopy, and on the left is a time trace which records the intensity along the polymer backbone throughout the experiment. After about one fifth of the experiment, an a.c. voltage was applied. If the a.c. field was above a certain threshold voltage, the stretched DNA collapsed. Close to the threshold voltage, an increased noise reminiscent of a phase transition was observed. At this point, we do not have a clear understanding of the collapsed state of DNA. However, we believe that the striking result holds in a much more general sense, and that stretched polyelectrolytes might be used as artificial muscles.

The underlying physical mechanism for the collapse can be described either on a microscopic basis, or globally via macroscopic parameters. In the microscopic picture, DNA strands are transversely polarized, and induced dipoles then exert a mutual attraction. The latter can be used to formulate a modified second virial coefficient, and a transition field strength for a vanishing second virial coefficient can be predicted. Since the DNA is stretched to 70% of its contour length in our channels mainly due to excluded volume effects, a contraction of the macromolecule follows. An alternative description follows the classical theory of electrostriction. The electrostrictive pressure is simply twice the electrostatic energy density, which in turn is proportional to the dielectric constant. Since the dielectric constant is particularly high for hydrated polyelectrolytes, and the spring constant rather low, the resulting electrostriction is giant, or about 70% in our case. That is probably one of the most extreme mechanical reactions of any material to the presence of an electric field.

References


Fabricating Nanoscale Aperture Arrays as Superfluid $^4$He Weak Links

CNF Project # 1494-06

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Abstract

Arrays of nanoscale apertures have been discovered to exhibit the characteristic signatures of so-called Josephson weak links in superfluid helium-4, $^4$He [1]. These devices can be used to detect quantum phase differences in a matter wave interferometer [2]. Thin (~ 60 nm) silicon nitride membranes were made by photolithography on silicon wafers and several varieties of aperture arrays shot on these by electron beam lithography. Using these arrays, we experimentally verified the relationship between superfluid velocity and the phase gradient in the macroscopic wavefunction [4] and linearized our interferometer [5].

We also found evidence for the possible detection of individual vortex lines [6].

Summary of Research

I. Physics behind the Experiment:

The superfluid Josephson effect refers to the strange phenomenon that occurs when two volumes of a quantum coherent fluid are connected by a passage (called a weak link) whose dimensions are a few tens of nanometers. If a pressure differential is applied across the apertures to force the liquid from one side to the other, the resultant motion is a flow oscillation at the “Josephson frequency”, which increases in direct proportion to the applied pressure head. Rather than pass through the aperture like an ordinary fluid subjected to a pressure head, the average superfluid current here is zero! This so-called “quantum whistle” was discovered in our laboratory in 2005 [1]. A “weak link” is analogous to a coherent source of light in optics. Just as two coherent light waves characterized by a phase difference interfere with each other to produce an optical interference pattern, superfluid matter-waves (described by macroscopic wavefunctions) can interfere to produce an acoustic interference pattern that can be detected (and heard) using a microphone. An arrangement with two weak-links in a loop (see Figure 1) therefore behaves as an interferometer (analogous to the superconducting dc-SQUID) where the resultant amplitude of current oscillations varies nonlinearly with the difference in phase drops across the weak-links [2]. The phase difference can be caused by (among other things) the Sagnac
effect [3] (due to rotation of the loop) or by an artificially induced superfluid current, which translates to a phase gradient for the superfluid wavefunction (see Figure 2). The latter was explicitly demonstrated by our group in 2007 using the aperture arrays we recently fabricated at CNF and by creating a superfluid counterflow using an injected heat current [4]. The same apparatus was used to linearize our interferometer using the heat current counterflow for negative feedback [5]. In the course of the experiment, we also found evidence suggesting that we had observed the motion of individual vortices in superfluid helium-4. This is still under active investigation in our group but preliminary results may be published soon [6].

II. Fabrication Details:

Very thin (~ 60 nm) freestanding membranes of silicon nitride are created on standard DSP silicon wafers using photolithography. 100 × 100 square arrays of apertures with 3 µm separation whose nominal size is ~70 nm (see Figures 3 and 4) are then shot through these membranes using electron beam lithography. The wafer is broken into individual chips, each of which acts as a “weak link” in our experiments. Varying sizes of arrays with different aperture sizes have been fabricated for future experiments. A particularly curious set of arrays was recently made where the apertures were positioned pseudo-randomly on the membrane. It is hoped that these chips may further help to test some recent theoretical predictions about this class of experiment [7].

