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

The National Science Foundation, the New York State Office of Science, Technology and Academic Research, Cornell University, Industry, and our Users.

The 2011-2012 CNF Research Accomplishments are also available on the web in secure PDF at:

http://www.cnf.cornell.edu/cnf_2012cnfra.html

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Table of Contents

2011-2012 CNF Research Accomplishments Reports, by Section ... ... ... ... .ii-ix
2011-2012 CNF Research Accomplishments Introduction ... ... ... ... ... x-xi
2011-2012 CNF-Research Related Patents, Presentations, & Publications ... xii-xxvi
Commonly Used Abbreviations & Their Meanings ... ... ... ... ... ... ... xxvii-xxx
2011-2012 CNF Research Accomplishments Reports ... ... ... ... ... ... ... ... 2-247
Index of Reports by CNF Project Number ... ... ... ... ... ... ... ... ... ... 249
Index of Principal Investigators and Users ... ... ... ... ... ... ... ... ... ... 250-252

We Wish to Thank our 35th Anniversary Corporate Sponsors:
**BIOLOGICAL APPLICATIONS,**
**PAGES 2-57**

<table>
<thead>
<tr>
<th>Title</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>Construction of Synthetic Ecologies</td>
<td>2</td>
</tr>
<tr>
<td>Retinal Implant Project</td>
<td>4</td>
</tr>
<tr>
<td>A Microfluidic Device for Structural Studies of Nucleic Acids</td>
<td>6</td>
</tr>
<tr>
<td>Microfluidic Cell Culture Analog Devices to Mimic Animal Exposures to Toxins and Drugs</td>
<td>8</td>
</tr>
<tr>
<td>Microchip Devices for the Study of Single Vesicle Fusion Events</td>
<td>10</td>
</tr>
<tr>
<td>Mimicry of Biological Adhesion Through Fabrication of Fibrillar Surfaces</td>
<td>12</td>
</tr>
<tr>
<td>Silicon Nitride Cantilevers for Muscle Sarcomere Force Measurements</td>
<td>14</td>
</tr>
<tr>
<td>Microfluidic 3D Hydrodynamic Flow Focusing for the Rapid Protein Concentration Analysis</td>
<td>16</td>
</tr>
<tr>
<td>Role of Tissue Factor and Tissue Factor Pathway Inhibitor Complex in Cancer Cell Adhesion</td>
<td>18</td>
</tr>
<tr>
<td>Biomimetic Blood Vessel Model to Elucidate Physicochemical Mediators of Tumor Angiogenesis</td>
<td>20</td>
</tr>
<tr>
<td>Micro-Patterned Surfaces for the Study of Signaling, Trafficking, and the Lysosomal Synapse</td>
<td>22</td>
</tr>
<tr>
<td>Confined Microfluidic Environments for Studying Cell Mechanics &amp; Cancer Invasion</td>
<td>24</td>
</tr>
<tr>
<td>Fabrication of Microfluidic Devices to Study the Fusion Kinetics of Influenza Virus to Biomimetic Membranes</td>
<td>26</td>
</tr>
<tr>
<td>Separation and Identification of Raft Associated Membrane Species Using a Patterned Supported Lipid Bilayer Extractor</td>
<td>28</td>
</tr>
<tr>
<td>Image-Based Actuation and Valving of Flows Inside Microfluidic Channels</td>
<td>30</td>
</tr>
<tr>
<td>Suspended Carbon Nanotube Devices for Single Molecule Sensing</td>
<td>32</td>
</tr>
<tr>
<td>X-Ray Lab-on-a-Chip: Sample Dialysis for Small-Angle X-Ray Solution Scattering</td>
<td>34</td>
</tr>
<tr>
<td>Therapeutic and Diagnostic Applications of Halloysite Nanotubes</td>
<td>36</td>
</tr>
<tr>
<td>A Novel Application of Micro Fluidic Structures in Clinical Diagnostics</td>
<td>38</td>
</tr>
<tr>
<td>Effect of Substratum Nanoscale Topography on Bacterial Attachment and Biofilm Formation</td>
<td>40</td>
</tr>
</tbody>
</table>
## TABLE of CONTENTS

### Biological Applications, Continued

- Revealing Responsive and Stochastic Switching in Bacteria .......................................................... 42
- Models of Microvascular Networks ........................................................................................................ 44
- Confinement of Collagen Gels in Three-Dimensional Spaces ................................................................. 46
- Near-Field Optical Angular Orientation for Manipulating Biological Materials .......................................... 48
- Non-Permanent Microfluidic Interconnects ................................................................................................. 50
- Conducting Polymer Electrodes for Human Electrophysiological Recordings ............................................ 52
- Microfluidic Devices for Examining Nuclear Mechanics ............................................................................ 54
- Effect of Interstitial Flow on Tumor Cell Migration Using a 3D Microfluidic in vitro Model .................... 56

### CHEMISTRY, PAGES 58-69

- Reactivity of Monolayer CVD Graphene Imperfections Studied Using SEM ........................................... 58
- Chemical Bonding across the Periodic Table at High and Ambient Pressures ........................................... 60
- High Temperature Micro-Reactors for Stable Isotopic Analysis ............................................................... 62
- Single Molecule Catalytic Studies of Nanoscale Metal-Metal Junctions .................................................... 64
- Mid-Infrared Transparent Substrate for Alkysiloxane Monolayers ......................................................... 66
- Formaldehyde Hydration Reaction and Transition State ............................................................................. 68
# Table of Contents

**Electronics, Pages 70-95**

A Ferroelectric and Charge-Based Hybrid Nonvolatile Memory ........................................... 70
Fabrication and Testing of Chemoreceptive Neuromorphic Neuron Transistors .................... 72
Improving the GaSb/High-k Oxide Interface Using an InAs Capping Layer ......................... 74
Memory Effects Induced by Space Charge Polarization in a SmNiO$_3$ Film ....................... 76
Wirebonding and Packaging of Various Integrated Circuits .................................................. 78
High Breakdown Voltage AlGaN/GaN HEMTs for Power Switching Applications ................... 80
Low-Frequency Ultrasensitive Magnetic Detectors .................................................................... 82
Optoelectronic Probes of Suspended CNT Transistors ............................................................ 84
Implantable Device for Prevention of Late-Phase Hemorrhagic Shock Using a Novel Non-Enzymatic Fuel Cell ............................................................. 86
Tunable Capacitive Micromechanical Ultrasonic Receiver Array ........................................... 88
Laser Crystallized Thin Film Silicon Transistors ....................................................................... 90
Organic Permeable Gate Transistor for Digital Printing Applications ..................................... 92
Fabrication of SOI Device for Characterization of Thin Film Solar Cell Surfaces .................... 94
<table>
<thead>
<tr>
<th>Topic</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>Investigation of Acid Diffusion</td>
<td>96</td>
</tr>
<tr>
<td>During Laser Spike Annealing with</td>
<td></td>
</tr>
<tr>
<td>Systematically Designed Photoacid Generators</td>
<td></td>
</tr>
<tr>
<td>Room-Temperature Nanoimprint</td>
<td>98</td>
</tr>
<tr>
<td>Process for Patterning Biomolecules</td>
<td></td>
</tr>
<tr>
<td>Development of an</td>
<td>100</td>
</tr>
<tr>
<td>Organic-Inorganic Hybrid Nanoparticle Photoresist for Next-Generation Lithography</td>
<td></td>
</tr>
<tr>
<td>Hafnium and Zirconium Oxide</td>
<td>102</td>
</tr>
<tr>
<td>Nanoparticles as Novel Photoresist Materials</td>
<td></td>
</tr>
<tr>
<td>Nanocalorimetry Study of Metastable Au-Si Alloy</td>
<td>104</td>
</tr>
<tr>
<td>Patterning of Polymer Brushes as Membranes and Investigating the Effects</td>
<td></td>
</tr>
<tr>
<td>Characterization of Molybdenum Disulfide Devices</td>
<td>108</td>
</tr>
<tr>
<td>Structure and Motion of Stacking Faults in CVD-Grown Bilayer Graphene</td>
<td>110</td>
</tr>
<tr>
<td>Design and Validation of High-Efficiency Chopper for Magnetoresistive Sensors</td>
<td>112</td>
</tr>
<tr>
<td>Colloidal Self-Assembly-Directed</td>
<td>114</td>
</tr>
<tr>
<td>Laser-Melt-Induced Crystalline Silicon Nanostructures</td>
<td></td>
</tr>
<tr>
<td>Ionic Conductivity in Thin Film Y-Zr-Oxide</td>
<td>116</td>
</tr>
<tr>
<td>Electrical Characterization of Nanocystal Arrays</td>
<td>118</td>
</tr>
<tr>
<td>Development of a Monochromatic Phonon Source for Studying Nanoscale Phonon Heat Transport</td>
<td>120</td>
</tr>
<tr>
<td>Vertically Aligned Carbon Nanotube Membrane for Solar Hydrogen Generation</td>
<td>122</td>
</tr>
<tr>
<td>Calculation of the Temperature Dependent Superconducting Superheating Field</td>
<td>124</td>
</tr>
<tr>
<td>Fabrication of Nanoscale Silicon Fracture</td>
<td></td>
</tr>
<tr>
<td>Test Specimens and Calculation of Ideal Strength of Silicon</td>
<td>126</td>
</tr>
<tr>
<td>Bismuth Electrodeposition on GaAs &lt;110&gt;</td>
<td>128</td>
</tr>
<tr>
<td>Origin of the Strain Sensitivity of Superconducting Triniobium-Tin</td>
<td>130</td>
</tr>
<tr>
<td>Mass Production of Patterned Nanowires via Electroplate-and-Lift Lithography</td>
<td>132</td>
</tr>
<tr>
<td>Disappearance Mechanisms of Sub-100 nm Diameter Aluminum Dots on Sapphire during Annealing</td>
<td>134</td>
</tr>
<tr>
<td>MECHANICAL DEVICES,</td>
<td></td>
</tr>
<tr>
<td>PAGES 136-163</td>
<td></td>
</tr>
<tr>
<td>Optomechanical Experiments</td>
<td></td>
</tr>
<tr>
<td>with Ultra Thin Mechanical Membranes</td>
<td></td>
</tr>
<tr>
<td>... ... ... 136</td>
<td></td>
</tr>
<tr>
<td>Graphene Optomechanical Systems</td>
<td></td>
</tr>
<tr>
<td>... ... ... ... 138</td>
<td></td>
</tr>
<tr>
<td>Resonant Properties of</td>
<td></td>
</tr>
<tr>
<td>High-Stress Silicon Nitride Membranes</td>
<td></td>
</tr>
<tr>
<td>... ... ... 140</td>
<td></td>
</tr>
<tr>
<td>Silicon Nanowire Atomic Force Microscopy</td>
<td></td>
</tr>
<tr>
<td>Probes for High Aspect Ratio Geometries</td>
<td></td>
</tr>
<tr>
<td>... ... ... 142</td>
<td></td>
</tr>
<tr>
<td>A Single Lithography</td>
<td></td>
</tr>
<tr>
<td>Self-Aligned Vertical NanoRelay</td>
<td></td>
</tr>
<tr>
<td>... ... ... ... 144</td>
<td></td>
</tr>
<tr>
<td>SQUID and XPS Analysis of Thin Nickel Films</td>
<td></td>
</tr>
<tr>
<td>... ... ... 146</td>
<td></td>
</tr>
<tr>
<td>The Nanoaquarium: A Device for in situ Electron</td>
<td></td>
</tr>
<tr>
<td>Microscopy of Processes in Liquids</td>
<td></td>
</tr>
<tr>
<td>... ... ... 148</td>
<td></td>
</tr>
<tr>
<td>Batch Compatible Integration</td>
<td></td>
</tr>
<tr>
<td>of Nanoscale Samples and MEMS Devices</td>
<td></td>
</tr>
<tr>
<td>... ... ... 150</td>
<td></td>
</tr>
<tr>
<td>Non-Linear Dynamics of Coupled MEMS Oscillators</td>
<td></td>
</tr>
<tr>
<td>... ... ... 152</td>
<td></td>
</tr>
<tr>
<td>MEMS-Based Piezoelectric</td>
<td></td>
</tr>
<tr>
<td>Vibrational Energy Harvesters</td>
<td></td>
</tr>
<tr>
<td>... ... ... ... 154</td>
<td></td>
</tr>
<tr>
<td>Fabrication and Characterization of</td>
<td></td>
</tr>
<tr>
<td>All-Thin-Film Multiferroic Cantilevers</td>
<td></td>
</tr>
<tr>
<td>for Magnetic Energy Harvesting</td>
<td></td>
</tr>
<tr>
<td>... ... ... ... 156</td>
<td></td>
</tr>
<tr>
<td>Squeeze Film Damping in Microsystems</td>
<td></td>
</tr>
<tr>
<td>... ... ... ... 158</td>
<td></td>
</tr>
<tr>
<td>Spraycoat Lithography onto</td>
<td></td>
</tr>
<tr>
<td>Silicon Carbide-Aluminum Nitride Wafers</td>
<td></td>
</tr>
<tr>
<td>... ... ... 160</td>
<td></td>
</tr>
<tr>
<td>Microscale Electrostatic Energy Harvester</td>
<td></td>
</tr>
<tr>
<td>... ... ... 162</td>
<td></td>
</tr>
</tbody>
</table>
# OPTICS AND OPTO-ELECTRONICS, PAGES 164-197

Large Bandwidth Continuously Tunable Delay Using Silicon Microring Resonators  ..... 164
Integrated Graded-Index Luneburg Lens for Robust Fiber-to-Chip Coupling  ..... 166
High Quality Factor and High Confinement Silicon Resonators using an Etchless Process  ..... 168
Fabrication of Nanofluidic Channels on Fused Silica Wafers  ..... 170
Leveraging Opto-Mechanics to Design High Performance RF Oscillators  ..... 172
Porous Polymer Waveguides and Ring Resonators  ..... 174
Fabrication of Phase Grating Based Angle Sensitive Pixels by CMOS Post-Processing  ..... 176
Demonstration of Optical Nanocircuits at Infrared Wavelengths  ..... 178
Continuous Operation of a Hybrid Solid-Liquid State Reconfigurable Photonic System without Resupply of Liquids  ..... 180
Microfabricated X-Ray Optics for CHESS  ..... 182
Directed Nano Assembly of Materials Using Silicon Photonics and Plasmonics  ..... 184
Optical Manipulation of Nanoscale Objects and Biomolecules with Photonic Crystal Nanotweezers  ..... 186
Highly Sensitive Spectroscopic Interferometers Using Photonic Crystal Structures  ..... 188
Gel Optical Waveguides with Live Encapsulation & Integrated Microfluidics  ..... 190
Fabrication of 3D Simple Cubic Silicon Woodpile Photonic Crystal Structures  ..... 192
Waveguide Investigation for Single Molecule Detection  ..... 194
A High-Frequency Silicon Optomechanical Oscillator  ..... 196
PHYSICS AND NANOSTRUCTURE PHYSICS, PAGES 198-243

Effects of Interfacial Electronic Structure on In-Plane Spin-Transfer Torque in MgO Magnetic Tunneling Junctions .......... 198
A Three-Terminal Spin Transfer Torque Device Utilizing the Giant Spin Hall Effect of Tantalum .......... 200
Spin-Torque Effect in Asymmetric FeCoB/MgO/FeB Magnetic Tunnel Junctions .......... 202
Photocurrent Measurements of Supercollision Cooling in Graphene P-N Junction Devices .......... 204
Manipulating Surface States in Topological Insulators .......... 206
Spin Injection into Graphene by the Spin Hall Effect .......... 208
Probing Spin Transfer Torque in Magnetic Nanoparticles .......... 210
X-Ray Imaging of Magnetic Normal Modes Driven by Spin Transfer Torque in Magnetic Nanopillar Devices .......... 212
Graphene in the Third Dimension .......... 214
Vortex Dynamics in Nanofabricated Superconducting Devices .......... 216
Melting Dynamics of Colloidal Crystals on Patterned Surfaces .......... 218
Electrical Transport Across Individual Grain Boundaries in Polycrystalline Graphene .......... 220
Quantum-Limited Measurement and Entanglement in Superconducting Circuits .......... 222
Investigation of Charge Transport in Quantum Dot Solids .......... 224
Perpendicular Magnetic Anisotropy in Ultrathin Co|Ni Multilayer Films Studied with Ferromagnetic Resonance and Magnetic XRM .......... 226
Fabrication of Nanoscale Josephson Junctions for Quantum Coherent Superconducting Circuits .......... 228
Superfluid Quantum Interference in Multiple-Turn Reciprocal Geometry .......... 230
Electronic Structure Calculations for Cr_{1-x}Al_{x} .......... 232
Development of Superconductor Circuits for Readout of Quantum Nanomechanical Resonators .......... 234
**Physics and Nanostructure Physics, Continued**

- Fabrication of Superconducting Devices for Quantum Information Science 236
- Photonic Crystal Nanocavities for Solid State Quantum Optics 238
- First-Principles Study of the Surface Chemistry of Metal-Oxide Nanostructures 240
- Electron Beam Lithography of Superconducting Qubits and Meander Wires 242

**PROCESS AND CHARACTERIZATION, PAGES 244-247**

- Determining Charge Mobility by Electric Force Microscopy Using a Transistor with Interdigitated Gate Electrodes 244
- Novel Fabrication Process of Silicon Nano-Field Effect Transistor for Detection Analysis 246

**INDEXES, PAGES 249-252**

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**COVER LEGEND & PHOTO CREDITS**

Cover Background: CNF Project Number 657-97, pages 4-5  
Green Oval: CNF Project Number 1782-09, pages 122-123  
Purple Oval: CNF Project Number 1469-06, pages 246-247  
Orange Oval: CNF Project Number 731-98, pages 8-9

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This Research Accomplishments publication highlights research projects carried out at the Cornell NanoScale Science and Technology Facility (CNF) in the past year, selected from the work of our approximately 700 new or continuing users.

We wish to thank our users for their contributions to this publication, and we hope that you will find this collection of work to be instructive and impressive, both for the quality of the research and for the range of fields in which the tools of nanotechnology are enabling new breakthroughs.

CNF is supported by the National Science Foundation through funding to the National Nanotechnology Infrastructure Network (NNIN). NNIN is an integrated network of fourteen university-based user facilities established in March 2004 to serve the needs of researchers in the fields of nanoscale science, engineering and technology. NNIN provides users from across the nation—from academia, small and large industry, and government—with open access to leading-edge tools and processes for nanofabrication, synthesis, characterization, design, simulation, and device integration.

We welcome inquiries from all researchers, especially those with no previous experience in nanofabrication, since the outstanding staff members of the NNIN are highly skilled at teaching new users. The NNIN also conducts extensive education, training and outreach activities for the general public. Additional information about NNIN can be found at www.nnin.org.

New Equipment and Capabilities

The CNF continues to upgrade its capabilities. The following new tools have been acquired over the past year and are installed or being installed. Please see the CNF web site for the names of the staff members to contact for training.