References

Superconducting Resonator Circuits

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Abstract

With the promise of applications in quantum computing, solid state electronic circuits with quantum mechanical properties have been proposed, developed, and investigated in recent years. The aim of our work is to replace the Josephson tunnel junction in such circuits by devices in which carbon nanotubes play the role of the tunnel barrier.

Summary of Research

Our research focuses on electrical circuits whose macroscopic behavior exhibits quantum mechanical properties. All these circuits are made of superconducting material and involve one or more Josephson tunnel junctions. Examples include solid-state qubits [1] and quantum-limited amplifiers, with applications in quantum computation. Several recent experiments showed that carbon nanotubes (CNTs) can support a proximity-induced supercurrent [2, 3], and we are planning to incorporate CNTs into our quantum circuits.

Given the unique properties of CNTs [4], such CNT quantum circuits are attractive from multiple points of view, for example: (i) CNTs have large inductances [5], making them interesting circuit elements, (ii) the non-linear current-phase relationship between a proximity-induced supercurrent in the CNT and the difference of superconducting phase between its metal contacts allows CNTs to act like Josephson junctions [3], and (iii) given their small diameter (of a few nanometers) CNTs are an ideal interface to the molecular world [3].

To meet some specific challenges in making CNT devices, we have recently started using the CNF with its extensive nanofabrication capabilities. Our fabrication protocol starts by exposing local, chip-level, and global alignment marks on a 4-inch Si wafer. These marks are etched into the substrate so that they survive the high furnace temperatures during CNT growth. Catalyst for CNT growth is deposited at small islands on the substrate in a lift-off process. Our CNTs are grown by chemical vapor deposition in Paul McEuen’s lab at Cornell. Following growth, we image the CNTs by atomic force microscopy and design contact electrodes for each CNT device. We have made CNT devices with good contacts, and we have developed the process to incorporate them into quantum circuit geometries.

References

Fabrication of Cantilevers with Integrated Mesoscopic Samples

CNF Project # 1527-07
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Abstract

A persistent current $I_p$ in a normal metallic ring is a mesoscopic phenomenon arising from interference of phase coherent electrons circumnavigating a ring threaded by a magnetic flux [1]. The aim of this project is to study persistent currents in normal metal rings using a cantilever as a torsional magnetometer [2]. We have fabricated high sensitivity cantilevers with integrated metal rings. At $T = 300$ mK, the force sensitivity of the cantilevers is demonstrated to be 1.7 aN/√Hz. The phase coherence length $\ell_\phi$ of electrons in co-deposited films is measured to be 3 µm. With these results, we expect to have the sensitivity to measure persistent currents in normal metal rings.

Summary of Research

When a ring is integrated onto the end of a cantilever, its persistent current produces a torque on the cantilever

$$\tau = \pi r I_p B_\perp$$

corresponding to a force

$$F = \pi r^2 I_p B_\perp$$

where $r$ is the radius of the ring, $\ell$ is the length of the cantilever, and $B_\perp$ is the component of the applied magnetic field in the plane of the ring. For a 1 µm radius ring, the expected values of $I_p$ are $\sim 100$ pA, which will produce a force of $\sim$ 10 aN on a 350 µm long cantilever.

Our ability to measure $I_p$ depends crucially upon the fabrication of both the cantilever and the ring. Our primary concerns are:

1) High force sensitivity of the cantilever. The force sensitivity of a cantilever is given by:

$$F_{\text{rms}} = \sqrt{\frac{4kT_n k}{\omega_0 Q}}$$

where $T_n$ is the noise temperature of the cantilever, $\omega_0$ is the resonant frequency, $k$ is the cantilever spring constant, and $Q$ is the cantilever’s quality factor.

2) Long electron phase coherence length $\ell_\phi$ (longer than the ring circumference) and long electron mean-free path $\ell_e$ (as long as possible). These conditions can be satisfied in micron-diameter rings with smooth surfaces and very high chemical purity.

Figure 1: SEM of fabricated silicon cantilever. Scale bar is 100 µm. Inset shows a detail of the Al ring at the end of the cantilever. Inset scale bar is 400 nm.