JEOL JBX 9500FS Electron Beam Lithography System:

This next-generation e-beam lithography system has been developed jointly by JEOL and CNF with funding from an NSF MRI grant, and is the first of its kind in the world. The new system is improved relative to previously-existing tools in nearly every category of specification: smaller spot size, increased speed, dramatically improved placement accuracy and stitching, and more user-friendly software. With the combination of our recently acquired JEOL JBX 6300FS system and the new tool (arriving in January 2013), the CNF is well-positioned to maintain its role as the leading center in the US for advanced electron beam lithography.

CorSolutions Microfluidic Probe Station:

Allows for nonpermanent connections to be made rapidly at any location on the surface of a microfluidic device, thereby providing a platform for efficient microfluidic experiments. Digital image capture allows for testing and documenting device function. To make a connection, a fluidic probe is positioned over a microdevice port and with the press of a button the probe is engaged making a leak-tight, compression connection. The system includes peristaltic pumps capable of fine computer-controlled fluidic flow in forward or backward directions or in biomimetic (pulsating) patterns.

Oxford Plasma-Enhanced Chemical Vapor Deposition System:

Improving our capabilities for oxide and nitride deposition with stress control, replacing an IPE system that served us for more than 20 years.

Oxford 100 Reactive Ion Etching System Upgrade:

Expanding the number of etching gases so as to be able to tune the ratio of chemical species in the plasma.

Reynolds Tech Electroplating Stations:

With solutions to deposit gold, copper and nickel. Contact us if you are interested in electroplating other materials.
Educational Outreach

The CNF participates in numerous educational outreach activities, both alone and as part of NNIN. One network-wide program is the NNIN Research Experience for Undergraduates (REU) Program. During the summer of 2012, 94 undergraduate students (selected from over 850 applications) participated in the 10-week NNIN REU internship program of focused research in micro and nanotechnology. Ten of those interns spent their summer in Ithaca, working with Cornell faculty, graduate students, and CNF staff. Graduates of the NNIN REU program are eligible to apply for the NNIN International REU (iREU) Program with placement in nanotechnology labs throughout Europe and Japan; this past summer, 17 REUs returned for the iREU Program. The research accomplishments for all the REU students can be viewed at http://www.nnin.org/research-experience-undergraduates. We are grateful to the National Science Foundation for their continued funding support for the Research Experience for Undergraduates activities, but we also seek corporate funds to augment this program. Please contact Dr. Lynn Rathbun, NNIN Program Manager, to discuss corporate sponsorship (rathbun@cnf.cornell.edu).

CNF continues to host many educational workshops and special events at Cornell. These include our short course, “Technology & Characterization at the Nanoscale” (CNF TCN), open to participants from academia, industry, and government. It includes lectures and demonstrations, and also hands-on lab activities in the cleanroom. The next short course will be held in June 2013 (http://www.cnf.cornell.edu/cnf5_courses.html).

Over the past year, CNF has hosted visits and tours for over 2500 people; from prospective graduate students and incoming faculty members, to visiting dignitaries and corporate executives, to school children. These include over one hundred distinct visits and events. We particularly enjoy meeting and working with high school students—introducing them to the nano-world we live in. We also have activities 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. Contact Ms. Melanie-Claire Mallison with your visit or workshop requests (mallison@cnf.cornell.edu).

As always, we welcome your comments about CNF and its operations, as well as suggestions for improvements.

Dan Ralph, Lester B. Knight Faculty Director
Donald Tennant, Director of Operations
A Selection of 2012 Patents, Presentations, and Publications Related to CNF Research


“Agarose gel optical waveguides with encapsulation of live cells and integrated microfluidics”; Jain, A., Erickson, D., Conference on Lasers and Electrooptics (CLEO), San Jose, May 2012.


“All-Thin-Film Ferroelectric Magnetic Field Sensors”; I. Takeuchi, International Symposium on Piezoelectric Materials and Devices, Hirokasi, Japan, April 2012.


“Apparatus, Methods, and Applications Pertaining to Point of Care Diagnostics”; D. Erickson, Dr. Ethel Ceserman, Matthew Mancuso, Li Jiang, Docket No. 5794-01-US, United States, MPR - Manuscript Provisional, APP No. 61/598,599, 2/14/12.


“Case study in new chemistry and physics at high pressure: ground and excited state properties of water ice, A”; Hermann, A., Industrial Research Limited Seminar Series, Wellington, New Zealand, 4/2012.


“Combining the use of x-ray synchrotron radiation and supersonic molecular beams to examine the growth of crystalline organic thin films: what in situ real-time techniques can tell us, and what they can’t”; James R. Engstrom, invited, NSLS/CFN Joint Users’ Meeting, Brookhaven National Laboratory, May 2012.


“Conductive and Flexible Metal Oxide Nanosheets”; R. Robinson, invited talk, 2D Materials Beyond Graphene Workshop, the US Army Research Office and the Ohio State University Institute for Materials Research, Columbus, Ohio, August 8, 2012.

“Confinement and collective behavior of 4He near the superfluid transition”; F.M. Gasparini, Invited talk, LT26, Beijing China, 2012.


“Controlling the growth morphology of carbon nanotubes: from suspended bridges to upright forests”; Y. Cao, Y.-Q. Xu, Nanoscale 4, 1682-1687, 2012.


“Developing the pieces of a superconducting surface code for quantum computing”; Strand, Joel, Raytheon BBN Technologies, June 7, 2012.

“Development of a dispersive read-out technique for quantum measurements of nanomechanical resonators”; Dr. F. Rouxino, Prof. M. LaHaye, APS March Meeting, February 27th, 2012.


“Electrical transport between single-crystal domains in graphene: bigger is not always better”; A.W. Tsen, L. Brown, M. Levendfor, F. Ghahari, P. Huang, C. Ruiz-Vargas, D. Muller, P. Kim, and J. Park, APS March Meeting, Boston, MA, February 2012.


“Engineered culture models to analyze tumor-microenvironment interactions”; C. Fischbach-Teschl, Keynote speaker, World Biomaterials Congress, Chengdu, China, June 1-5, 2012.


“Entropic Spectral Broadening in Carbon Nanotube Resonators”; Arthur W. Barnard, Vera Saznova, Arend van der Zande, Paul L. McEuen, Noise and Nonlinearities in Mechanical Resonators, Barcelona, Spain, 5/31/12, poster).


“Ethics and Sciences in Modern Times; Nanotechnology and One Scientist’s View”; S. Tiwari, Openmind (2012).


“Flash memory for true number generation and device fingerprinting”; co-inventor: E. Suh, KK. Yu, Y. Wang, provisional patent by Cornell Research Foundation, 2012.


“Graphene and Other Two Dimensional Crystals”; M. Spencer, WOCSEMA, Napa, CA, February 2012.


“Harmonic RFID for indoor localization based on nonlinear transmission line”; Y. Ma, provisional patent by Cornell Research Foundation, 2012.


“High Speed High Quality Factor Resonator Modulator”; M. Lipson, Mohammad Soltani, Lawrence Tzuoang, Docket No. 5962, 6/18/12.


“Lab-on-a-Syringe Point of Care Diagnostics for Simple Visible Readouts in Resource Limited Settings”; Dr. Ethel Cesaran, D. Erickson, Li Jiang, Matthew Mancuso, Docket No. 5794, 1/20/12.


“Microengineered blood vessel mimics to analyze the tumor angiogenic switch”; Verbridge, S., Keystone Symposium, poster, 2012.


“Microfluidics for cancer cell chemotaxis”; Mingming Wu, Invited, Physical Oncology public lecture at University of Southern California, Los Angeles, CA, April, 2012; Invited, EMBL conference on microfluidics, Heidelberg, Germany, July 2012.

“Microfluidics for cell migration”; Mingming Wu invited, Center for Cell Control, University of California at Los Angeles, April, 2012, Los Angeles, CA.


“Microsystems Pathways to a Greener World”; Amit Lal, JASON’s Session, April 2012; Vellore Institute of Technology, Jan 2012.


“Multiferroic mechanical devices”; I. Takeuchi, Multifunctional Materials Workshop #7, Panama City, Panama, August 2012.


“New inorganic EUV resist with high etch resistance, A”; Markos Trikeriotis, SPIE Advanced Lithography, Extreme Ultraviolet (EUV) Lithography III, San Jose, California, USA, February 2012.


“Optically induced microfluidic reconfiguration”; Krishnan, M., Erickson, D., Lab-on-a-Chip, 12, 613-621, 2012.


“Overcoming the Temperature Increase Hurdle in Photonic Crystal Molecular Tweezers”; Serey, X., Chen, Y.F., Fain, R., Kang, P., and Erickson, D. Conference on Lasers and Electrooptics (CLEO), San Jose, May 2012.


“Quantitative Measurements of Spin Torque and the Discovery of a Giant Spin Hall Effect”; Ralph, D. C., CQS Collective Dynamics of Quantum Systems Workshop Austin, TX, April 20, 2012.

“Quantitative Super-resolution Imaging Uncovers Reactivity Patterns and Dynamics on Single Nanocatalysts”; X. Zhou, N.M. Andoy, G. Liu, E. Choudhary, K.-S. Han, H. Shen, and P. Chen, Poster, Gordon Research Conference on Noble Metal Nanoparticles, Mount Holyoke College, June 2012.


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“Spin dependent tunneling and possible spin torque effects in magnetic nanoparticle tunnel junctions”; C. Wang, 2012 March Mtg of the APS, Feb. 27-March 2, 2012, Boston, MA.


“Spin orbit driven ferromagnetic resonance and torques in single ferromagnetic layers”; Ferran Macia, C. Pepin and A. D. Kent, 2012 APS March Meeting, Boston, MA.

“Spin Torque in Asymmetric CoFeB/MgO/FeB Magnetic Tunnel Junctions”; Hsin-wei Tseng, 2012 March Meeting of the American Physical Society, Feb. 27-March 2, 2012, Boston, MA.