We fashion our cantilevers from the 300 nm thick device layer of a high-resistivity silicon-on-insulator wafer. In the first step, an array of cantilever dies (each containing 12 cantilevers) is patterned on the wafer with photolithography (CNF HTG System II-Contact Aligner) and a reactive ion etch (RIE) (CNF Oxford PlasmaLab 80+ RIE System). We next use electron beam lithography (CNF JEOL 9300) to define rings at the end of the cantilevers. To minimize the presence of magnetic impurities in the deposited rings, the metal evaporation is performed at Yale University in an evaporator dedicated to non-magnetic materials. The cantilevers are freed by a back side photolithography step which includes a deep RIE (CNF Unaxis 770) of the backside silicon and an oxide etch of the buried oxide layer. The final step is a critical point dry (CNF Critical Point Dryer).

Rob Ilic, a research staff member at the CNF, was greatly instrumental in the success of this project.
Cantilevers, such as the one shown in Figure 1 have been fabricated at the Cornell NanoScale Facility. The power spectral density of the cantilever’s motion at 300 mK is shown in Figure 2. The cantilever’s $Q = 127,000$, $k = 4 \times 10^{-4} \text{ N/m}$, indicating a force sensitivity of $1.6 \text{ aN/Hz}$ at 300 mK. This corresponds to a current sensitivity of $20 \text{ pA/Hz}$ at $B = 9 \text{T}$; hence we expect to be able to measure persistent currents with cantilevers such as these [3].

To characterize $\ell_\phi$ of the deposited films, we performed weak-localization (WL) measurements [4] on co-deposited aluminum meanders, such as the one shown in the inset of Figure 3. The meanders are 200 µm long and have the same width (80 nm) and thickness (80 nm) as the rings. A representative WL measurement is shown in Figure 3. The fractional change in resistance of the meander as a function of applied magnetic field is plotted. WL theory allows us to extract an $\ell_\phi$ of $3 \mu\text{m}$ at 2.2 K. This is a promising result for the observation of a persistent current.

We have measured the magnetic response of the aluminum ring in Figure 1 in its superconducting state. The plot of cantilever $\omega_0$ vs. $B$ in Figure 4 shows sawtooth jumps in $\omega_0$ due to flux quantization [5]. In the very near future (after a few minor modifications to the refrigerator), we will attempt to measure persistent currents in normal state aluminum rings.

References


Robust Shadow-Mask Evaporation Through Lithographically Defined Undercut in PMMA/PMGI Resist Bilayer

CNF Project # 111-80
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Abstract
We have developed a new recipe for fabricating a robust two-layer resist stack for shadow-mask evaporation. Instead of using the conventional low/high molecule weight PMMA or P(MMA-MAA)copolymer, we used PMGI-9F lift-off resist as the bottom layer of the resist stack with 950k PMMA as the top layer. The dissolution rate of PMGI in base solution is characterized, and can increase nearly three times with electron beam exposure.

Summary of Research
Lateral spin-valve composed of ferromagnet/non-magnetic metal or semiconductor junctions is a simple structure to study the injection, propagation and detection of spin polarization of electrons. Minimal interfacial oxidation between the ferromagnet (FM) and the non-magnetic metal (NM) is crucial for high spin-injection and detection efficiency. Shadow-mask evaporation is a simple and robust method for fabricating high-quality lateral spin-valve structures since evaporation of the entire structure is done without breaking the vacuum, leaving the FM/NM interface intact. This process entails using e-beam lithography to fabricate an undercut structure on the double-layer resist to form a shadow-mask, then evaporate different materials at different angles to form the desired junction structure, and last lift-off the resist. Conventionally, this is done by using low-sensitivity e-beam resist, usually the large molecular weight PMMA 950k on top, and high-sensitivity e-beam resist such as P(MMA-MAA)copolymer at the bottom. However, successful fabrication of a shadow-mask structure greatly benefits from exposure of the bottom layer by scattered electrons off the original beam direction, commonly seen in 30 keV e-beam lithography. However, our 100 keV e-beam lithography at CNF greatly reduced such scattered electrons and made fabricating this type of shadow-mask much more difficult. Moreover, since both PMMA and copolymer are developed using the same developer, typically MIBK:IPA or IPA:water, it becomes very challenging to create features smaller than 100 nm on PMMA and a large enough undercut—larger than 800 nm on copolymer.

After trying with PMMA/copolymer unsuccessfully, we have developed a new recipe using poly(methylglutarimide) (PMGI) from MicroChem. The biggest advantage of PMGI over a copolymer is that it can be developed in an aqueous base solution, but does not dissolve in MIBK or IPA. Therefore, the development of PMMA and PMGI can be separately timed and controlled. By using PMMA of larger concentration in MIBK solution, the collapsing PMMA suspended bridge can be solved. Moreover, the dissolution rate of PMGI in base solution can be controlled by e-beam exposure. This provides great flexibility in the shadow-mask design to create the desired structure.

Figure 1. Scanning electron micrograph (SEM) cross-section of 300-nm-wide lines on PMMA undercut by 950 nm on PMGI.
By cross-sectional SEM of the resist stack, we are able to measure the undercut as a function of the e-beam exposure in the center of the strip to define the feature on PMMA, and exposure on the side to modify the dissolution rate of PMGI. We found that an undercut as large as 1 µm can be defined and the dissolution rate can be changed by nearly a factor of three with e-beam exposure without affecting the top layer.

Currently in progress is the design of the e-beam exposure pattern to create a shadow-mask for evaporating the entire lateral spin-valve structure in a single pump-down. Using the structure, we will study the spin-transfer torque by pure spin-current, intrinsic vs. extrinsic spin Hall effect, etc.

References
Ultrasound Enhanced Electrostatic Batch Assembly of Microstructures

CNF Project # 1121-03
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Abstract

The assembly of released parts of microsystems is the last, but a very crucial, step that often requires tradeoffs in the yield and performance of the whole system. Various applications with different concerns have triggered some of the assembly methods today available. Methods that make use of surface tension forces [1], magnetic forces [2], centrifugal forces [3], on chip actuators and robotic assembly with micro-grippers [4] are some examples. A recent assembly method developed in our group is thermokinetic actuation which exploits forces due to the interplay between temperature and pressure of the gas molecules which surround the microstructures as well as ultrasonic actuation for stiction reduction [5].

Introduction

The method we present inherits the simplicity of electrostatic actuation and unique attributes of ultrasound like reducing stiction and providing lubrication during the course of the assembly. The apparatus for the assembly is shown in Figure 1. Here, a DC voltage is applied to an ITO coated glass slide which acts as a global, top electrode. Initially uniform electric field due to this voltage works to pull the structures away from the substrate. At the same time, the bulk PZT (lead zirconium titanate) ceramic plate ultrasonically actuates the MEMS die from bottom, via vacuum-suction coupling through a custom made chuck. The whole assembly setup is heated to reduce humidity-related stiction. DC voltages on the order of kilovolts are used in order to accommodate large electrostatic gaps and to enable the assembly of large structures. The PZT bar is excited via a harmonic voltage source, the frequency of which is also swept. Peak to peak amplitude is usually kept below 40V.

All the samples that we present results for are fabricated via PolyMUMPS™ [6], while wet release, critical point drying and microscopy are performed in Cornell NanoScale Facility. Figure 2 shows the assembly results of

Figure 1: Sketch of the experimental setup for the assembly process. The die has a hinged paddle and a paddle supported by torsional beams, both of which are common in assembled systems.

Figure 2: Assembly of 5 × 8 and 8 × 8 arrays of hinged structures, which are made of 80 × 280 µm² (left) and 80 × 180 µm² (right) paddles. Yield of the array on the left is 95% while, that of the one on the left is 100%.
The results of our experiments quantitatively demonstrate the effect of ultrasound on anti-stiction. Moreover, the presented assembly method, which merges ultrasound and electrostatic actuation, is applicable to many processes without any extra layer or post-processing step. It lets the assembly of microsystems in a simple and quick manner without any special packaging, probing or wire-bonding. The maximum force is limited with the breakdown of air [7], and this should be considered in the design phase. We were able to assemble 8 × 8 arrays of hinged structures with yields of up to 100%. The future work is on progress to use the method in the assembly of more complex structures used in optical systems on chip.

Acknowledgments

We would like to thank David Reyes and Robert Schildkraut of Block MEMS LLC for helpful discussions. This work was funded by DARPA and NSF.