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<thead>
<tr>
<th>Abbreviation</th>
<th>Meaning</th>
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<tbody>
<tr>
<td>%</td>
<td>percentage of one hundred</td>
</tr>
<tr>
<td>%/k</td>
<td>percentage of one thousand</td>
</tr>
<tr>
<td>µl</td>
<td>microliter</td>
</tr>
<tr>
<td>µm</td>
<td>micrometer</td>
</tr>
<tr>
<td>µN</td>
<td>micro-Newton</td>
</tr>
<tr>
<td>&lt;</td>
<td>is less than</td>
</tr>
<tr>
<td>&gt;</td>
<td>is greater than</td>
</tr>
<tr>
<td>~</td>
<td>approximately</td>
</tr>
<tr>
<td>1D</td>
<td>one dimensional</td>
</tr>
<tr>
<td>2D</td>
<td>two dimensional</td>
</tr>
<tr>
<td>2DEG</td>
<td>two dimensional electron gas</td>
</tr>
<tr>
<td>3D</td>
<td>three dimensional</td>
</tr>
<tr>
<td>3DOM</td>
<td>three-dimensionally ordered macroporous carbon</td>
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<tr>
<td>α-Si</td>
<td>amorphous silicon</td>
</tr>
<tr>
<td>A&amp;M</td>
<td>Agricultural &amp; Mechanical</td>
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<tr>
<td>AC</td>
<td>alternating current</td>
</tr>
<tr>
<td>AFM</td>
<td>atomic force microscopy/microscope</td>
</tr>
<tr>
<td>AFOSR</td>
<td>Air Force Office of Scientific Research</td>
</tr>
<tr>
<td>Ag</td>
<td>silver</td>
</tr>
<tr>
<td>AgNO₂</td>
<td>silver nitrate</td>
</tr>
<tr>
<td>AgSR</td>
<td>silver-alkanethiolate</td>
</tr>
<tr>
<td>AIC</td>
<td>aluminum-induced crystallization</td>
</tr>
<tr>
<td>Al</td>
<td>aluminum</td>
</tr>
<tr>
<td>Al₂O₃</td>
<td>aluminum oxide</td>
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<tr>
<td>AlGaN</td>
<td>aluminum gallium nitride</td>
</tr>
<tr>
<td>AM</td>
<td>amplitude modulation</td>
</tr>
<tr>
<td>APD</td>
<td>avalanche photodiode</td>
</tr>
<tr>
<td>APS</td>
<td>advanced photon source</td>
</tr>
<tr>
<td>Ar</td>
<td>argon</td>
</tr>
<tr>
<td>ARC</td>
<td>anti-reflective coating</td>
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<td>ArF</td>
<td>argon fluoride</td>
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<td>As</td>
<td>arsenide</td>
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<tr>
<td>ASC</td>
<td>aspartate transaminase</td>
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<tr>
<td>atm</td>
<td>standard atmosphere (as a unit of pressure)</td>
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<td>ATRP</td>
<td>atom transfer radical polymerization</td>
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<td>bisphenol aminomethyl</td>
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<td>BPB</td>
<td>bisphenol base</td>
</tr>
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<td>BPF</td>
<td>bisphenol F</td>
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<td>BSA</td>
<td>bovine serum albumin</td>
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<td>C</td>
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<td>C₆H₄N₂H₂SO₃</td>
<td>dihydroxyaniline</td>
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<td>C₆H₄N₂SO₃H</td>
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<tr>
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<tr>
<td>Abbreviation</td>
<td>Meaning</td>
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<td>ABBREVIATIONS AND THEIR MEANINGS</td>
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<tr>
<td>EMCCD</td>
<td>electron multiplying charge coupled device</td>
</tr>
<tr>
<td>EO</td>
<td>electro-optic</td>
</tr>
<tr>
<td>EOT</td>
<td>equivalent oxide thickness</td>
</tr>
<tr>
<td>EPICS</td>
<td>electronic photonic integrated circuits</td>
</tr>
<tr>
<td>EPR</td>
<td>enhanced permeability and retention</td>
</tr>
<tr>
<td>Er</td>
<td>erbium</td>
</tr>
<tr>
<td>ErAs</td>
<td>erbium arsenide</td>
</tr>
<tr>
<td>ESM</td>
<td>effective screening medium</td>
</tr>
<tr>
<td>EUV</td>
<td>extreme ultraviolet</td>
</tr>
<tr>
<td>ex vivo</td>
<td>Latin for “out of the living” — that which takes place outside an organism</td>
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<tr>
<td>F</td>
<td>fluorine</td>
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<tr>
<td>Fe</td>
<td>iron</td>
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<tr>
<td>FeDRAM</td>
<td>ferroelectric dynamic random access memory</td>
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<tr>
<td>FEM</td>
<td>finite element method</td>
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<tr>
<td>FES</td>
<td>functional electrical stimulation</td>
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<td>FESEM</td>
<td>field-emission scanning electron microscopy/microscope</td>
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<tr>
<td>FET</td>
<td>field-effect transistor</td>
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<td>FFT</td>
<td>fast Fourier transforms</td>
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<tr>
<td>fg</td>
<td>femto gram</td>
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<td>FIB</td>
<td>focused ion beam</td>
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<td>FIR</td>
<td>far infrared</td>
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<tr>
<td>f</td>
<td>femto seconds</td>
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<td>FLT</td>
<td>field-like torque</td>
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<td>FM</td>
<td>frequency modulation</td>
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<td>FMR</td>
<td>ferromagnetic resonance</td>
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<td>FOTS</td>
<td>fluorosilane, tridecafluoro-1,1,2,2-tetrahydrooctylchlorosilane</td>
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<td>FRAP</td>
<td>fluorescence recovery after photobleaching</td>
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<td>FRET</td>
<td>fluorescence resonance energy transfer</td>
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<td>FTIR</td>
<td>Fourier transform infrared spectroscopy</td>
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<td>FWM</td>
<td>four-wave mixing</td>
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<td>Ga</td>
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<td>GaAs</td>
<td>gallium arsenide</td>
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<tr>
<td>GaAsN</td>
<td>gallium arsenide nitride</td>
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<tr>
<td>GaInNAs</td>
<td>gallium indium nitride arsenide</td>
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<td>GaN</td>
<td>gallium nitride</td>
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<tr>
<td>GaP</td>
<td>gallium phosphide</td>
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<tr>
<td>GaSb</td>
<td>gallium antimonide</td>
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<tr>
<td>GASP</td>
<td>growth advantage in stationary phase</td>
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<tr>
<td>GB</td>
<td>glass bead</td>
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<td>GBLMA</td>
<td>gamma butyrolactone methylacrylate</td>
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<tr>
<td>GC</td>
<td>gas chromatograph</td>
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<td>GC-C-IRMS</td>
<td>gas chromatography combustion iso- tope ratio mass spectrometry</td>
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<td>germanium</td>
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<td>GEDJ</td>
<td>geometrically enhanced differential immunocapture microdevices</td>
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<td>GFET</td>
<td>graphene field effect transistor</td>
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<td>GHz</td>
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<td>GI</td>
<td>gastrointestinal</td>
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<td>GMF</td>
<td>gross mean fluorescence intensity</td>
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<td>GMR</td>
<td>giant magnetoresistance</td>
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<td>gold nanorod</td>
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<td>graphene nanoribbons</td>
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<td>gradient refractive index</td>
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<td>GUI</td>
<td>graphical user interface</td>
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<td>hexamethyldisilazane</td>
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<td>HMGB</td>
<td>high-mobility group box protein</td>
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<tr>
<td>HOMO-LUMO</td>
<td>highest occupied molecular orbital &amp; lowest unoccupied molecular orbital</td>
</tr>
<tr>
<td>HOPG</td>
<td>highly oriented pyrolytic graphite</td>
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<tr>
<td>HRS</td>
<td>high resistance state</td>
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<td>HRTEM</td>
<td>high-resolution transmission electron microscopy</td>
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<td>HS-dsDNA</td>
<td>thiol terminated single-stranded deoxyribonucleic acid</td>
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<td>HSQ</td>
<td>hydrogen silsesquioxane</td>
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<td>IARPA</td>
<td>Intelligence Advanced Research Projects Activity</td>
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<td>integrated circuit</td>
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<td>ICP</td>
<td>inductively coupled plasma</td>
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<td>ICP-MS</td>
<td>inductively coupled plasma mass mass</td>
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<tr>
<td>ICP-RIE</td>
<td>inductively coupled plasma reactive ion etcher</td>
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<tr>
<td>IFVD</td>
<td>impurity free vacancy diffusion</td>
</tr>
<tr>
<td>IGERT</td>
<td>Integrative Graduate Education &amp; Research Traineeship</td>
</tr>
<tr>
<td>IID</td>
<td>impurity induced disordering</td>
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<tr>
<td>IEI</td>
<td>ion implant enhanced interdiffusion</td>
</tr>
<tr>
<td>ICMSE</td>
<td>International Journal of Computational Materials Science &amp; Surface Engineering</td>
</tr>
<tr>
<td>In</td>
<td>indium</td>
</tr>
<tr>
<td>in situ</td>
<td>Latin phrase which translated literally as ‘in position’ — to examine the phenomenon exactly in place where it occurs</td>
</tr>
<tr>
<td>in vitro</td>
<td>Latin for “within glass” — refers to studies in experimental biology that are conducted using components of an organism that have been isolated from their usual biological context in order to permit a more detailed or more convenient analysis than can be done with whole organisms</td>
</tr>
<tr>
<td>in vivo</td>
<td>Latin for “within the living” — experimentation using a whole, living organism</td>
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<td>InAlN</td>
<td>indium aluminum nitride</td>
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<td>indium arsenide</td>
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<td>indium arsenide nanowires</td>
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<td>INDEX</td>
<td>Institute for Nanoelectronics Discovery and Exploration</td>
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<tr>
<td>InGaAsN</td>
<td>indium gallium arsenide nitride</td>
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<tr>
<td>IPA</td>
<td>isopropyl alcohol</td>
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<tr>
<td>IPE</td>
<td>ion and plasma equipment</td>
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<tr>
<td>IPT</td>
<td>in-plane torque</td>
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<tr>
<td>IR</td>
<td>infrared</td>
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<td>IRMS</td>
<td>iso- tope ratio mass spectrometry</td>
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<tr>
<td>IrO2</td>
<td>iridium oxide</td>
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<tr>
<td>IrOx</td>
<td>iridium oxide</td>
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<tr>
<td>ISFET</td>
<td>ion-sensitive field effect transistor</td>
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<td>ITO</td>
<td>indium tin oxide</td>
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<td>JP-8</td>
<td>Jet Propellant 8</td>
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<tr>
<td>k</td>
<td>dielectric constant</td>
</tr>
<tr>
<td>k</td>
<td>Kelvin (a unit of measurement for temperature)</td>
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<tr>
<td>kDa</td>
<td>kilodaltons</td>
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<td>KFM</td>
<td>Kelvin force microscopy</td>
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<td>kg</td>
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<td>KPFM</td>
<td>Kelvin probe force microscopy</td>
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<td>L/D</td>
<td>length-to-diameter</td>
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<td>LAO</td>
<td>lanthanum aluminum oxide</td>
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<td>LASSP</td>
<td>Laboratory of Atomic &amp; Solid State Physics</td>
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<td>LED</td>
<td>light-emitting diode</td>
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<tr>
<td>LER</td>
<td>line edge roughness</td>
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<td>LO</td>
<td>local oscillator</td>
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<td>low-k</td>
<td>low dielectric constant</td>
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<td>LPCVD</td>
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<td>lpm</td>
<td>liter per minute</td>
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<td>LR8</td>
<td>low resistance state</td>
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<tr>
<td>Abbreviation</td>
<td>Meaning</td>
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<td>LSQR</td>
<td>localized surface plasmon resonance</td>
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<td>lutetium</td>
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<td>LWGs</td>
<td>liquid-core/liquid-cladding waveguides</td>
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<td>line width roughness</td>
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<td>M-OPTG</td>
<td>microring-based optical pulse-train generator</td>
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<td>metal-assisted chemical etching</td>
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<td>MAMA</td>
<td>methyl adamantyl methacrylate</td>
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<td>MBIE</td>
<td>molecular beam epitaxy</td>
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<tr>
<td>MCBJ</td>
<td>mechanically controllable break junction</td>
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<td>MD</td>
<td>molecular dynamics</td>
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<td>ME</td>
<td>magnetoelastic</td>
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<td>MEG</td>
<td>maleimide-ethylene glycol disulfide</td>
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<td>microelectromechanical systems</td>
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<td>MF</td>
<td>microfluid</td>
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<td>MFMR</td>
<td>microfabricated micro-reactors</td>
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<td>magnesium oxide</td>
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<td>MgGs</td>
<td>molecular glasses</td>
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<td>MHz</td>
<td>megahertz</td>
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<tr>
<td>micron</td>
<td>micrometer, aka µm</td>
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<td>metal-insulator-ferroelectric-insulator-semiconductor</td>
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<tr>
<td>mm</td>
<td>minutes</td>
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<tr>
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<td>millimeter</td>
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<td>methyl-methacrylate-co-methacrylic acid</td>
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<td>mmHg</td>
<td>millimeters of mercury; unit of pressure measurement</td>
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<td>MOCVD</td>
<td>metal oxide chemical vapor deposition</td>
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<td>MONOS</td>
<td>metal/oxide/nitride/oxyde/semiconductor</td>
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<td>metal oxide semiconductor</td>
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<tr>
<td>MoS2</td>
<td>molybdenum disulfide</td>
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<td>MOSFET</td>
<td>metal oxide semiconductor field effect transistor</td>
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<td>MOVPE</td>
<td>metal organic vapor phase epitaxy</td>
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<td>MP</td>
<td>multiphoton microscopy</td>
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<td>MQCA</td>
<td>magnetic quantum-dot cellular automata</td>
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<td>MQW</td>
<td>multiple quantum well</td>
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<td>MRA</td>
<td>multifunction reconfigurable antenna</td>
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<tr>
<td>MRAM</td>
<td>magnetic random access memory</td>
</tr>
<tr>
<td>MRFM</td>
<td>magnetic resonance force microscopy</td>
</tr>
<tr>
<td>MRI</td>
<td>magnetic resonance imaging</td>
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<tr>
<td>ms</td>
<td>microsecond</td>
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<td>millivolt</td>
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<td>MVD</td>
<td>molecular vapor deposition</td>
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<td>MWNT</td>
<td>multiwalled carbon nanotube</td>
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<td>MG</td>
<td>megohms</td>
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<td>nitrogen</td>
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<td>National Aeronautics &amp; Space Administration</td>
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<td>NBCCT</td>
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<td>NCRR</td>
<td>National Centers for Research Resources</td>
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<td>NEMS</td>
<td>nanoelectromechanical systems</td>
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<td>NEXAFS</td>
<td>near edge x-ray absorption fine structure</td>
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<td>NH3</td>
<td>ammonium fluoride</td>
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<td>Ni</td>
<td>nickel</td>
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<td>NIDCD</td>
<td>National Institute on Deafness &amp; Other Communication Disorders</td>
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<td>NIH</td>
<td>National Institutes of Health</td>
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<td>NIR</td>
<td>near-infrared</td>
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<tr>
<td>nl</td>
<td>nanoliter</td>
</tr>
<tr>
<td>nm</td>
<td>nanometer</td>
</tr>
<tr>
<td>NMR</td>
<td>nuclear magnetic resonance microscopy / spectroscopy</td>
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<tr>
<td>NNIN REU</td>
<td>National Nanotechnology Infrastructure Network Research Experience for Undergraduates (NNIN REU) Program</td>
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<tr>
<td>NORIS</td>
<td>nanometrology optical ruler imaging system</td>
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<td>NPR</td>
<td>nonlinear polarization rotation</td>
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<td>NPs</td>
<td>nanoparticles</td>
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<td>NPs</td>
<td>nanopores</td>
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<td>ns</td>
<td>nanosecond</td>
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<td>NSF</td>
<td>National Science Foundation</td>
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<tr>
<td>NSF-SGER</td>
<td>National Science Foundation Small Grants for Exploratory Research</td>
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<tr>
<td>NSOM</td>
<td>near-field scanning optical microscopy</td>
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<tr>
<td>NSSP</td>
<td>nanostructured semipolar</td>
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<td>NVM</td>
<td>non-volatile memory</td>
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<td>NW FETs</td>
<td>nanowire field-effect transistors</td>
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<td>NYSTAR</td>
<td>New York State Office of Science, Technology &amp; Academic Research</td>
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<tr>
<td>O</td>
<td>oxygen</td>
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<td>OFET</td>
<td>organic field effect transistor</td>
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<tr>
<td>Oh number</td>
<td>Ohm's number</td>
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<tr>
<td>OLED</td>
<td>organic light-emitting diode</td>
</tr>
<tr>
<td>ONO</td>
<td>oxide/nitride/oxide</td>
</tr>
<tr>
<td>ONR-MURI</td>
<td>Office of Naval Research Multidisciplinary University Research Initiative</td>
</tr>
<tr>
<td>ORPS</td>
<td>optical particle sizer</td>
</tr>
<tr>
<td>OPV</td>
<td>organic photovoltaic cells</td>
</tr>
<tr>
<td>ORMO-MRAM</td>
<td>orthogonal spin-transfer magnetic random access memory</td>
</tr>
<tr>
<td>OTFT</td>
<td>organic thin-film transistor</td>
</tr>
<tr>
<td>p-n, p/n</td>
<td>p-type and n-type semiconductors joined together</td>
</tr>
<tr>
<td>p-type</td>
<td>positive semiconductor</td>
</tr>
<tr>
<td>p/E</td>
<td>program/erase</td>
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<tr>
<td>Pa</td>
<td>pascals</td>
</tr>
<tr>
<td>PAB</td>
<td>post-apply bake</td>
</tr>
<tr>
<td>PAE</td>
<td>power-added efficiency</td>
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<tr>
<td>PAG</td>
<td>photocatalytic generator</td>
</tr>
<tr>
<td>PAMAM</td>
<td>polyamidoamine</td>
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<tr>
<td>PANOMs</td>
<td>planarized apertures for near-field optical microscopy</td>
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<tr>
<td>Pb</td>
<td>lead</td>
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<tr>
<td>PBG</td>
<td>photonic bandgap</td>
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<tr>
<td>PBPK</td>
<td>physiologically-based pharmacokinetic</td>
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<tr>
<td>PBS</td>
<td>phosphate-buffered saline</td>
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<tr>
<td>PtSe</td>
<td>lead selenide</td>
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<tr>
<td>PC</td>
<td>persistent current</td>
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<tr>
<td>PC</td>
<td>photocurrent</td>
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<tr>
<td>PCB</td>
<td>printed circuit board</td>
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<tr>
<td>PCBM</td>
<td>fullerene derivative [6,6]-phenyl-C61-butyric acid methyl ester</td>
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<tr>
<td>PCM</td>
<td>phase change material</td>
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<tr>
<td>PCT</td>
<td>photonic crystal nanocavity</td>
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<tr>
<td>Pl</td>
<td>palladium</td>
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<td>PD</td>
<td>photodetector</td>
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<td>PDMs</td>
<td>polydimethylsiloxane</td>
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<td>PE-GNR</td>
<td>polyelectrolyte gold nanorod</td>
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<td>PEPS</td>
<td>post-exposure bake</td>
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<tr>
<td>PEC</td>
<td>photoelectrochemical</td>
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<tr>
<td>PECVD</td>
<td>plasma enhanced chemical vapor deposition</td>
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<tr>
<td>PEDOT-PSS</td>
<td>poly(3,4-ethylenedioxythiophene): poly(styrenesulfonate)</td>
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<tr>
<td>PEG</td>
<td>polyethylene glycol</td>
</tr>
<tr>
<td>PEI</td>
<td>poly(ethylene imine)</td>
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<tr>
<td>PFM</td>
<td>piezoresponse force microscopy</td>
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<tr>
<td>pH</td>
<td>potential of hydrogen</td>
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<td>Ph.D.</td>
<td>doctorate of philosophy</td>
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<tr>
<td>PhC</td>
<td>photonic crystal</td>
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<tr>
<td>PID</td>
<td>proportional-integral-derivative</td>
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<tr>
<td>PL</td>
<td>photoluminescence</td>
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<td>pi</td>
<td>picoliter</td>
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<td>PLS</td>
<td>pulsed laser deposition</td>
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<tr>
<td>PLGA</td>
<td>poly(lactic-co-glycolic) acid</td>
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<tr>
<td>PMG</td>
<td>poly(methyl glutarimide)</td>
</tr>
<tr>
<td>PMMA</td>
<td>poly(methyl methacrylate)</td>
</tr>
<tr>
<td>PnP</td>
<td>poly(m-phenylenevinylene-co-2,5-dioctoxy-p-phenylenevinylene)</td>
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<tr>
<td>psi</td>
<td>polycrystalline silicon</td>
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<td>POP</td>
<td>polyolefin plastomer</td>
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<td>PPM</td>
<td>photolithographic phase masks</td>
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<td>PS</td>
<td>polystyrene</td>
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<td>PSL</td>
<td>polystyrene latex</td>
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<td>PSMO</td>
<td>praseodymium strontium manganese</td>
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<tr>
<td>PSSM</td>
<td>phase separation micro-molding</td>
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<tr>
<td>Pt</td>
<td>platinum</td>
</tr>
<tr>
<td>PtIr</td>
<td>platinum/iridium</td>
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<tr>
<td>PTX</td>
<td>paclitaxel</td>
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ABBREVIATIONS AND THEIR MEANINGS

2011-2012 CNF RESEARCH ACCOMPLISHMENTS
<table>
<thead>
<tr>
<th>Abbreviation</th>
<th>Meaning</th>
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<tbody>
<tr>
<td>PVA</td>
<td>polyvinyl alcohol</td>
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<tr>
<td>PVC</td>
<td>polyvinyl chloride</td>
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<td>PVDF</td>
<td>polyvinylidene fluoride</td>
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<td>PVP</td>
<td>polyvinylpyrrolidone</td>
</tr>
<tr>
<td>Py</td>
<td>Ni$_2$Fe$_3$_u</td>
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<tr>
<td>PZT</td>
<td>lead zirconate titanate (Pb$<em>2$Zr$</em>{1-x}$Ti$_x$O$_3$)</td>
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<td>Q</td>
<td>high quality factor</td>
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<td>QD</td>
<td>quantum dots</td>
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<tr>
<td>QW</td>
<td>quantum well</td>
</tr>
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<td>QWI</td>
<td>quantum well intermixing</td>
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<td>RA</td>
<td>resistance-area</td>
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<td>Re #</td>
<td>Reynolds number</td>
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<td>RF</td>
<td>radio frequency</td>
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<tr>
<td>RF MEMS</td>
<td>radio frequency microelectromechanical systems</td>
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<td>RFFID</td>
<td>radio frequency identification</td>
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<tr>
<td>RIE</td>
<td>reactive ion etch</td>
</tr>
<tr>
<td>RMS or rms</td>
<td>root mean square</td>
</tr>
<tr>
<td>RNA</td>
<td>ribonucleic acid</td>
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<tr>
<td>ROS</td>
<td>reactive oxygen species</td>
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<td>RPEVCD</td>
<td>remote plasma-enhanced chemical vapor deposition</td>
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<td>RRAM</td>
<td>resistive random access memory</td>
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<td>RTA</td>
<td>rapid thermal anneal</td>
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<td>RTD</td>
<td>resistance temperature device</td>
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<td>seconds</td>
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<td>sulfur</td>
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<td>SA-MOVPE</td>
<td>selective area metal organic vapor phase epitaxy</td>
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<td>SBAC</td>
<td>surface active block copolymers</td>
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<td>SAEED</td>
<td>selected area electron diffraction</td>
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<td>SAMs</td>
<td>self-assembled monolayers</td>
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<td>tin</td>
</tr>
<tr>
<td>SBH</td>
<td>Schottky barrier height</td>
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<td>Sc</td>
<td>scandium</td>
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<td>SCAN</td>
<td>single-chromatin analysis at the nanoscale</td>
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<tr>
<td>sccm</td>
<td>standard cubic centimeters per minute</td>
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<td>scCO$_2$</td>
<td>supercritical carbon dioxide</td>
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<td>SCOFET</td>
<td>single crystal organic field effect transistor</td>
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<td>SCORE</td>
<td>SNARE Complex Reporter</td>
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<td>SDS</td>
<td>sodium dodecyl sulfate</td>
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<td>selenium</td>
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<td>sec</td>
<td>seconds</td>
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<td>SEC</td>
<td>scanning electrochemical microscopy</td>
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<tr>
<td>SEM</td>
<td>scanning electron microscopy/microscope</td>
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<td>SERS</td>
<td>surface enhanced Raman spectroscopy</td>
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<td>SF$_6$</td>
<td>sulfur hexafluoride</td>
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<td>SFLS</td>
<td>supercritical fluid-liquid-solid</td>
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<td>SH</td>
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<td>silicon</td>
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<td>silicon aluminum oxy-nitride</td>
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<td>SiC</td>
<td>silicon carbide</td>
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<td>SiH$_3$</td>
<td>silane</td>
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<td>silicon nitride</td>
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<td>silicon nanowires</td>
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<td>SiO$_2$</td>
<td>silicon dioxide</td>
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<td>SiROF</td>
<td>sputtered iridium oxide film</td>
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<td>supported lipid bilayers</td>
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<td>single-layer graphene</td>
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<td>SLM</td>
<td>spatial light modulator</td>
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<td>SLUG</td>
<td>superconducting low-inductance undulatory galvanometer</td>
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<td>SNPs</td>
<td>silver nanoparticles</td>
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<td>SNR</td>
<td>signal-to-noise ratio</td>
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<td>solid oxide fuel cells</td>
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<td>SOI</td>
<td>silicon-on-insulator</td>
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<td>SPCM</td>
<td>scanning photocurrent microscopy</td>
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<td>SPD</td>
<td>switching phase diagram</td>
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<td>SPR</td>
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<td>Sr$_2$Ru$_2$O$_3$</td>
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Construction of Synthetic Ecologies

**CNF Project Number: 398-91**

**Principal Investigator: Robert H. Austin**

**User: Guillaume Lambert**

**Affiliation: Department of Physics, Princeton University**

**Primary Source of Research Funding: National Institutes of Health**

**Contact:** austin@princeton.edu, GL56@nyu.edu

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**Abstract:**

We continue to have a wide range of projects: (1) separation of cells in blood using deterministic bumping, (2) studies of the ecological order that emanates from bacterial populations competing under starvation conditions, (3) developing new approaches to study the evolutionary dynamics of drug resistance by studying collective response to drug-induced stress gradients, and (4) evolution dynamics of cancer cells in 3D structures.

**Summary of Research:**

(1) We created a linear series of ecological patches (MHPs) consisting of two parallel rows of MHPs. The two rows of MHPs are connected by a line of nanoslits to each other in a linear fashion, and the top and bottom sides of the two rows are connected to larger nutrient channels through a set of nanochannels. The idea was to design a micro-ecology in which two different strains of bacteria would be physically separated but would share media, so that the two populations could share DNA with each other but would not be able to intermingle. (See Figure 1.)

(2) We show that MDA-MB-231 metastatic breast cancer cells collectively invade a three dimensional (3D) matrix by following a glucose gradient, while non-metastatic MCF-7 breast cancer cells do not. The dynamic nature of the front cell leadership is presumably due to the long range strain field effective drag penalizing the leader and aiding the following cells. (See Figure 2.)

(3) We developed a microfluidic device for mL/min flow rate, continuous-flow capture of viable circulating tumor cells from blood using deterministic lateral displacement (DLD) arrays. We show here that a DLD array device can isolate CTCs from blood with capture efficiency greater than 85% CTCs at volumetric flow rates of up to 10 mL/min with no effect on cell viability. (See Figure 3.)

(4) We use comparative genomic hybridization on a custom chip to show that within 20 minutes of ciprofloxacin exposure replication of multiple copies of genes expressing ATP dependent transporters appear. We show that this rapid genomic amplification is done in a time efficient manner via placement of the genes encoding the pumps near the origin of replication on the bacterial chromosome. The de-amplification of multiple copies back to the wild type number is a function of the duration is a function of the ciprofloxacin exposure duration. (See Figure 4.)

**References:**


Figure 1: (A) The general schematic of the Double 1-D MHP Device. (B) Higher magnification SEM view of a single MHP.

Figure 2: The experimental setup used to study the collective invasion of cancer cells in a 3D microenvironment. (A) Schematic 2D view of the device. (B) 3D view of the collagen gel with the green beads inside and the cancer cells on top.

Figure 3: A concentrator deterministic lateral displacement array with one input and two outputs (collection and waste). Fluorescent micrographs of the trajectories of 3.1 micron beads in a demonstration array with 12 micron gaps, 18 micron triangular posts, 1/20 array tilt and 2.0 micron critical size highlight device function. Using a mirrored design, large particles dispersed in the inlet are focused against the central channel wall, where they can be collected at the narrow collection output while other particles enter the waste outlet.

Figure 4: DNA copy number changes. Track 1 represents wild-type E. coli treated by 5 micrograms/ml kanamycin for 30 minutes. Track 2 represents wild-type E. coli treated by 10 micrograms/ml chloramphenicol for 30 minutes. Track 3 represents ciprofloxacin resistant E. coli treated by 100 ng/ml ciprofloxacin for 30 minutes. Track 4 represents wild-type E. coli treated by 20 ng/ml ciprofloxacin for 30 minutes. Track 5 represents wild-type E. coli treated by 100 ng/ml ciprofloxacin for 30 minutes. (See full color version on inside front cover.)
Retinal Implant Project

CNF Project Number: 657-97
Principal Investigators: Douglas Shire, Ph.D.\textsuperscript{1,2,3}, John Wyatt, Ph.D.\textsuperscript{2}
Users: Marcus Gingerich, Ph.D.\textsuperscript{1,3}, Bruce McKee, M.S.\textsuperscript{3}, Douglas Shire, Ph.D.\textsuperscript{1,2,3}


Primary Source of Research Funding: Sources of Funding: VA Boston Healthcare System, Center for Innovative Visual Rehabilitation; NIH ARRA 1-R01-HL090856-01A1
Contact: dbs6@cornell.edu, mdg37@cornell.edu, bruce.mckee7@gmail.com
Web Site: http://www.bostonretinalimplant.org

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 that 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.

To date, the CNF-fabricated components of this system have been various proof-of-concept test structures and tools used in the research effort and an integrated combination of the external flexible circuit and a stimulating electrode array. Si wafers serve as carriers for these freestanding films during processing. The electrode leads are fabricated inside of ‘sandwiches’ of polyimide and amorphous silicon carbide (SiC), while the IrO\textsubscript{x} 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 and solder attachment of discrete components onto an internal flexible circuit board, which is hermetically sealed into an ultraminiature Ti can. The coils are soldered and glued to the integrated external flex-array which is in turn thermo-sonically bonded to the hermetic feedthrough of the Ti can. Finally, the thermo-sonic bonds are protected and insulated with an overmold. 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 a radio frequency (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.

Scientific challenges still remain in realizing a chronically implantable retinal prosthesis. While our first generation device was primarily encapsulated in polymers for short term proof-of-concept implant studies, our second generation...
A key component of a high-density implantable prosthetic package assembly process is the reliable attachment of a flexible, microfabricated electrode array to the feedthroughs that carry signals into and out of the hermetic, sealed prosthetic package that contains the sensitive electronics. The hermetic Ti case is shown in Figure 1 where the feedthroughs are located on the lower surface rather than on the edge as in more traditional Ti can configuration. Also shown are the suture arms for in vivo stabilization, the RF coil and a portion of the flex-array. Utilizing the CNF, a flexible, array was microfabricated with 256+ densely-packed sputtered iridium oxide film (SIROF) stimulation electrodes of a section of which is shown in Figure 2.

We have continued our effort to optimize the prosthesis efficiency by microfabricating custom acute electrode arrays, shown in Figure 3, to study the stimulus pulse parameters needed for effective retinal stimulation. In addition, we have continued the pursuit of 3D structures to improve electrode-cell coupling, and thus device performance. Through our efforts at the CNF, we have successfully developed a microfabricated polyimide-backed subretinal penetrating electrode array, an SEM image of two electrodes is shown in Figure 4. The purpose of the structure is to cause the electrode to penetrate into the retinal tissue placing it in very close proximity to the target cells. The structure was formed with SU-8 and has SIROF, a high charge transfer material, on the tip as the stimulating electrode. These structures have been electrochemically evaluated and compared to traditional planar SIROF electrodes on the same polyimide substrate.

References:
A Microfluidic Device for Structural Studies of Nucleic Acids

CNF Project Number: 692-98
Principal Investigator: Lois Pollack
User: Christopher D. Jones

Affiliation: School of Applied and Engineering Physics, Cornell University
Primary Research Funding: National Science Foundation
Contact: LP26@cornell.edu, cdj39@cornell.edu

Abstract:
We assembled a microfluidic device that enables high-resolution structural studies of nucleic acids. The device contains a mineral matrix capable of rapidly generating hydroxyl radicals to cleave the solvent accessible backbone of nucleic acids. Protection from radical cleavage (footprinting) can identify protein binding sites or the presence of tertiary structure. Here we report the fabrication of a microfluidic prototype containing micron-sized particles of pyrite that generate enough radicals within 20 ms to cleave DNA sufficiently for footprinting. This prototype enables the development of high-throughput and/or rapid reaction devices with which to probe nucleic acids.

Summary of Research:
Protection analysis (footprinting) with hydroxyl radicals (•OH) has long been a valuable tool for the study of DNA and RNA structure and complexes of nucleic acids with proteins [1-4]. Our approach exploits the advantages of microfluidic systems: small sample volumes, short reaction times and the potential for multiplexed and/or high throughput applications. Iron sulfide (fool’s gold, or pyrite) micro-particles are captured by a constriction in a micro-channel. Hydrogen peroxide (H2O2) flowing through the pyrite matrix generates sufficient quantities of •OH to cleave co-flowing nucleic acid

Figure 1 (adapted from [5]): Device schematic and microscopy result. Top) Full-length DNA and H2O2 are pushed into the device and after interacting with the pyrite region, the DNA backbone is cleaved at different locations. Bottom) Experimental data from a confocal microscope, with dye flowing from left to right. Prior to interaction with the pyrite (left side), the dye emission is brighter than in the post-pyrite region (right side). The shape and packing of the pyrite is observable in the center of the image.

Figure 2 (adapted from [5]): Device and pyrite at different length-scales. The Zeonor is cut square to a slightly larger dimension than the 1 cm channel length, visible in the top-left image, and the entire device is potted in PDMS. Gold-colored pyrite is readily visible in the top-right image; channel width is 200 µm. Aggregates of the pyrite, and an individual particle are shown in the bottom panels. (See full color version on inside front cover.)

The microfluidic device consists of a single linear channel with a constriction in the middle to trap the pyrite particles (Figure 2). The device is fabricated from the thermoplastic Zeonor. Positive Si wafer masters of our device channel geometry are made using standard photolithography and plasma etching techniques, and we use the wafer to emboss the devices on a hotpress [6, 7]. Inlet and outlet holes at the ends of the channels are punched with a custom die set, and tubing is sealed to the device with TorrSeal epoxy. Because we work with radioactively labeled DNA, the devices are potted in PDMS as an added leak-prevention safety measure.
The devices are sealed by applying a piece of clear one-sided polyester pressure sensitive tape (ARseal 90697) [8]. The resulting device has excellent optical clarity and high resistance to chemical decomposition. The use of tape enables more efficient positioning of the pyrite particles, if the particles get stuck in the adhesive at an undesirable location or clog, that region can be surgically removed and “patched” with another piece of tape. Using this technique, embossed devices can be sealed with nearly 100% efficiency about one hundred times faster than with curable silicones.

This powerful nucleic acid analytical technique has applications to biological questions including identifying and studying the binding of proteins to DNA and RNA, and monitoring folding and structural transitions of DNA and RNA, e.g. Schlatterer and Brenowitz [9]. We are able to show significant fragmentation (12%), yielding good signal to background for clear fragment detection. Not surprisingly, DNA fragmentation is affected by solution flow rate and the amount of pyrite in the device. As expected, the amount of fragmentation increases as more pyrite is added or as the flow rates decreases (Figure 3). Based on the performance of our prototype, we anticipate achieving single digit millisecond time resolution for the footprinting reaction. The time resolution is sharp, clearly defined, and readily calibrated.

Our device could be integrated with lab-on-a-chip modules that conduct the post-exposure processing necessary to visualize •OH reaction products. Our versatile and simple-to-implement approach is readily combined with other fluidic elements, bringing a powerful new analytical tool to the chip.

Acknowledgements:
This work is a collaboration with Joerg Schlatterer and Michael Brenowitz at Albert Einstein College of Medicine.

References:
Microfluidic Cell Culture Analog Devices to Mimic Animal Exposures to Toxins and Drugs

CNF Project Number: 731-98
Principal Investigator: Michael L. Shuler
Users: Mandy Esch, Jean-Mathieu Prot, Paula Miller, Yu Ling Huang, Christopher Hall

Affiliations: 1) Biomedical Engineering, Cornell University; 2) University of Central Florida
Primary Source of Research Funding: NSF/NBTC, NYS Office of Science, Technology and Academic Research (NYSTAR), US Dept. of Defense (DOD)
Contact: mls50@cornell.edu, mbe2@cornell.edu
Web Site: http://www.bme.cornell.edu/people/profile.cfm?l=Shuler&f=Michael

Abstract:
Our group has developed microfluidic in vitro devices that 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 combine these in vitro device experiments with physiologically-based pharmacokinetic model simulations to predict toxin and drug dynamics in humans [2].

Summary of Results:
Microfluidic GI Tract Modules with Three-Dimensionally Shaped Membranes. To simulate the oral uptake of drugs and substances with μCCAs, we have developed a microfabricated gastrointestinal tract model that consists of an on-chip membrane and apical and basolateral fluidic chambers (Figure 2). The membrane can be shaped so that intestinal villi are recreated when epithelial cells (Caco-2) and mucous-producing cells (HT-29) are co-cultured to confluence on the membrane [4]. We showed that key aspects of the intestinal epithelium such as tight junctions developed on the villi shaped membranes [4].

Development of a Twelve-Organ Chamber μCCA. Micro cell culture analog (μCCA) devices are physical representations of physiologically-based pharmacokinetic models (PBPK). To better represent the PBPK model, we expanded our device design to include twelve tissue compartments, representing ten organs (Figure 3). The twelve chambers were scaled from an average male organ sizes by a factor of 105. Since cells will be seeded with matrigel to create a 3D microenvironment, each chamber was designed with a height of 150 μm for matrigel-cell at the bottom and medium flow above. The estimated in vivo volumetric flow...
rate at each organ was scaled down so that physiological residence times were maintained. In addition, the flow pattern was considered as a hydraulic circuit, treating the flow rate as the current, the drag as the resistor, and the pressure drop as the applied voltage. For each channel, the hydraulic diameter was calculated and the Reynolds number was obtained (typically smaller than 1). The pressure drop across each stream was adjusted and maintained approximately to be 500 Pascal based on the hydraulic diameter and flow rate. Finally, the pressure drop across the distributor and the exit triangle is calculated using the Ergun equation, with the assumptions for the pillars in the distributor as baffles. The shear stress for each channel was less than 1.8 dyn/cm².

**Measuring Contractile Forces Generated by Muscle Cells within µCCAs.**
Microcantilevers are widely used devices allowing for the measurement of bending moment due to physical stress applied on the beam structure. The application of such sensors has previously been mainly in gas and chemical vapor sensing but more recently, they were also used in biochemical detection as a mass sensor to determine the concentration of biological agents.

Our group is developing this technology to determine contractile forces generated by muscle cells that are cultivated on a cantilever arrays. The stress generated by myotube contractions can be followed in a detection chamber by a photodetector, allowing the measurement of the deflection of the bridge. Cantilevers are a good alternative to study muscle cell dysfunction and allow the extraction of several physiological parameters of interest like contractile force, refractory period and tetanic contraction [3].

The cantilever array was fabricated on silicon-on-insulator (SOI) wafers. Dimension and geometry were defined by contact photolithography and total backside etching was achieved to release the cantilever using DRIE etching. Cantilever characteristics were determined using a scanning electron microscope to confirm the thickness, length and width of the bridges (Figure 4).

**References:**


Microchip Devices for the Study of Single Vesicle Fusion Events

CNF Project Number: 848-00
Principal Investigator: Manfred Lindau
Users: Brian Kim, Ying Zhao, Kassandra Kisler

Affiliations: School of Applied and Engineering Physics, Cornell University
Primary Source of Research Funding: National Institutes of Health
Contact: ML95@cornell.edu, BNK25@cornell.edu, YZ86@cornell.edu, KJK29@cornell.edu

Abstract:
Neurotransmitters and hormones are stored within secretory vesicles inside the cell and release their contents in a quantal event by fusion with the plasma membrane. We develop and fabricate microchip devices that we apply in cell experiments for electrochemical measurements of quantal release events from adrenal chromaffin cells. Surface patterned electrodes on transparent surfaces are applied to relate the fusion events to fluorescence changes observed under a microscope with simultaneous electrochemical detection. CMOS microchip devices are used for highly parallel detection of quantal release events from a large number of cells.

Summary of Research:
Adrenal chromaffin cells release adrenaline or noradrenaline in response to stimulation. These molecules are stored in secretory vesicles inside the cell and are released by fusion of the secretory vesicles with the plasma membrane. Single vesicle release events, also termed quantal release events, can be detected as an amperometric current using a carbon fiber or microfabricated electrode [1]. We use microfabricated electrode arrays to characterize the spatiotemporal relation between conformational changes of the fusion complex protein SNAP-25, located on the plasma membrane, and the opening of a fusion pore. In these experiments a SNAP-25 construct named SCORE (SNARE COMplex REporter) [2] is used, which incorporates the fluorescent protein groups CFP and Venus. In cells, this construct responds to stimulation with a small change in fluorescence resonance energy transfer (FRET). The construct was expressed in adrenal chromaffin cells and individual fusion events were spatiotemporally correlated with the FRET changes. The results indicate that the conformational change indicated by the SCORE constructs precedes fusion by ~ 90 ms (Figure 1).

We developed and tested transparent microelectrode arrays capable of simultaneous amperometric measurement of oxidizable molecules and fluorescence imaging through the electrodes [3]. Surface patterned microelectrodes were fabricated from indium-tin-oxide (ITO), nitrogen-doped diamond-like carbon (DLC) deposited on top of ITO, or very thin (12-17 nm) gold films on glass substrates. Chromaffin cells loaded with lysotracker green or acridine orange dye were placed atop the electrodes and vesicle fluorescence imaged with total internal reflection fluorescence (TIRF) microscopy while catecholamine release from single vesicles was measured as amperometric spikes with the surface patterned electrodes. Electrodes fabricated from all three materials were capable of detecting amperometric signals with high resolution. Unexpectedly, amperometric spikes recorded with ITO electrodes had only about half the amplitude and about half as much charge as those detected with DLC or gold electrodes, indicating that the ITO electrodes are not...
as sensitive as gold or DLC electrodes for measurement of quantal catecholamine release [3] (Figure 2).

To test pharmacological and molecular manipulations of the fusion complex and other regulators of transmitter release in a high throughput approach, we develop an electrochemical CMOS sensor array.

The CMOS IC incorporating a $10 \times 10$ array of potentiostats as well as timing and readout circuitry (Figure 3a) was fabricated at MOSIS, and $15 \mu m \times 15 \mu m \text{AlCu}$ contacts were included in the design to serve as interconnection between electrode material and underlying integrated amplifier (Figure 3b). Post-fabrication of the Pt electrode array was performed photolithographically. A schematic of the geometry with an aligned image of a single electrode of the array (Figure 3c) is shown on expanded scale in Figure 3d.

For live-cell measurement of single vesicle release events, chromaffin cells were plated on the biosensor (Figure 4). Out of the twelve electrodes shown in Figure 4, eight electrodes were fully or partially covered by chromaffin cells. After a high-$k^+$ stimulation, many amperometric events were recorded by most of cell-covered electrodes as well as adjacent electrodes (Figure 4). Each amperometric spike corresponds to quantal release of the contents from a single vesicle. This parallel recording of amperometric spikes confirmed the biosensor’s capability to record quantal release events simultaneously from live-cells over the typical recording time ($\sim 150$ s) with sub-millisecond resolution and pico-ampere current resolution that is comparable to conventional low-noise amplifiers.

References:
Mimicry of Biological Adhesion Through Fabrication of Fibrillar Surfaces

CNF Project Number: 1225-04
Principal Investigator: Anand Jagota
Users: Nichole Nademann, Ying (Richard) Bai


Primary Source of Research Funding: Department of Energy (DE-FG02-07ER46463)
Contact: anj6@lehigh.edu, nkn3@cornell.edu, yib308@lehigh.edu

Abstract:
We show that highly enhanced and selective adhesion can be achieved between surfaces patterned with complementary micro-channel structures. An elastic material, poly(dimethylsiloxane) (PDMS), was used to fabricate such surfaces by molding into a silicon master with micro-channel profiles patterned by photolithography. We carried out adhesion tests on both complementary and mismatched micro-channel/micro-pillar surfaces. Adhesion, as measured by energy release rate required to propagate an interfacial crack, can be enhanced by up to forty times by complementary interfaces compared to a flat control, and slightly enhanced for some special non-complementary samples, despite the nearly negligible adhesion for other mismatched surfaces. For each complementary surface, we observe defects in the form of visible striations, where pillars fail to insert fully into the channels. The adhesion between complementary micro-channel surfaces is enhanced due to a combination of a crack-trapping mechanism and friction between a pillar and channel, and is attenuated by the presence of defects.

Sample Fabrication:
The fabrication process is illustrated schematically in Figure 1. We began by molding an elastomer, poly(dimethylsiloxane) (PDMS), into etched silicon masters (with parallel micro-channels on the surface) patterned by photolithography. The channel width of the original masters was fixed at 10 µm, channel depth, d, was varied (10, 20, and 30 µm), and minimum center-to-center spacing or smallest period, c, was varied in the range 20-125 µm. Molding was done by mixing liquid PDMS precursor (silicone elastomer base), with curing agent (Sylgard 184, Dow Corning) in weight ratio of 10:1. The liquid silicone mixture was degassed under vacuum for 30 minutes before applying to the master and was cured at a temperature of 80°C for two hours.

After curing, we peel the solid PDMS replica off the Si master. Samples have pillar and channel widths $w^+_p = 10 \mu m$, $w^+_c$, and interchannel spacing $c = w^+_p + w^+_c$. The positive replica is called the pillar side since the pillar width is fixed at 10 µm.