References


Summary

During a typical assembly experiment, first the DC voltage is applied with ultrasound turned off. Then at some point before the breakdown of air, ultrasound is turned on while DC voltage is kept constant. Figure 4 plots the ratio of the assembled flaps in an array of 64 as a function of the DC voltage for 2 different samples. The main premise of this plot is the large jump in the number of flaps for both of the samples as soon as the ultrasound is turned on at 3000V. This shows the effectiveness of ultrasound as an anti-stiction tool when electric field remains useless even close to the breakdown.

Figure 4: Percent number of assembled flaps as a function of DC Voltage for 2 dies. For $V_{dc} < 3000$V ultrasound is turned off. After the ultrasound is turned on at this voltage, the ratio of the assembled flaps comes close to 100%, as highlighted in the figure.
Fabrication and Surface Chemical Modification of Support Structures for Synthesis of Self-Assembled Bioinspired Nanoarchitectures

CNF Project # 1306-05
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Abstract
Bionanofabrication—a process that takes advantage of the structural specificity of biological systems to create various types of micro/nanostructures—allows parallel fabrication of extremely small feature sizes (< 50 nm) with controlled diameters and without the slow throughput of conventional ion/e-beam lithography and the high cost of x-ray lithography. Two dimensional surface layer (S-layer) protein lattices were employed in the fabrication of metallic nanoparticle arrays (NPs). Nickel films and gold NPs were further used as catalysts for the templated growth of carbon nanotubes and semiconducting nanowires. Our results show the feasibility of S-layer technology for the nanofabrication of device components.

Summary
We previously established the ability of S-layer proteins to act as biotemplates for patterning metallic and semiconducting nanoparticles (NPs) as well as quantum dots (QDs). Our results demonstrated that it is possible to exploit the physico-chemical/structural diversity of prokaryotic S-layer scaffolds to vary the morphological patterning of nanoparticles of different surface functionalities, providing us with a very diverse array of nanostructures. It was interesting to further expand our research in the formation of self-assembled two-dimensional (2-D) arrays of dendrimer-encapsulated platinum nanoparticles (Pt-DENs) using prokaryotic S-layer proteins as biomacromolecular templates.

We synthesized Pt-DENs (mean core diameter 1.8 ± 0.5 nm) by chemical reduction of metal ion species complexed within the interior of fourth-generation, hydroxyl-terminated, starburst poly(amidoamine) dendrimers (G4 PAMAM-OH). Detailed structural and elemental composition analyses were performed using high-resolution transmission electron microscopy, energy-dispersive x-ray spectroscopy, electron energy loss spectroscopy, and x-ray photoelectron spectroscopy (XPS), showing that at least some quantity of the platinum found within the particles is present in the expected zerovalent state.

By using the S-layer lattices from the acidothermophilic archaeon Sulfolobus acidocaldarius (SAS) and the Gram-positive bacterium Deinococcus radiodurans (HPI) as a biotemplate, hexagonal- and honeycomb-ordered arrays of the Pt-DENs were successfully fabricated under a range of different pH conditions via non-covalent nanoparticle-protein interactions. Fast Fourier transform analyses of transmission electron microscopy images verified that the fabricated Pt-DEN assemblies displayed mean periodicities that corresponded well with the lattice constants of the native protein templates (i.e., 22 and 18 nm for S. acidocaldarius and D. radiodurans S layers, respectively).

These results demonstrate that utilizing pre-synthesized Pt-DENs in conjunction with microbial S-layer proteins...
displaying highly periodic topochemical properties can be an effective, novel route for creating patterned arrays of Pt NPs with potential technological applications.

To further demonstrate the potential application range of the S-layer templates, we proceeded in designing and fabricating bottom-up structures. The previously described arrays of NPs, as well as thin films patterned using S-layer as masks, were used as catalysts for the growth of nanowires/nanotubes hence reporting for the first time such advanced processing of these bio-templated nanostructures.

Our initial attempts involved growth of carbon nanotubes based on S-layer templated nickel films. Both SAS and HPI S-layers were examined as potential masks for the templated growth of CNTs. A thin film of nickel (2 nm) was e-beam evaporated on a Si/SiO$_2$ chip with or without adsorbed S-layer patches. The organic protein layer was removed by plasma etching, for 2 min, using an oxygen reactive ion etching process. Finally the chip was transferred to the chemical vapor deposition (CVD) chamber for carbon nanotube growth using ethylene as the carbon source (6 sccm ethylene, 750°C, 10 min growth time).