A second set of PDMS samples, complementary to the first set, was fabricated by the following replica molding process. The original PDMS samples were coated by a monolayer of n-hexadecyltrichlorosilane ($C_{16}H_{33}Cl_3Si$). Samples with a complementary surface profile were obtained by a second molding and curing of PDMS on this first set of PDMS samples with pillar and channel widths $w^-p = w^-c$; $w^-c = w^-p = 10 \mu m$. The negative replica is called the channel side since the channel width is fixed at 10 µm.

A typical sample is 635 µm thick, 30 mm long and 10 mm wide. Figure 2 shows SEM images of a pair of complementary samples with 35 µm center-to-center spacing, c.

Defects Accommodate Relative Shear and Rotation Between Strips. For complementary samples, since we took no special care to align the two PDMS strips, the first question is whether pillars will insert into their complementary channels. Stated another way, one can ask whether and how the two strips will accommodate the necessarily present misalignment. For the photolithographically fabricated channel/pillar structures studied in this work, we invariably observe defects in the form of visible striations tens to hundreds of microns in width and separated by distances on the order of millimeters.

Figure 3 (a-c) shows three complementary samples with pillar-side pillar width of 10 µm and varying spacing, c. In each case, these low-magnification micrographs have clear, featureless regions where pillars have been inserted fully into channels. In addition there are white-ish, light-scattering,
striations. These striations become wider as the inter-channel spacing, \( c \), increases; occupying most of the interfacial area for larger spacing. We observe that striations can form with different orientations with respect to the pillar/channel direction. In addition, they either end on the sample boundaries (Figure 3b) or form internal loops (Figure 3a).

To understand the nature of these striations, consider Figure 3 (d,e), which shows a complementary sample with \( c = 20 \mu m \). Figures 3d and 3e show two magnified views of a striation that runs approximately perpendicular to the channel direction. Figure 3d shows that the striation has a characteristic width comprising of two regions: an inner core with an outer region on either side of the core. Away from the striation are featureless regions where the pillars are fully inserted into the channels. The vertical lines making up the striation are pillar/channel combinations that are no longer fully inserted, i.e., the striation is visible because of light scattering from interfacial regions in partial contact. A close examination (Figure 3e) reveals that in the core region pillars are extracted from their complementary channel and shifted over by one period (a distance, \( c \)) and partially inserted into another complementary channel. Because of the complementary and symmetric nature of the two PDMS sheets, each such shift in one sheet is mirrored by an equal and opposite shift in its complementary sheet. We have observed that these shifts always have a magnitude equal to one periodic spacing, i.e., when a pillar is forced out of its channel, it shifts over exactly one periodic spacing \( c \) into which to insert itself. The striations in Figure 3 can be viewed as “screw dislocations” that carry a Burgers vector of magnitude \( c \) aligned orthogonal to the channel direction in the plane of the sheet.

Adhesion Measurements of Complementary Interfaces.
We measure the equilibrium crack length and then convert this measurement into effective interfacial adhesion energy. Figure 4 plots the energy release rate required for interfacial opening normalized by its value for a flat control. For each set of samples, we made ten measurements (error bars represent standard deviation). Half of these measurements were performed with one of the complementary sheets on the glass slide and the rest were performed with the other complementary sheet on the glass slide. We found no significant dependence on whether the pillar or channel side was wedged open. The first striking observation is that one can achieve a sizable enhancement of adhesion, up to a factor of forty, over the flat control (represented by the horizontal line in the figure) in these complementary structures. Secondly, we note that, except for one sample (\( d = 30 \mu m \)), the effective adhesion decreases monotonically as inter-channel spacing increases. Eventually, the effective work of adhesion decreases with increasing inter-channel spacing to a value lower than that of the flat control.

References:
Silicon Nitride Cantilevers for Muscle Sarcomere Force Measurements

CNF Project Number: 1255-04
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Abstract:
Titin is a giant structural protein in muscle that spans the half sarcomere from the z-band to the M-line in skeletal muscle. Although much is known about titin’s mechanical properties from tests on isolated molecules [1] or fragments of titin produced recombinantly, there is little information on its behavior within the structural confines of a sarcomere. Since the passive properties of single myofibrils are primarily associated with titin, we tested the hypothesis that titin properties might be reflected well in single myofibrils. To measure the forces produced during our experiments, we fabricated silicon nitride cantilever pairs at the CNF using photolithography and reactive ion etching [2].

Summary of Research:
Titin is a giant structural protein in muscle which spans a half sarcomere from the z-band to the M-line (Figure 1) and has been associated with passive force production in cardiac and skeletal muscles. It has spring like properties in its I-band domain dominated by extensible regions associated with the proximal and distal Ig segments and the PEVK region, named so because of its predominance in proline (P), glutamate (E), valine (V) and lysine (K) residues. Since its discovery in the mid 1970s [3], titin has emerged as an important stabilizer of sarcomeres [4], a producer of passive force [5], a regulator of active force [6], and has been associated with a variety of signaling, structural, and mechanical properties [7]. Titin is considered the third sarcomeric protein [8], and knowing its mechanical properties is essential for explaining passive characteristics of muscles.

Rabbit psoas myofibrils were used for testing. Myofibrils were harvested from rabbit psoas, chemically and mechanically isolated as described in our previous works [9], and prepared for mechanical testing using silicon nitride cantilevers (stiffness 68 pN/nm) for force measurement at one end of the myofibril (resolution < 0.5 nN), and a glass needle attached to a motor for producing sub-nanometer step sizes at the other end (Figure 2).

Testing:
Myofibrils (n = 28) were passively stretched from a nominal initial average sarcomere length of 2.5-2.7 µm by 1.0, 2.0, 2.5, and 3.0 µm at a speed of 0.1 µm/s-sarcomere and then returned to the original length at the same speed.

A distinct change in stiffness of the force-elongation curves was observed in eight of the tested myofibrils (Figure 3). The smallest sarcomere length where this was observed was 3.5 µm, while the average force at this inflection point was 68 nN (± 5 nN) when normalized to 1.0 µm of myofibril cross-sectional area.

The results of this study suggest that the properties of isolated titin molecules are well reflected in whole myofibril testing. Titin properties appear to be well preserved when titin operates within its structural boundaries of a sarcomere. This result is insofar exciting as passive myofibril testing is rather simple compared to the complex isolation, stabilization and mechanical testing of single titin proteins. Not only is a myofibril approach much easier technically, it also offers the advantage that titin can be studied in its native environment and that titin’s properties can be directly related to sarcomere forces and lengths, and thus can be extrapolated to myofibril, fibre and muscle properties.

References:
Figure 1: Schematic illustration of a sarcomere with the sarcomeric filaments titin, actin, and myosin. Titin is anchored in the z-lines of the sarcomere and extends to the M-band in the middle of the sarcomere, thereby spanning a half sarcomere. Due to its spring-like properties in the I-band region, titin is positioned to stabilize myosin filaments in the centre of the sarcomere, provide stability to sarcomeres and to act as a force modulator when sarcomeres are stretched.

Figure 2, left: (A) Photograph of a myofibril attached to a glass needle (for imposing controlled displacements) at one end and to a pair of cantilevers (for force measurements) at the other end. (B) Close up view of a myofibril. (C) A single sarcomere. (D) Schematic illustration of the three myofilament structure of a sarcomere.

Figure 3, above: Passive force-elongation curve of a single myofibril showing a characteristic inflection point (arrow) as has been found in tests using isolated titin molecules (inset point c). This inflection point is thought to reveal the length at which Ig domain unfolding starts to occur in the sarcomeres of a myofibril. Note the similarity in shape of the single molecule and single myofibril curves.
Microfluidic 3D Hydrodynamic Flow Focusing for the Rapid Protein Concentration Analysis

CNF Project Number: 1393-05
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Abstract:
A simple microfluidic 3D hydrodynamic flow focusing device has been developed and demonstrated quantitative determinations of protein concentrations. This device had a step depth cross junction structure at a hydrodynamic flow focusing point at which the analyte stream was flowed into a main detection channel and pinched not only horizontally but also vertically by two sheath streams.

Summary of Research:
Microfluidic devices were fabricated on poly (dimethylsiloxane) substrate (PDMS; Dow Corning, Midland, MI) by standard micro-molding procedures. Two lithographic processes were used to fabricate a mold of the shallow and deep channels on a silicon wafer separately. SU-8 2010 photoresist (Microchem Corp., Newton, MA) was spin-coated with the thickness of 10 µm on a silicon wafer and exposed by EV620 contact aligner. Then, 50 µm thickness of SU-8 2050 (Microchem Corp., Newton, MA) was spin-coated on the smaller channel patterned silicon surface. The second mask was aligned with the alignment markers. At this process, the shallow channel was connected to the deep channels to form the step junction structure. After the exposure and development processes, the micro-mold for microfluidic devices was made on the silicon wafer. Then, uncured PDMS mixture was dispensed over the lithographically patterned silicon mold and cured at 65°C for two hours. The cured PDMS substrate was peeled off from the master silicon mold. The PDMS microchannel substrate was bonded to quartz cover slide glass (170 µm thickness) by oxygen plasma treatment for 30 seconds. A schematic diagram and scanning electron microscope (SEM) image of the 3D microfluidic flow focusing device were shown in Figure 1 (a) and (b), respectively.

The microfluidic device was placed on the Nikon 2000 Inverted microscope stage as shown in Figure 2. Analyte and sheath streams were delivered through their inlets and 375 nm laser (16 mW) was focused through 60X water immersion objective lens. Whenever analyte molecules passed through the detection spot were excited and emitted fluorescent photon bursts.
Figure 1: The schematic diagram of microfluidic channel. (a) Chip-level view. (b) The SEM image of the cross junction area (scale bar: 10 µm).

Figure 2: Experimental setup.
Role of Tissue Factor and Tissue Factor Pathway Inhibitor Complex in Cancer Cell Adhesion

CNF Project Number: 1465-06
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Abstract:
A method for cancer cells to metastasize to distant organs is through the circulation system, in which circulating tumor cells must adhere to the blood endothelium in order for extravasation into new organs [1]. Tissue factor (TF) was over-expressed by many malignant cancer cells [2]. We hypothesize that cancer cells can use the over-expressed TF to form a complex with its endogenous inhibitor, tissue factor pathway inhibitor (TFPI), aiding in cancer cell adhesion to the blood endothelium. The hypothesis was tested using a static and dynamic flow approach. Static adhesion experiments showed significantly higher adhesion of TF expressing cells to immobilized TFPI. Dynamic flow experiments using straight microfluidic channels are currently on-going.

Summary of Results:
We fabricated 0.8 µm × 0.8 µm PDMS wells by casting PDMS onto a PDMS master. The wells were sterilized with 70% ethanol, coated with proteins (TFPI, TF9 – an antibody to TF, fibronectin for positive control, nothing for negative control) and blocked with 1% BSA to prevent nonspecific adhesion. Two breast cancer cell lines were used, 1) MDA-MB-231, a metastatic cell line that overexpresses TF and 2) MCF-7 a non-metastatic cell line that does not or weakly expresses TF. Our results showed that MDA-MB-231 had significantly higher adhesion to immobilized TFPI and TF9 than MCF-7 cells (Figure 1). Pre-treatment of MDA-MB-231 cells with an anti-TF antibody significantly decreased the adhesion to TFPI, demonstrating that the adhesion is TF-dependent (Figure 2).

Straight rectangular microfluidic channel of dimensions 125 µm × 125 µm was fabricated using soft lithography. The channels were sterilized, and coated with proteins as the static experiments. MDA-MB-231 and MCF-7 cells were introduced into the channel at physiological shear stress (0.5-4 dyn/cm²). Cancer cell adhesion to immobilized proteins was monitored and counted at pre-determined areas. This part of the study is still on-going.

References:
Figure 1: Representative phase contrast images of adherent cancer cells in PDMS wells with different protein coatings. Significantly higher adhesion of MDA-MB-231 cells to TFPI and TF9, an anti-TF antibody, was observed.

Figure 2: Representative phase contrast images of adherent MDA-MB-231 cells to TFPI-coated wells. Pre-treatment of MDA-MB-231 with an anti-TF antibody significantly decreased adhesion.
Biomimetic Blood Vessel Model to Elucidate Physicochemical Mediators of Tumor Angiogenesis

CNF Project Number: 1540-07
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Abstract:
We are developing microfluidic cell cultures in remodelable hydrogel scaffolds to recapitulate tumor angiogenesis in a physiologically relevant, spatiotemporally-controlled in vitro system. Despite significant progress in vascular research, many of the parameters contributing to aberrant endothelial function remain unclear. To address these questions, we micro-tissue engineered biomimetic blood vessels in dense collagen scaffolds and subjected them to gradients of tumor-secreted soluble factors. This platform helped illuminate hitherto underappreciated biophysicochemical mediators of tumor angiogenesis, including endothelial cell density, vessel geometry, and morphogen gradients.

Summary of Research:
The recruitment of vasculature for transport of nutrients and waste is a bona fide hallmark capability in tumor pathogenesis and constitutes an enduring target for therapeutic intervention. However, highly anticipated anti-angiogenic drugs, such as Avastin®, demonstrated underwhelming success in clinical implementation. The disparity between preclinical results and actual patient benefit indicates a need for improved assays that are capable of accurately mimicking the physiological complexities of cancer, while selectively delineating these cues with appropriate physicochemical control and spatio-temporal resolution.

By precise definition of salient biological features, microfabricated culture systems afford the independent interrogation of specific biophysical characteristics of the tumor microenvironment [1]. We exploited these technologies to explore the process of tumor angiogenesis in tissue-engineered blood vessels. Specifically, we used soft-lithography to pattern microchannels in natural extracellular matrix scaffolds. A three-channel design was used to generate stable chemical gradients across a central endothelialized vessel (Figure 1). Human umbilical vein endothelial cells (HUVEC) formed a functional, confluent vessel (Figure 2),

Figure 1, above: Cut-out view of micropatterned, type I collagen-based platform for stable, gravity-driven perfusion and gradient generation across a fully-enclosed, biomimetic blood vessel. Inset shows channel cross-section with endothelialized center channel and morphogen concentrations [C1] and [C2] in the source and sink channels, respectively.

Figure 2, left: Confocal image of quiescent, confluent endothelium, showing maximum intensity projection along the z-axis.
and vascular endothelial growth factor (VEGF) was added to the source reservoir to stimulate vessel sprouting (Figure 3). The outlet reservoirs were periodically aspirated to maintain steady, gravity-driven perfusion through all channels.

In addition to directional bias toward the VEGF source, we observed that a number of hitherto under-appreciated biophysicochemical characteristics of the vessels also regulated angiogenic sprout formation. For example, a preponderance of sprouts originated at the vessel corners, which are known to have unique mechanical and chemical profiles (Figure 4). Furthermore, the abundance of sprouts demonstrated a marked dependence on the endothelial cell density in the channel, with higher densities correlating with more sprouts. On-going investigations are identifying novel synergistic mechanisms of endothelial invasion, such as cross-talk between pro-inflammatory and pro-angiogenic signals, as well as moving toward co-culture experiments with tumor cells or stromal components [2].

References:
Micro-Patterned Surfaces for the Study of Signaling, Trafficking, and the Lysosomal Synapse

CNF Project Number: 1726-08
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Abstract:

We demonstrate the use of lithographic processes in combination with selective chemical modification to prepare micron-sized arrays of patterned biomaterial. We have previously explored the application of these patterns to mast cell signaling and IgE-receptor (FceRI) involvement in the allergic immune response [1], and are continuing membrane trafficking research with this experimental system. Recent work has focused on characterizing epidermal growth factor (EGF) receptor signaling in NIH-3T3 cells and uptake mechanisms of aggregated low-density lipoproteins (agLDL) by macrophage cells. These patterns are also aiding the study of lysosomal synapse formation with fibrillar β-amyloid plaques formed in the brains of patients suffering from Alzheimer’s disease.

Summary of Research:

Standard photolithography techniques and a polymer lift-off method allow selected biomaterial to be patterned on silicon and glass substrates. Photoresist, exposed by the ASML PAS 5500 DUV stepper, is used to prepare square, micron-sized features (0.25, 0.5, 1, and 2 µm) over a range of periodicities (2.5, 5, 10, and 20 µm). An oxygen plasma etch is then performed to transfer these features to an underlying one micron thick layer of Parylene-C. Deposition of biomaterial, and the subsequent lift-off of parylene from the silicon or glass substrate, establishes the spatially defined protein or lipid micro-patterns.

Considering the size of a single mast cell (~ 15 µm diameter), the lithographically defined features enable direct observation of either a single contact site or multiple contact sites per cell while observing many cells within a single microscope field of view. These patterned surfaces have served as a powerful tool for visualizing the spatial distributions of various signaling molecules, including FceRII bound immunoglobulin E (IgE) in Rat Basophilic Leukemia (RBL) mast cells (Figure 1). We are also looking into stimulated membrane trafficking with RBL mast cells.

In addition, we have begun to investigate signaling processes mediated by other receptors. Micro-patterned EGF surfaces reveal transiently transfected paxillin-EGFP will cluster to EGF receptor signaling complexes in NIH-3T3 cells (Figure 2).
This paxillin recruitment depends on EGF receptor tyrosine kinase activity and the actin cytoskeleton, with F-actin localizing to pattern sites. In addition, we have detected the recruitment of downstream signaling partners Ras, MEK and Erk to the EGF receptor signaling complex in a ligand-dependent manner. Ongoing work is focused on understanding the roles of paxillin and actin cytoskeleton in the activation of EGF receptor downstream signaling partners.

We further extend this patterning technique to study macrophage cell interactions with agLDL. Macrophages generate an extracellular hydrolytic acidic compartment, termed a lysosomal synapse, in order to degrade agLDL [2]. The use of micro-patterned streptavidin-LDL surfaces provides the unique opportunity to learn more about this novel macrophage-associated structure. J774 macrophages are incubated on micro-patterned protein arrays of agLDL for 30 minutes. Upon interaction between agLDL and macrophages, regions of low pH can be seen at contact sites (Figure 3). Since the agLDL is immobilized on the patterned surface, our results demonstrate that the acidic, lysosomal synapse domains are extracellular. A medically important function of macrophages is their interaction with agLDL in the walls of blood vessels. This interaction results in massive uptake of cholesterol and transformation of macrophages to foam cells [3]. An understanding of agLDL uptake that doesn’t require receptor mediated endocytosis, such as the one described, may play an important role in foam cell formation in vivo and could thus be useful in preventing atherosclerosis.

Additional work with micro-patterned surfaces will involve tracking the secretion of lysosomal contents into the agLDL-containing lysosomal synapse. Signal transduction mechanisms and the SNARE proteins that participate in the fusion events are other key aspects to be examined in this system. Further, we are researching the uptake mechanism of aggregated β-amyloid peptide by microglia. Understanding the process of microglia mediated β-amyloid degradation may contribute to new insight on the pathomechanisms operating in Alzheimer’s disease.

References:


Confined Microfluidic Environments for Studying Cell Mechanics and Cancer Invasion

CNF Project Number: 1743-09
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Abstract:
We develop microfluidic devices with design features that can elicit mechanical invasion dynamics. We analyze the behavior and responsivity of single cells as they interact with mechanical barriers that define the microarchitecture of their environment, and we elucidate the mechanics of intracellular elements.

Summary of Research:
Cell mechanics is important in many aspects of biology, from development to disease states. In particular, metastasis, the leading cause of cancer related deaths, is intrinsically a mechanical transport phenomena in which cancer cells must break free from the primary tumor, invade across physiological barriers including dense and fibrous matrices, intravasate across endothelial junctions into vascular networks, circulate under shear stress, traffic in microvessels, and then extravasate into distant tissue [1]. While many molecular signals and factors have been identified that drive this process, including autocrine and paracrine chemokine gradients and growth factor signaling, the effects of the mechanical environment as well as the mechanical behavior of cells during the invasion process are not well known. In our work, we develop microfluidic environments with dimensions that mimic highly confined physiological spaces, and we study the mechanical dynamics of cell invasion [2].

As shown in Figure 1, microbeam obstacles integrated into a confined microchannel environment induce dynamic responses in subcellular components. The cell nucleolus navigates across these barriers and the spatiotemporal dynamics of its deformations can be analyzed in the context of cell invasion. Viscoelastic properties can have implications on the cell state and phenotype [3], so here we provide a platform for assessing these properties during the invasion process.

References:

Figure 1: Timelapse image stack displaying cancer cell invasion. As the cell invades across microbarriers in a confined environment, intracellular deformations and dynamics are elicited.
Fabrication of Microfluidic Devices to Study the Fusion Kinetics of Influenza Virus to Biomimetic Membranes

CNF Project Numbers: 1816-09, 1636-08
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Abstract:
The aim of this work is to mimic influenza fusion to biomimetic membranes in microfluidic channels, using total internal reflection microscopy (TIRF) to track individual virus fusion events. Using this single particle approach, we can also distinguish between hemifusion and pore formation. Fusion of the influenza to the biomimetic membrane is initiated at low pH. We have integrated a proton uncaging technique to initiate a rapid pH drop in the microfluidic channel. This assay allows us to obtain accurate kinetic information, such as the hemifusion and pore formation rate constants, and the number of fusion proteins required to initiate fusion.

Summary of Research:
Influenza is a membrane-enveloped virus, which infects cells using membrane fusion [1] to release its genome, as illustrated in Figure 1. There are two steps in the influenza fusion process, the merging of the outer lipid monolayers (hemifusion), followed quickly by opening of the fusion pore through which the viral RNA is released. Inhibition of these steps is currently the target of antiviral and antibody therapy and it is important to understand the kinetics of this process in a controlled, in-vitro environment. We use microfluidic devices to mimic the endosomal environment. Using vesicle-vesicle fusion, solid supported lipid bilayers (SLBs) are formed in microchannels. The SLBs contain sialic acid residues to which the virus binds. The virus is dual labeled [2] with a self-quenching membrane dye, R18, an internal dye, sulforhodamine B (SRB), to monitor hemifusion and pore formation respectively. An illustration of the microfluidic device used is shown in Figure 2. Fusion is typically initiated by flowing acidic buffer at a high flow rate through the microchannels. This high shearing flow can damage the membrane as well as removing virus particles from the SLB. The pH change is also slow and uncoordinated, which has a significant effect on fusion statistics at a single virion level. To overcome this limitation we use a proton-uncaging technique to reduce the pH in the channel on the order of microseconds, in a quiescent environment. The channel is filled with a water-soluble, photoisomerizable compound, o-nitrobenzaldehyde (o-NBA) which upon UV irradiation releases a proton into solution [3].

Upon analysis of the kinetic data obtained using the uncaging method, we see that the hemifusion rate constant (k_H) and decay rate (k_HD) are in agreement with published data as seen in Figure 4. However, the number of proteins (N) required to initiate hemifusion was shown to be between 1 and 2, which is lower than previous reported values of 3. Control experiments showed that N is sensitive to the rate of acidification, and slower acidification times that occur during acidic buffer exchange smear the time distribution of fusion events.

We can now use this method to determine the effects of laboratory adaptation on influenza fusion kinetics and how this affects virulence of different strains. Influenza is typically passaged in eggs to increase the yield of virus for vaccine and antiviral development. These passages result in lab adaptations, which change the virus morphology and the receptor specificity from human to avian type receptors. Preliminary results using a clinical strain indicate that clinical strains exhibit slower fusion kinetics and are independent of pH below pH 5.5, in comparison to lab-adapted strains.

References:
Figure 1: A) TEM of influenza particle. Influenza is membrane enveloped virus which contains three transmembrane proteins on the viral surface. The most abundant protein, hemagglutinin (HA) mediates entry into the cell. B) Influenza particle binds to sialic acid receptors on the cell surface via the HA protein in the viral membrane. The virus is then engulfed into the cell via endocytosis. The endosome becomes more acidic and at a critical pH (5.5) the HA undergoes a conformational change and the viral and endosomal membrane fuse. A pore opens and the RNA is released into the cytosol and transported to the nucleus for replication.

Figure 2: A multichannel microfluidic device containing influenza virus bound to a solid supported lipid bilayer. The channel is filled with a solution of o-NBA (hexagons) and flow is stopped. The channel is then irradiated with a short UV pulse and a proton is cleaved from each o-NBA molecule, reducing the pH in the region of the UV laser and initiating fusion.

Figure 3: Images of single virus particle undergoing hemifusion and pore-formation. At $t = 0$ acidification occurs and 32 seconds later the virus hemifuses (as denoted by fluorescence dequenching) and 20 seconds a pore forms in the same virion and the internal dye is released.

Figure 4: Fusion kinetics of influenza as a function of pH. Hemifusion rate constants obtained using acidic buffer exchange (black circles) are in good agreement with literature values. When fusion is initiated using o-NBA (diamonds) at three different calibrated pH values, the hemifusion rate constant are comparable to those attained using buffer exchange. However, $N$, is seen to be significantly reduced when o-NBA method of initiation is used.
Separation and Identification of Raft Associated Membrane Species Using a Patterned Supported Lipid Bilayer Extractor

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Abstract:
New methods toward manipulation and study of membrane species within a lipid bilayer platform are desired because lipid bilayers are able to maintain native conformation and function of intrinsic membrane species. This report demonstrates design and use of a bilayer patterned device for separation, sorting, and categorization of membrane biomolecules based on their affinity for different co-existing lipid phases within a patterned, heterogeneous supported lipid bilayer.

Summary:
Membrane proteins and glycolipids are the primary targets for therapeutic development, but processing and handling any membrane-bound species while maintaining intact structural information, proper orientation, and necessary lipid associations remains a large bottleneck to characterizing and understanding their structure-function behavior [1]. Many purification strategies rely on the use of harsh chemicals and conditions that cause denaturation (in the case of proteins) and disrupt orientational order necessary for binding to soluble species and maintaining critical lipid membrane interactions or environments (e.g., lipid microdomains) that are important for function [2].

We recognized that the physico-chemical properties and spatial chemical heterogeneity of the supported bilayer can be tailored to facilitate carrying out unit operations on membrane-bound species. Our new approach separates membrane-bound species based on partitioning preference of a species between two lipid phases within a supported lipid bilayer. This separation process in the two-phase lipid bilayer is somewhat analogous to a classical single stage liquid-liquid extraction except here the immiscible “liquids” are two immiscible but mobile lipid bilayers that meet along an interface inside a microfluidic device. One kind of lipid domain, termed a lipid raft, is defined as a cholesterol and sphingolipid rich lipid phase. The other domain in our system is a more fluid phospholipid-rich domain. This design can be used to identify and isolate lipid raft residents from species that prefer more disordered lipid environments by tracking their partitioning behavior in the heterogeneous bilayer [3].

In this report, we use microfluidics to construct heterogeneous SLBs with controllable phase patterns. As illustrated in Figure 1, via laminar flow patterning, we create parallel stripes of lipid raft phase (black) and POPC-rich phase (grey) in the microchannel and load in the perpendicular side channel a mixture of membrane-bound biomolecules to sort (white and dark grey disks) in the side channel. The interface between the phases is contiguous, allowing molecules to diffuse across and partition into a preferred phase as they are transported down the main channel towards collection ports.
(Figure 2). By modifying the chemical composition of the lipid phases, channel dimensions, or the convection rate of the mixture down the channel, extraction efficiency can be tuned. We demonstrate separation and sorting of two fluorescently labeled lipid species using this device: the glycolipid, GM1, an important cell signaling molecule and lipid raft marker labeled in the headgroup, and BODIPY-DHPE, a fluorescently-labeled phospholipid.

For this prototype single stage, the extraction efficiency is defined as the amount of species extracted into the raft region normalized by the total amount of that species that entered the separation region by the end of the experiment. This quantity is a function of time because the initial input (the mixture) is a discrete plug of material. Alexa 594-GM1 entering the separation region with the POPC-rich phase is more significantly extracted to the raft phase, as expected because it is a known raft marker, while the BODIPY-DHPE remains primarily in the POPC-rich phase. For this device design and experimental conditions, almost 30% of the Alexa 594-GM1 is extracted while close to 15% of the BODIPY-DHPE is extracted into the raft phase (Figure 3). Once prepared, this device achieves an affinity-based separation and in particular, can separate and discriminate lipid raft residents from non-raft species outputting two spatially separated phases without the need for operator intervention or complicated device design.

References:
Image-Based Actuation and Valving of Flows Inside Microfluidic Channels

CNF Project Number: 1858-10
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Abstract:
We introduce a contact-free light-controlled method to actuate and valve flow in a microfluidic device. Poly(N-isopropylacrylamide) (PNIPAAm), a smart polymer that changes its wettability as a function of temperature, is grafted onto PDMS. A mixture of carbon black and PDMS is spin-coated onto the surface to convert light energy to heat. This allows for the use of light to create various temperature profiles, thus changing the surface properties of the channel. We characterize the flow rate as a function of temperature and demonstrate directing the flow into different chambers by using projector images. By extending this technique to work with sunlight, microfluidic flow can be manipulated without the need for any electrical components, making this ideal for use in resource-limited areas.

Summary of Research:
Diagnostic devices based on the lateral flow assay such as dipstick tests and paper microfluidics have been successful due to their ease of use and accuracy when detecting targets in simple samples [1]. However, these techniques are unable to analyze complex samples, as they are unable to perform processes such as mixing, filtration and concentration. Typically, pumps, valves, electrical components and controllers are needed to achieve such functionality. The resulting systems are bulky, expensive, and difficult to operate without training. Through the use of opto-thermal heating, we can achieve these operations while maintaining a simple and inexpensive architecture, making it ideal for limited-resource environments.

To control light images and heating conditions, our lab designed a setup that focuses a projector image onto the chip, with the image shape and intensity controlled by a computer [2]. We incorporate a uniform carbon black layer into the PDMS device, which converts the light to heat energy [2]. We have further modified this technique to allow for successful grafting of PNIPAAm onto the channel surface.

PNIPAAm is a “smart” polymer that is hydrophilic below 32°C but transitions to hydrophobic above that [3]. The grafting method, based on the UV photopolymerization technique developed by Schneider, et al., for poly(acrylic acid) [4], is modified here to produce PNIPAAm-grafted surfaces that span a contact angle range from 40° to 100° within a 20°C temperature change (Figure 1).
An interesting phenomenon of these surfaces is that at low temperatures, the droplet wets the surface gradually over about a minute before reaching a steady contact angle (Figure 2). We believe this is due to the rough surface topography. The contact angle recorded at each temperature is defined as the contact angle at which the width changes by less than 2% over 10s (Figure 2b). The lag of the droplet in reaching the final contact angle also affects the flow rate through microchannels, as capillary-driven flows are characterized based on the assumption that the contact angle is reached instantaneously. As Figure 3 shows, we characterize the flow as a function of temperature and show that the flow rate decreases with increasing temperature.

Finally, we use a projector illumination source to heat specific regions of the chip. We demonstrate pumping and valving by grafting a T-junction microfluidic channel that empties into two large chambers (Figure 4). A small bottle of water is connected to the microfluidic chip and leveled to minimize the pressure gradient between the bottle reservoir and the chip. An image is projected over either the right (Figure 4c) or left (Figure 4d) arm of the channel, which becomes valved off while flow continues to fill the other chamber.

We have demonstrated opto-thermally driven flow manipulation using projector imaging. Instead of using conventional mechanical pumping, which typically requires electrical power and an external pump, this method achieves flow pumping and control using heat converted from light, bypassing the need to use electricity. In the field, such a device could be designed to use solar energy as the heating source, allowing for a power-free method for point-of-care diagnostics.

References:
Suspended Carbon Nanotube Devices for Single Molecule Sensing

CNF Project Number: 1888-10
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Abstract:
We have fabricated suspended carbon nanotube (CNT) devices specifically designed for biological sensing applications. We use 6-inch-wafer processes to maximize yield. Our fabrication process produces suspended CNT with clean surfaces and minimal environmental noise. The electrode layout is compatible with a microfluidic system for delivering liquids and biological molecules to the sensor. We recently showed that this geometry is the quietest existing platform for electric field sensing in biological systems.

Summary of Research:
The two-terminal resistance of a carbon nanotube (CNT) device is sensitive to both the binding of biomolecules onto the CNT surface, and the internal motions of single biomolecules. Our group, and others, have demonstrated these effects using CNTs lying on substrates [1, 2]. However, there are compelling reasons to extend these experiments to suspended CNT biosensor devices. Suspended CNTs are distant from surfaces that have fluctuating charge. In addition, suspended CNTs allow measurements of the interactions between biomolecules and the CNT without interference from the substrate.

Figure 1 shows a scanning electron microscope (SEM) image of a completed device. The CNT is hanging about 1 μm above an oxide surface. The metal electrodes are made from Pt which can withstand exposure to high temperature (820°C) during the CNT growth process. We published fabrication details [3]. Because the CNT is grown last, we expect the CNT surface to be pristine (no photoresist residue). We have confirmed the quality of our CNT devices using Raman spectroscopy. The absence of a defect peak at 1350 cm⁻¹ is indicative of a pristine CNT (see Figure 2).

We submerge the suspended CNT devices in aqueous solutions and measure the electrostatic noise in the environment. Our measurements are made in the subthreshold regime (the CNT transistor is nearly turned off), where current fluctuations can be related to environmental noise [4]. Figure 3 and 4 show representative noise measurements made on two different devices, one CNT is surface-bound, the other is suspended. The environmental noise measured by the surface-bound CNT (~ 1.8 mV rms) is consistent with the lowest noise devices that have previously been reported [4]. The noise floor of the suspended CNT device is three times lower. We have begun writing a manuscript describing these findings. Experiments to detect single-molecule binding events are ongoing.

References:
**Figure 1:** SEM of a suspended CNT device. The CNT is hanging about 1 μm above an oxide surface. The CNT diameter is between 1 to 2 nm. The metal electrodes are made from Pt.

**Figure 2:** Raman signal from an individual suspended CNT. The large G peak is characteristic of sp² bonded carbon. The D peak, which is usually seen at 1350 cm⁻¹, is completely absent, suggesting that the CNT is pristine.

**Figure 3:** Environmental voltage fluctuations detected by a surface-bound carbon nanotube. Current fluctuations were first measured in the subthreshold regime (200 Hz sampling rate) and then divided by transconductance (dI/dV). The surface bound CNT was 1.6 μm long. The RMS average of these fluctuations is 1.8 mV. Note that RMS environmental noise in CNT devices scales inversely with the square root of the CNT length [4].

**Figure 4:** Environmental voltage fluctuations detected by a suspended carbon nanotube. Current fluctuations were first measured in the subthreshold regime (200 Hz sampling rate) and then divided by transconductance (dI/dV). The suspended CNT was 1 μm long. Despite being a shorter length, the RMS fluctuations are more than three times smaller than Figure 3.
X-Ray Lab-on-a-Chip: Sample Dialysis for Small-Angle X-Ray Solution Scattering

CNF Project Number: 1940-10
Principal Investigators: Richard E. Gillilan, Søren S. Nielsen
Users: Søren S. Nielsen, Magda Møller, Amani Alkayyali*

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Abstract:
The popularity of biological small-angle x-ray solution scattering (BioSAXS) has grown dramatically in recent years due to a confluence of algorithmic advances, availability of synchrotron sources, and changing needs of researchers in molecular biology. The method now plays an essential complementary role in molecular and structural biology [1]. To carry out BioSAXS, a wide range of sample concentrations must be measured. In actual practice, the upper limit for achievable protein concentration is not known a priori and attempts to concentrate samples can result in irreversible protein aggregation and sample loss. By designing microfluidic chips capable of concentrating protein in situ on the synchrotron beamline, “reverse concentration series” can be produced that start at the lowest detectable sample limits and rise to maximum achievable limits with minimal loss.

To accomplish this goal, regenerated cellulose dialysis membranes have been incorporated into microfluidic chip designs. Real-time concentration monitoring is accomplished through the use of on-chip fiber optic based absorption spectroscopy. Concentration factors of ~six-fold have now been achieved in 40 min, a reasonable time for data collection on the CHESS F2 beamline. X-ray scattering results show no concentration-induced aggregation.

Summary of Research:
Small-angle x-ray solution scattering on biological samples (BioSAXS) allows molecular biologists to deduce important structural information about proteins and other biomolecules from dilute solutions. The wide range of solution conditions that can be examined with BioSAXS has prompted the development of high-throughput, low-volume fluid handling systems integrated with x-ray beamline components [2]. An essential step in BioSAXS analysis, however, is that protein samples be examined at a wide range of concentrations. Typically, samples are concentrated to a maximum limit determined by solubility, then diluted to create a series. Concentration of protein samples, however, is risky since it can introduce irreversible aggregation and lead to sample

Figure 1: Second generation microfluidic chip designed for dialysis-based protein concentration on a synchrotron beamline. A square sheet of dialysis membrane is shown at center superimposed on the fluid-flow labyrinth. Optical fibers for protein concentration measurement by UV absorption are just above the membrane, but not easily visible in the picture. Circular x-ray exposure cell is visible at top.
loss. Starting with dilute protein and working upward in concentration to create a series can avoid the problem of irreversible aggregation. Such series are also expected to be valuable in the analysis of concentration-dependent oligomerization and molecular crowding effects, where maximum achievable concentration is unknown.