Overall, optimization of the growth procedure is highly desirable not only to increase the yield of growth but also to successfully transfer the pattern features of the S-layer to the final CNT array. Lower growth temperatures and possibly the use of plasma enhanced-CVD are expected to lead to better results.

Au NP arrays are well known to act as catalysts for the growth of nanowires [2]. Therefore, in collaboration with Los Alamos National Laboratory, we examined the possibility of S-layer templated Au NP arrays to nucleate growth of nanowires. Indeed, semiconducting nanowires (SCNWs) of germanium and germanium/silicon alloys were successfully grown via a vapor-liquid-solid mechanism, using chemical vapor deposition. HPI S-layers were employed in the fabrication of Au NPs arrays for the spatially controlled growth of SCNWs. Under the vapor deposition conditions used, neither the Au NP diameter, nor the protein organic layer and native oxide were limiting factors for nucleation. A density of 0.14 NW/µm$^2$ GeNWs with an average diameter of ~ 153 nm was achieved for NWs grown on non-patterned Au NPs substrates. On the other hand, GeNWs grown from biotemplated Au NPs exhibited higher densities (25 ± 4 NWs/µm$^2$) and smaller diameters (< 21 nm). Optimization of the CVD parameters and crystallographic substrate orientations is underway to achieve better pattern transfer of the highly-order S-layer-patterned catalysts into that of the synthesized NW arrays. We envision that vertical growth will allow the three dimensional integration of more complex structures such as room temperature ultraviolet NW nanolasers and vertical field-effect-transistor arrays.

References

The CNT growth was examined under a scanning electron microscope SEM (Figure 2). In both S-layer samples, the observed growth yield was lower than the control samples or areas where no S-layer fragments were present. This observation points to the fact that the Ni particles templated through the S-layer are very closely spaced and aggregate during the high temperature processing. Larger particles are then not able to nucleate CNT growth [1]. Between the two distinct S-layers, SAS allowed CNT growth to a higher yield than the HPI. This is attributed to the less dense lateral spacing provided by SAS compared to the closely packed HPI S-layer.

Figure 2: SEM images of carbon nanotubes grown on; A) SAS and B) HPI S-layers biotemplated Ni. Images were taken at 3 kV accelerating voltage. Scale bar = 1 µm.

The growth was examined under a scanning electron microscope SEM (Figure 3). In both S-layer samples, the observed growth yield was lower than the control samples or areas where no S-layer fragments were present. This observation points to the fact that the Ni particles templated through the S-layer are very closely spaced and aggregate during the high temperature processing. Larger particles are then not able to nucleate CNT growth [1]. Between the two distinct S-layers, SAS allowed CNT growth to a higher yield than the HPI. This is attributed to the less dense lateral spacing provided by SAS compared to the closely packed HPI S-layer.
PDMS Microchambers for Single-Nanoparticle Catalytic Studies

CNF Project # 1512-06
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Abstract

To better understand the activity of nanoparticle catalysts, we endeavor to study in real-time the catalytic reactions of Au nanoparticles on a single nanoparticle basis through fluorescence microscopy techniques. Our experimental strategy centers on a fluorogenic probe reaction catalyzed by Au nanoparticles, and the use of microchambers to both contain the fluorescent product for detection and isolate individual nanoparticles from one another.

Summary of Research

We are using the facilities at the CNF to make the polydimethylsiloxane (PDMS)-based microchambers, using silicon wafer molding techniques. We have fabricated arrays of cylinders onto silicon wafers using photolithography, molecular vapor deposition, and reactive ion etching. The cylinders were spaced about 10 µm apart with a 5 µm height, a 4.5 µm diameter base, and a 3.5 µm diameter top. They were arranged in a square array containing 360,000 cylinders (Figure 1). We used the wafer mold to generate PDMS stamps containing the microchambers.

These microchambers are easily observable through an optical microscope (Figure 2) and can encapsulate solutions of a fluorescent dye (Figure 3). We are currently using the described setup to study the reactions of individual Au nanoparticles.

Figure 1: SEM image of an array of silicon columns.

Figure 2: Bright field image depicting microchamber arrays in PDMS.

Figure 3: Array of fluorescent spots formed from PDMS microchambers.
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