By incorporating a dialysis membrane into a microfluidic chip, we have been able to concentrate protein in situ on an x-ray beamline for direct analysis. Preliminary chip designs (fabricated by CNF summer student Amani Alkayyali) utilized the CNF VersaLaser to cut channels in PMMA and thin medical adhesive. By flowing 100 mg/ml polyethylene glycol (PEG) on one side of the membrane, and dilute (1 mg/ml) lysozyme solution on the other side, first protein concentration was observed. In all experiments, we used standard, commercially available regenerated cellulose membranes (Spectra/Por). A second design iteration involved using photolithography to create wafer molds for PDMS. The finer, more precise chip features allowed us to achieve two-fold protein concentration in ~ 100 min. Embedded optical fibers served to measure protein concentration by UV absorption at 280 nm (Figure 1). Time series of UV absorption can be seen in Figure 2 where three successive cycles of concentration are shown. A Guinier plot for the concentrated protein demonstrates that no detectable aggregation occurred during the runs.

Our current design also uses PDMS, but with plastic molds produced by precision milling rather than photolithography (Figure 3). The dialysis chip consists of two identical 5 mm thick pieces of PDMS with a 22 cm long, 1 mm wide and 150 µm deep channel. The two pieces of PDMS, a top and a bottom piece, are pressed together with a Spectra/ Por regenerated cellulose membrane in-between. A 250 µm by 250 µm groove on each side of the final segment of the dialysis channel makes room for a 250 µm diameter optical fiber used for the UV cell. From there, the channel becomes progressively deeper until it reaches the full depth of the PDMS pieces itself, 2 mm. This last part of the channel functions as the exposure channel. Two pieces of 25 µm polystyrene are placed above and below the channel, sealing the channel when pressure is applied on both sides, and acting as x-ray windows. To date, ~ 6-fold concentration factors have been obtained from a starting condition of 1 mg/ml lysozyme flowing at 20 µl/min for 40 min. Such flow rates are compatible with typical exposure times on CHESS F2 synchrotron beamline.

References:
Therapeutic and Diagnostic Applications of Halloysite Nanotubes

CNF Project Number: 1986-11  
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Abstract:
Naturally-occurring halloysite nanotubes bear a potential for use in the diagnosis and treatment of cancer. Halloysite nanotubes are characteristically large for nanoparticles, and these dimensions provide unique capabilities for cell capture and drug delivery. This has been successfully applied to the rapid capture of viable circulating tumor cells from clinical samples [1]. Additionally, the hollow nature of these nanotubes is suitable for sustained release of cationic drug, providing a promising platform for a new targeted drug delivery vehicle.

Summary of Research:
Halloysite nanotubes are alluminosilicate nanoparticles isolated from certain clays. Particle dimensions are somewhat variable, ranging from a few hundred nanometers to a few micrometers in length, 20 to 200 nm outer diameter, and 10-70 nm inner diameter [2].

In recent years, our group has developed a microfluidic cell capture device that is constructed from a 300 µm inner diameter microtube with a functionalized inner surface containing immobilized selectin proteins. In mimicking an inflamed post-capillary venule, this device induces a rolling cellular adhesion behavior that lends insight into a natural phenomenon, and also, with the addition of antibodies, provides a platform for the rapid capture of rare target cells from a fast-flowing mixture. It has previously been shown that a monolayer of halloysite nanotubes, when functionalized with selectin proteins, will provide greatly enhanced capture of flowing target cells [3]. This enhancement in capture was proven to be due to not only an increased surface area and greater protein adsorption, but also due to the orientation of the nanotubes on the surface.

It is evident from Figure 1, produced by atomic force microscopy (AFM), that individual nanotubes protrude above the surface to a distance of several hundred nanometers. Such an orientation of individual nanotubes can act as “tethers” and provide for more efficient cell capture because cells do not have to sediment completely through the hydrodynamic lubrication layer that slows sedimentation of a body onto the reactive surface.

Recently, the nanotube-coated microtube device has been applied to the capture of viable circulating tumor cells (CTC) from patient samples [1, 4]. CTC are cells that have broken free from a primary tumor mass, migrated into the blood stream, and utilize the circulatory system to travel to a distant site within the body to seed a secondary tumor. The microtube device was functionalized with E-selectin as well as with antibodies specific to epithelial epitopes. This device was used successfully to capture viable cells, which were maintained in culture for several days, and routinely outperformed the gold standard technique for CTC enumeration, CellSearch® (Veridex) (Figure 3).

It was very interesting to note that the halloysite nanotube coating did not provide a clear benefit in terms of the number of CTC captured from each patient sample, however samples processed on the nanotube coating were significantly more pure than identical samples processed in the absence of the nanotube coating. Contaminating cells that decrease CTC

Figure 1: AFM of the halloysite nanotube coating on a planar surface.
purity in this case are healthy leukocytes and the nanotube coating was able to prevent leukocyte adhesion, allowing us to achieve purities of > 50%. Further investigation into this feature of leukocyte contamination suggested that the roughness contributed by the nanotube coating prevented leukocyte adhesion, and the average lateral distance between large nanotubes on the surface mirrored the degree to which leukocytes could firmly adhere to the surface.

The hollow nature of halloysite nanotubes lends itself directly to acting as a vessel for drug delivery and controlled drug release. Indeed, the size of the nanotubes would impart greater payload capacity than more conventional nanoparticle vehicles. We have shown that drug release can be sustained for several days following a simple encapsulation procedure (data not shown).

In a pilot study, targeted nanotubes coated with E-selectin were incubated with cells for 10 min and then samples were immediately fixed in order to assess the adhesion efficiency of the nanotubes to target cells. Using scanning electron microscopy (SEM) nanotubes were visualized adhering to cell surfaces (Figure 4), showing the promise of halloysite nanotubes as a drug delivery vehicle.

**References:**


**Figure 2, top:** Schematic of cell capture on the halloysite nanotube-coated microtube device.

**Figure 3, middle:** CTC capture using the selectin-functionalized microtube device. The microtube device, with and without the halloysite nanotube coating, is compared to the CellSearch® technique on identical samples. Data on the left of the graph represents samples from patients diagnosed with metastatic carcinoma (breast, prostate, lung, or ovarian), and data on the right, designated Norm, represents samples from healthy participants.

**Figure 4, bottom:** SEM shows that halloysite nanotubes adhere to the surfaces of target cells.
A Novel Application of Micro Fluidic Structures in Clinical Diagnostics

CNF Project Number: 2016-11
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Primary Source of Research Funding: Ortho Clinical Diagnostics
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Abstract:
This work reports fabrication of microfluidic structures in silicon by deep reactive ion etching process for controlling the fluid flow in devices used for clinical diagnostics. The geometry of the microstructures has been defined by photolithography and Bosch process using a Unaxis 770 deep silicon etcher tool.

Summary of Research:
The demand for quantitative and quick results for patient testing is growing rapidly. Microfluidic structures have various applications in clinical diagnostics, particularly in miniaturized bioassays [1]. Capillary action drives the volume of the fluid through the microstructures. The fluid flow is controlled by the geometry and the surface properties of the microstructures.

Semiconductor technology is used to fabricate the microfluidic structures on a silicon wafer. The layout of these structures is defined by the photolithography process and the geometry is defined by deep reactive ion etching process of silicon.

Figures 1 and 2 represent the microfluidic structures fabricated using the deep silicon etcher Unaxis 770 tool at the Cornell NanoScale Facility.

References:
Effect of Substratum Nanoscale Topography on Bacterial Attachment and Biofilm Formation

CNF Project Number: 2017-11  
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Primary Source of Research Funding: USDA  
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Abstract:
In the United States, 48 million illnesses per year are attributed to foodborne pathogens. Many of these food safety problems result from the contamination of food processing equipment with harmful bacteria. Reducing microbial attachment to surfaces may decrease the incidence of foodborne illness. Previous research indicates that substrate surface topography may influence bacterial attachment behavior. In this work, silicon dioxide surfaces — exhibiting specifically engineered nano- and micro-scale topography — were fabricated in the Cornell NanoScale Facility. The attachment properties of common foodborne pathogens to these surfaces were then studied. The goal of this project is to generate an understanding of the rules of bacterial attachment to nanostructured surfaces.

Summary of Research:
In this work, the effect of nanoscale surface topography on the attachment of three bacterial species, E. coli ATCC 25922, L. innocua FSL C2-008, and P. fluorescens 1150, were studied. Chemically identical, but topographically distinct silica dice were used as substrates. The silica surfaces were thermally grown on standard silicon wafers, and subsequently patterned using DUV lithography. The surfaces were designed to exhibit surface details comparable in size and shape to that of bacterial cells. Smooth, non-patterned silica was used as a control. A schematic of the surfaces used in this study is shown in Figure 1. Substratum surface features smaller than the bacterial cell were expected to reduce effective contact area between cell and substratum, and thusly have a repelling effect. On the other hand, surfaces with topographical details larger than the cell were expected to effectively increase contact area and be conducive for attachment.

Attached bacteria were visualized using fluorescence microscopy and scanning electron microscopy. Images obtained from fluorescence microscopy were further analyzed to quantify the number of attached cells. Overall, the results of this study indicate that surface topography influences both the total amount of attached bacteria and the orientation of cells relative to surface details. However, attachment patterns were specific to the microorganism and not universally observed.

Further investigation into the mechanisms behind the observed differential attachment might allow for a better understanding of the rules of attachment to nanoscale topography and the subsequent design of materials capable of repelling bacteria.
Revealing Responsive and Stochastic Switching in Bacteria

CNF Project Number: 2034-11
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User: Guillaume Lambert

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Abstract:
Microorganisms respond to environment changes by switching their behavior and phenotype between different states. This phenotypic switching can either be stochastic in nature or purely deterministic. Here, we developed a microfluidic device to culture and monitor small populations of bacteria for hundreds of generations and use it to study the responsive and stochastic switching of E. coli bacteria under fluctuating environmental conditions.

Summary of Research:
Living organisms have to respond to environmental changes in order to survive and maintain their ability to reproduce. Microbial species can adapt to new environmental conditions by using different strategies; the optimal use of these strategies, however, strongly depends on the type of change faced [1, 2]. For instance, a cell may respond to a new environment such as a change in nutrient source by expressing a different set of genes — a process called responsive switching. On the other hand, it may also be beneficial to acquire new phenotypes by stochastically responding to changes through genetic change. The acquisition of mutations that confer antibiotic resistance is an example of a stochastic switch. Each of these strategy (responsive and stochastic switching) are used by microorganisms in nature and a better understanding of type of switching that allows bacteria to resist and adapt to harmful environments — such as antibiotic treatment — is essential to the scientific understanding of pathogens and microbial diversity.

Here, we use microfluidic technologies to continuously culture and monitor small populations of bacteria (~ 100) at the single cell level in fluctuating environments. Our goal is to characterize the type of switching used by E. coli as it undergoes environmental changes. A schematic representation of the device used is shown in Figure 1. The design is based on a similar one developed to study bacterial aging [3], where a main flow channel brings nutrients to cells growing inside chambers on each side of the channel. As cells grow in length, they push on each other and this “peer” pressure helps maintain cells near the end of the growth chamber (see Figure 2). As a result, the population inside each chamber is fixed in size and can be monitored for hundreds of generations.

Figure 1: Representation of the microfluidic device used to culture E. coli bacteria. The height of the chambers (1 micron) is chosen such that it matches the diameter of the cell, thereby restricting cells to grow as a monolayer. Cells are pushed into the flow channel and removed from the device as the population grows and divides.

Figure 2: Fluorescence micrograph of E. coli cells growing inside the device. We use the green fluorescent protein intensity to identify each cell and track its lineage over time.
As opposed to conventional culture methods (i.e., test tubes or chemostat), we can use our device to induce rapid and reversible changes in the environment. Indeed, due to the inherently small volumes involved in our microfluidic device, we have the ability to instantaneously switch between two (or more) medium. For instance, we are able to study the dynamics of the lac operon by alternating minimal media containing either glucose or lactose as the sole carbon source. We observe that the response of cells to a glucose-to-lactose transition only occurs after the environment has changed (i.e., responsive switching). Figure 3 shows fluorescence intensity of the cells during a few transitions: we see that a rapid environment changes result in momentary decreases in fluorescence intensity, which itself is tied to protein production through the rpsL promoter.

We can also study stochastic switching by monitoring the occurrence of genetic changes for cells inside our device. Most genetic changes occur at very low frequencies, but we use engineered cells which turn on the production of a specific gene, in this case green fluorescent proteins, when a promoter region undergoes methylation changes [4]. Since these type of epigenetic change occur at a rate more than 1000 faster than DNA mutations, we are able to observe how stochastic changes occur and spread within a small population. Figure 4 shows an example of stochastic switching where a single gene is turned on in a population.

Future work will use these type of epigenetic switches to stochastically trigger the expression beneficial genes in order to study the dynamics of stochastic switches within a population.

References:


Models of Microvascular Networks

CNF Project Number: 2038-11
Principal Investigator and User: Russell T. Carr

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Abstract:
Replicas of microvascular blood vessels are useful for studying blood flow in capillary networks. Tree type networks with cylindrical cross sections can be constructed from fine-bore wires, wax and polydimethylsiloxane (PDMS). These networks cannot be made reproducibly. Arcade networks with cross branches are difficult to fabricate by this method. Soft lithography promises to overcome these limitations. Our previous attempts at soft lithography were hampered by poor dimensional control. We have learned strategies at Cornell NanoScale Facility to control channel dimensions when using soft lithography and developed techniques to make arcade networks with round channels using wires.

Summary of Research:
In vitro replicas of microvascular blood vessels are used to study red blood cell distribution [1] and flow dynamics in capillary networks [2]. Our laboratory has used a “wire and wax” technique for many years to make tree networks for blood flow experiments [3]. The method involves making a network of fine-bore wires using melted wax for junctions, pouring polymer over the wire frame, and pulling out the wires after the polymer cures to leave cylindrical channels. This technique gives good fidelity of vessel diameter reproduction, but poor control of vessel lengths. As a result, each network is unique and reproduction of the same network is impossible. Another shortcoming is the difficulty of making arcade networks with cross branches. An arcade network is one with a complete cycle. These can be constructed easily if there is no cross branch in the middle by making a polygon from the wires. However, even a simple network similar to a Wheatstone bridge is difficult to make. For these reasons, soft lithography presents an interesting fabrication alternative.

Conceptually, soft lithography presents a microfabrication method that is reproducible, so multiple copies of the same network can be produced. It should be possible to control both the lengths and widths of channels. Because wires do not need to be pulled, cross branches inside of cycle can be easily made. Following methods in the literature [4] and manufacturer brochures, soft lithography methods were attempted to make microvessel networks. These attempts resulted in poor dimensional control [5].

A mask with 35 µm channel widths resulted in master molds with channels ~ 80 µm wide, and 50 µm channels in the mask created channels ~ 180 µm wide.

Using the training and equipment at CNF, the dimensional fidelity of our channel designs improved tremendously. Figure 1 shows a master mold made at CNF with nominal...
channel widths of 50 µm. Measurements of channel widths using a Profilometer ranged from 57 to 61 µm, a significant improvement compared to our previous work. Figure 2 is a photograph of another master mold made at CNF with channels 20 and 30 µm wide. Channels widths were measured at 20 and 31 µm using a profilometer.

Using the same mask that produced the network in Figure 1, we have been adapting the techniques learned at CNF to our lab at University of New Hampshire. No cleanroom is available at UNH. Spin-coating wafers and development is done in a chemical fume hood. There is no contact aligner, and exposure is done with an Uvitrone Intelliray 400 UV flood lamp on a lab bench. Measuring channel widths through a light microscope with a video caliper, the channel widths for our most recent device range from 42 to 52 µm, with an average of 48 µm. This indicates that the methods for microfluidic device fabrication can be transferred to a non-cleanroom environment.

In addition to transferring the soft lithography techniques microfluidic device fabrication from CNF to UNH, we have also made progress in our wire and wax method. It is now possible to make arcade networks with cross branches as shown in Figures 3 and 4. The new technique is to join a bent wire to a straight wire “side to side” with wax — then, do not pull the all wires completely out. For example, in Figure 3, the bent wire to the left is only pulled out to the point that the wire end reaches the junction. When the other wires are pulled completely out, this network has the same topology as Figure 1. Figure 4 shows how this technique can produce a network of nested rings. Again the bent wires are pulled out just until the straight end reaches the joint. This results in a ring with three in-/out-lets connected to an inner ring by three branches.

This project represents progress to developing microfluidic devices which can be used for in vitro experiments on microvascular blood flow in networks.

References:


Confinement of Collagen Gels in Three-Dimensional Spaces

CNF Project Number: 2040-11
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Abstract:
Both mechanical cues and biochemical cues present within the microenvironment govern the behavior of in vivo. Since metastatic cells invading through interstitial spaces encounter collagen I, we designed a microfluidic system to examine the behavior of cells in confined 3D spaces filled with collagen I gels. Using SU-8 photolithography on silicon substrates to create high aspect ratio features, we fabricated a poly(dimethyl)siloxane (PDMS)-based device that allows for externally imposed tensile strain to be applied on a confined collagen I gel. Simultaneous application of tensile strain and a chemical gradient were also incorporated in the design. This device should allow for the quantification of the effect of strained collagen in 3D space on cell motility.

Summary of Research:
The cellular microenvironment is comprised of mechanical cues, such as the stiffness of the surrounding ECM, and biochemical cues, such as the ECM proteins to which the cells bind. Similarly, several growth-factors and chemokines/ cytokines are present in the interstitial spaces that can induce a response from the invading cells. While the cell-ECM interaction that occur in vivo are difficult to accurately reproduce in vitro, collagen I gels have been widely used as model systems to study cell-ECM interactions, primarily due to the ease of use [1]. However, collagen I gels suffer from an inherent disadvantage: it is difficult to control and monitor the tensile state of the fibers that comprise the gel [2]. In turn, quantifying the forces between the cell and the collagen I fibers becomes more difficult, especially in the light of recent results which indicate that strain can alter the susceptibility of collagen fibers towards proteolytic cleavage [3, 4].

In order to induce a predetermined degree of tension on collagen I, we designed a microfluidic channel made completely of PDMS (Figure 1). To allow for fresh collagen to be introduced, we designed Y-shaped inlets and corresponding cross-channels which allow for the addition of a chemo-attractant. Using controlled motion of stepper motors, we propose to alter the strained state of the gel, and thereby induce a controlled stress on the fibers of collagen I immediately attached to the cell. We have also incorporated in the design the ability to simultaneously apply strain and a chemoattractant gradient. Such a system is expected to monitor the tug-of-war between strain and chemoattractant gradient in determining the resulting effects on cell motility.
Methods and Materials:
The pattern was written on six-inch quartz mask using the PG3600. Four-inch silicon wafers were cleaned with ethanol/acetone and treated with hexamethyldisiloxane (HMDS) vapors in the YES vapor prime oven. Subsequently, SU-8-2150 was spun on the wafer at 1200 rpm with 300 rpms\(^{-1}\) acceleration. Soft-bake was done for 5 min, 10 min, and 10 hrs at 50ºC, 65ºC, and 95ºC respectively.

After slowly cooling the wafer to room temperature, 1600 mJ/cm\(^2\) of near-UV exposure was made in steps of 15 seconds each. After a post-exposure relaxation of 15 minutes, the exposed wafer was baked at 50ºC, 65ºC, and 95ºC for 5 min, 10 min, and 10 hrs, after which the wafer was allowed to cool slowly to room temperature.

Once developed and cleaned, the wafer with features was treated with fluoro-octyl-trichloro-silane (FOTS) to allow for easy release of the PDMS. Previously reported procedures for molding PDMS on silicon were followed [5] and two mirrored PDMS stamps were aligned to create an all-PDMS device.

Future Work:
Negatively charged PDMS prevents the anchoring of collagen fibers onto the PDMS surface, leading to slippage. We propose to use layer-by-layer (LbL) deposition of charged polymer-surfactants to promote favorable anchorage of collagen onto the PDMS surface. The device will then be utilized to examine cell behavior.

References:
Near-Field Optical Angular Orientation for Manipulating Biological Materials

**CNF Project Number: 2044-11**
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**Abstract:**
Near-field optical manipulation techniques have demonstrated abilities to trap, transport, and handle micro- and nanoscale materials. To show further capabilities of our near-field optical devices, we demonstrate angular orientation of biological materials with a photonic crystal resonator using. A single microtubule is rotated by optical torque resulted from polarization in electric field, changing its angular orientation parallel to the direction of the electric field on a silicon nitride photonic crystal (PhC) resonator. Simultaneous application of optical force and torque extends the functionality of our near-field PhC resonators to be more powerful tool in biophysics study, and micro- and nanoscale physics.

**Summary of Research:**
The near-field photonics have shown better capabilities [1] for which traditional optical tweezers have accomplished within the limit of diffraction. Yet despite the potential significance in the optical manipulation, the angular orientation based on near-field optics has not been demonstrated to date. The control of the angular orientation can provide an additional capability to the near-field optical manipulation techniques. Furthermore, in biophysical study, the orientation control especially over elongated objects can be potentially significant in manipulation in that most biomolecules such as DNA, virus, and bacteria are the rod-like shapes [2].

Here we demonstrate that our resonators can perform the orientation for biological materials. In other words, we exhibited the angular orientation of a single microtubule, in which optical torque induced by dipole moment on the microtubule aligns the microtubule parallel to electric field of linearly polarized electromagnetic wave. The single microtubule is subject to the linearly polarized electric field (transverse electric mode) on the silicon nitride photonic crystal resonator we developed [3]. The rotation was described in exponential form as $f(\theta, t) = f(\theta_0, 0) \cdot e^{t/\tau}$, where $\tau$ is the time constant of the orientation [4]. Figures 1, 2, and 3 illustrate that a single microtubule is oriented by the optical torque. When the microtubule moved on the resonator, it started to interact with the electric field, partially polarized as long as a width of the resonator, $w_{res} = 600 \text{ nm}$. At $t_0 = 0$ the orientation angle of the microtubule is $\pi/2$ with respect to the horizontal direction across the resonator located vertically. To minimize the interaction between the translational motion and the rotational motion, the hydrodynamic flow was relaxed prior to observing the orientation. The microtubule was moved to the resonator as close using slow flow ($u = 1 \mu L/hr$) from left to right, so that it can interact with the resonator at the beginning. At the end the microtubule was oriented parallel to the polarized direction of the electric field.

The time constant of the orientation, $\tau$, was determined by measurements of orientation angles as a function of time, $\theta(t)$. Figure 4 shows the measured orientation angles of the microtubule with respect to time during the orientation. For the quantitative orientation measurement, a software ImageJ with a plugin, OrientationJ Measure, was used to measure the orientation angles. When the microtubule is rotating under the optical torque, the orientation angles approaches exponentially to the final orientation angle $\theta = 0$ with respect to time as modeled [5] in $f(t, \theta) = f(0, \theta_0) \cdot e^{t/\tau}$. The fitting to $e^{t/\tau}$ resulted in the time constant of the orientation $\tau = 2.603 \text{ s}$.

**Acknowledgements:**
The trapping, fabrication, and characterization in this research were supported by the U.S. Department of Energy (DE-SC0003935). The protein preparation and trapping work was also supported by the U.S. National Institutes of Health (1R21EB009202).

**References:**
BIOLOGICAL APPLICATIONS

2011-2012 CNF RESEARCH ACCOMPLISHMENTS

Figure 1, left: The angular orientation of the microtubule by the optical torque. The orientation of the microtubule was observed by the epi-fluorescent microscopy using the fluorescent-labeled microtubule. The initial orientation angle, $\theta_0$, at time $t = 0$ was selected to be $\pi/2$ such that it is perpendicular to the direction of the applied electric field. The orientation was observed until the microtubule is oriented parallel to the direction of the imposed electric field. The length of the microtubule is estimated to be 10 µm (the scale bar 5 µm) and the diameter is assumed to be 25 nm. The silicon nitride photonic crystal resonator is located vertically in the center of the field of view. The electromagnetic field propagating through the resonator from bottom to top is polarized in the transverse electric (TE) mode such that the direction of the electric field is perpendicular to the resonator.

Figure 2, center: The orientation of the microtubule at $t = 6.9$ second from $t_0$.

Figure 3, right: The orientation of the microtubule at $t = 12.9$ second from $t_0$.

Figure 4: The quantitative analysis of the orientation of the microtubule. The time constant of the orientation was determined by measuring the orientation angle as a function of time. The orientation of the microtubule is modeled with the exponential form [4] as $f(t, \theta) = f(0, \theta_0) \cdot e^{-t/\tau}$. The orientation angle with respect to time, $\tan(\theta(t))$, was well fitted to the exponential function. The fit provided the time constant of the orientation $\tau = 2.603$ sec.
Non-Permanent Microfluidic Interconnects

CNF Project Number: 2049-11
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Abstract:
Little attention has been placed on interfacing microfluidic devices to the macro-world. A reliable, user-friendly interface is highly desirable because it would allow for microfluidics to employ proven, established pumping and detector technologies of the macro-world. This lack of micro-to-macro interconnect is a well acknowledged deficiency [1-7], as researchers continue to glue pipette tips and tubing to their highly sophisticated devices [4]. While the current focus of the field is demonstrating fundamental advantages of microfluidic devices, the next stage of development needs to address the engineering of easy-to-use instrumentation to solve the micro-to-macro interface problem.

Research Summary:
A novel means of making leak-tight interconnects between the macro-world of pumps and detectors, and microchips is being developed. Similar to electronic probe workstations, this flexible technology is amenable to devices of widely varying dimensions and architectures. To use the technology, a user presses a lever on the probe arm, shown in Figure 1, to first release the compression force, before aligning the end of the probe over a microchip port. In this example, a PDMS device is shown. An under-mount microscope helps to ensure proper alignment, the view from which is shown in Figure 2. If the probe is inadequately aligned to the port, the indexing probes have been designed to slide on a breadboard surface, allowing for a user to simply and quickly re-position the probe. Finally the user releases the lever, which compresses an inert polymer elastomer gasket against the microchip, forming a seal. The compression force can be adjusted for the substrate and the tubing material implemented.

The gasket material is a perfluorinated fluoroelastomer, which is a highly inert polymer, known to have excellent chemical resistance. The gaskets are formed by imprinting, micro-molding or laser cutting depending on the required structure. To date gaskets with internal diameters as small as 90 µm have been fabricated allowing for the use of 90 µm outer diameter fused-silica tubing for ultra-low volume tube connections.

Connections can be made by this compression mechanism within seconds, and they are non-permanent, making this approach useful for not only end-use, but also for intermediate processing steps and channel cleaning.

Using this approach, connections remain leak-tight to pressures exceeding 1000 psi for glass and silicon devices. The micro-gaskets used on the end of the probe which are responsible for making the leak-tight seal are re-usable and can accommodate a variety of tubing sizes and material types. As an example in Figure 1, 1/32-inch and 1/16-inch outer diameter tubing is shown. Additionally a single probe can be used to make one or more connections. As an example, in Figures 3 and 4, the probe has been fitted with a manifold capable of making three connections simultaneously on a glass microchip device, allowing microchip ports to be much tighter in density. Figure 3 shows the top-side of a three-port manifold where three leak tight connections are made with a single compression probe arm. Figure 4 shows a view of the same three connections from an under-mount microscope.

All connections made by this approach are relatively low-dead volume as the seal is made at the surface of the device and radial surface of the tube’s end. This compression technology is compatible with a wide variety of substrate materials as no adhesives are used and it is readily amenable for use with devices with varying architectures, as probes can be repositioned quickly. The low-profile probes are 4.0 × 1.0 × 2.0² in dimension and provide a large range of motion to address ports on microdevices ranging up to a 6-inch diameter. Longer-term this approach is amenable to automation, and has the potential of allowing microdevice connections to be made robotically.
References:


Conducting Polymer Electrodes for Human Electrophysiological Recordings

CNF Project Number: 2054-11
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Abstract:
The simultaneous recording of brain activity in humans at multiple length and time scales presents many challenges. One concerns long-term electroencephalography (EEG) measurements: today’s electrodes require skin preparation and the use of conducting gels, which are not ideal for long-term recordings. The purpose of this work was to develop flexible, conducting polymer-coated electrodes for EEG that would record long-term signals. We developed a fabrication process to adapt small dry electrodes, already used for multisite recordings in the auditory cortex in animals. Based on initial results, we discuss the influence of conducting polymer on the quality of the recordings.

Summary of Research:
Improving the interface between biology and electronics is a step forward towards the understanding of physiological phenomena [1]. Conducting polymers are naturally compatible with flexible substrates, and are known to decrease electrode impedance [2] and improve the quality of recordings of neuronal activity [3]. Moreover, they are commercially available and can be deposited from solution. Doped polythiophenes, particularly poly(3,4-thylenedioxythiophene) doped with poly(styrene sulfonate) (PEDOT:PSS), is a particular example of a conducting polymer with state-of-the-art conductivity.

We used Parylene-C (PaC) as substrate and insulator layer. Parylene is vapor deposited to yield a flexible, biocompatible thin film. Electrodes patterning was achieved using standard photolithography techniques and gold lift-off. To provide high conductivity and biocompatibility, PEDOT:PSS was used as the electrode material. A last photolithography and etching step defined the PEDOT:PSS site on the electrode and the conducting polymer was spin coated on the sample.

Figure 1: Fabrication process steps of the device. (A) Vapor deposition of PaC and patterning of metal lines. (B) Vapor deposition of sacrificial PaC layer. (C) Deposition of PEDOT:PSS. (D) Peeling off of the electrode.

We optimized film morphology by varying the deposition conditions, in an effort to minimize the impedance and maximize the signal-to-noise ratio of our recordings. After rinsing, the electrode was attached to a sub-millimeter-thick polydimethylsiloxane layer (PDMS) to increase the stiffness of the original 4 µm thick electrode (Figure 1).

When working on electrodes, one objective is to optimize the surface area and the electrode material as they both change the electrochemical impedance of the device with the biological tissue. So we studied the size dependence of a gold electrode on its impedance by electrochemical spectroscopy. As expected, the impedance decreases when the surface area increases. We also used PEDOT:PSS-coated electrodes and we carried out the same electrochemical characterization
choosing the gold electrode as a control (Figure 2). The use of conducting polymer decreases the impedance by almost one order of magnitude in the frequency range of interest in comparison with the metal electrode. This can be explained by a larger effective surface area of PEDOT:PSS due to ion penetration in the film.

We characterized in vivo the new electrodes using a commercially available EEG Emotiv helmet. We recorded the electrical activity along the scalp on the fronto-central regions AF3 and AF4 without using any conducting gel at the interface. We used one gold electrode and one PEDOT:PSS-coated electrode with the same surface area as recording sites and reference electrodes on both mastoids. The amplitudes of the recorded signals confirm the impedance difference between the two electrodes as the conducting polymer signal is twice larger than that of the metal electrode (Figure 3). Moreover, a calculation of the signal-to-noise ratio (SNR) for both electrodes demonstrates the superior SNR of the conducting polymer electrode. The SNR was calculated by taking the highest peak caused by an eye-blinking activity and the standard deviation of the noise during a period of low background activity. For the PEDOT:PSS electrode recordings, these values were 80 µV and 1.75 µV, respectively, yielding an SNR of 33 dB. The same calculation for the gold surface electrode yielded an SNR of 26 dB.

We showed that conducting polymers are adapted to the irregular scalp surface and provide low skin-electrode interface impedance. These electrodes fit with the context of long-term EEG recording, as they avoid the variation of impedance with time associated with changes in the conducting gel. The ease of attaching these electrodes to the skin makes the simultaneous recording of EEG and other techniques possible. The results indicate that conducting polymer coatings represent a viable solution for improving the performance of EEG electrodes.

References:
Microfluidic Devices for Examining Nuclear Mechanics

CNF Project Number: 2065-11
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Abstract:
It is now emerging that the physical properties of cells can in part determine to what extent cells are able to penetrate and invade tissues. The low mechanical stiffness of immune cells, for example, enables them to travel through infected tissues. However, this characteristic is also a hallmark of metastasizing cancer cells. We have developed two complementary microfluidic devices to assess the deformability of cells. In the first, cells in suspension are perfused through narrow channels; in the second, cells migrate through narrow constrictions towards a chemotactic gradient. Using these devices, we are probing the deformability of cancerous and non-cancerous cells, as well as cells with modified nuclear envelope proteins, which we hypothesize play a role in cellular mechanics and are altered in cancerous cells.

Summary of Research:
In many biological processes, the ability of cells to deform and squeeze through narrow openings is a critical and often rate-limiting step. For cancer cells, it has been shown that invading/metastasizing cells can pass through pores as small as 3 µm, but smaller pore sizes may result in the cell nucleus getting caught. Some immune cells, faced with similar obstacles, have evolved highly flexible and lobulated nuclei to facilitate passage through the endothelial cell layer in blood vessels and through other dense tissue spaces. In our lab, we have previously shown that modifications in nuclear stiffness through the alteration of nuclear envelope proteins can have dramatic effects on the ability of cells to pass through narrow constrictions.

The aim of this project is to develop novel microfluidic devices that will enable the quantification of cell deformability. We use two approaches: in the first, single cells are perfused through microscopic pores and the time needed to pass through these pores is assessed. The devices consist of small channels with precisely defined constrictions through which hundreds of cells can be perfused per minute. In the second approach, a chemotactic gradient is created across microscopic channels, enabling the observation of cells migrating through narrow constrictions under their own impetus. Cancerous and noncancerous cells will be compared in these devices, along with cells with altered expression of nuclear envelope proteins, which change their ability to transmit forces to the nucleus. The migratory ability of the cells will be assessed as they traverse the narrow spaces via either perfusion or migration. The devices are constructed by first creating a mold of the device using SU-8, then using this mold to create a polydimethyl siloxane (PDMS) replica, which is subsequently bound to a glass slide. Holes are punched into the PDMS to allow the introduction of liquids and cells, resulting in a microfluidic device.

Figure 1: Perfusion-based device design. The cells enter the device at one wide channel that bifurcates four times, creating 16 channels. The bifurcations have rounded corners to prevent cells from becoming caught on sharp edges. Round pillars are used to prevent the PDMS ceiling from collapsing and blocking the channels.
The perfusion-based device consists of 16 constriction channels in parallel with each other (see Figure 1). Cells flow through the device into these constriction channels, deform as they transit the constriction channels, and then exit the device. Each constriction channel is composed of six actual constrictions, with widths of either 3.0 µm, 5.0 µm, or 8.0 µm (depending on the cells to be studied). The constrictions are connected by areas of larger width to allow the cells to expand in between the constrictions. The height of the channels is 9.7 µm. The time needed for a cell to deform and pass through a constriction channel can be measured during microscopic observation (see Figure 2) and correlated with nuclear stiffness.

In the migration-based device, a chemoattractant gradient is formed to promote cellular migration through the constriction channels. Two large reservoirs on either side of the constriction channels provide a constant source and sink of the chemoattractant, which enables the formation of a steady gradient across the device. There are several constriction channel sizes and designs, ranging from 2.0 µm to 5.0 µm in width and from 4.9 µm to 11.5 µm in height. We have observed cells migrating through constriction sizes as small as 2.0 µm in width and 9.6 µm in height (see Figure 4) and are currently examining even smaller channel heights.

Acknowledgements:
National Institutes of Health awards [grant numbers R01 NS059348 and R01 HL082792]; the Department of Defense Breast Cancer Idea Award [grant number BC102152]; and an award from the Progeria Research Foundation.

References:
Effect of Interstitial Flow on Tumor Cell Migration
Using a 3D Microfluidic *in vitro* Model

**CNF Project Number: 2068-11**
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**Abstract:**
Interstitial flow is ubiquitous in mammalian tissue, but its effects on tumor cell invasion and metastasis are not fully understood. We have developed a PDMS-based microfluidic device that generates a pressure-driven fluid flow across type I collagen matrix, entrapped by contact line pinning. Using fluorescence recovery after photobleaching (FRAP), we demonstrate that our device is capable of providing a steady and slow (4 µm/s) flow across the collagen matrix. Using our device, we demonstrate the effects of interstitial flow on cell morphology. With proper co-culture set-up, we will also be able to model cancer cell migration from the primary tumor and into blood/lymphatic vessels. Overall, our device enables more accurate quantitative studies of interstitial flow *in vitro*.

**Summary of Research:**

**Device Design.** The microenvironment, including chemical and fluid flow conditions, is known to be critical for cancer progression [1]. To understand better how cancer interacts with its environment, systematic control over the microenvironment is important. Our device design is shown in Figure 1. The horizontal channels are used to apply interstitial flow, and our 3D collagen is introduced in the vertical channels (400 µm wide each). The collagen is confined by contact-line pinning mechanism [2]. The master mold is fabricated on a silicon wafer, with two steps of DRIE, the first one for the contact lines (5 µm), and the second for the rest of the channel (200 µm). After treated the wafer with FOTS, the master will be used to pattern PDMS. We treat the PDMS surface with oxygen plasma and then incubate the pieces in room temperature for several days to optimize the contact-line pinning effect. Once the PDMS piece is ready, it is assembled between a glass slide and a Plexiglas® top plate. Then 0.15% type I collagen is introduced into the vertical channels. Depending on the experiments, we can either have one line of collagen at the center, or two lines separated by an empty line in the middle.

**Flow Characterization.** After the collagen polymerization of the collagen, the empty space is filled with fluorescein solution, and then a syringe pump is connected to the side ports to create a flow. A dark spot is created by photobleaching the fluorescein with a 40x objective, and then we observe the movement of the dark spot with a 10x objective to obtain the flow speed across the collagen. The flow is steady and uniform in our device.

**Cells’ Response to Fluid Flow.** For cell experiments, 1 × 10⁶ cells/ml of malignant breast cancer cell line (MDA-MB-231) is mixed with the collagen, and we fill the empty channel with media after collagen polymerization. A time-course series of images at intervals of five minutes for 16 hours was taken under a bright-field microscope (Olympus IX 81 and Hamamatsu Orca-ER) in an environmentally-controlled chamber (37°C, 5% CO₂, and 90% humidity). Tumor cells possess a round shape when they are introduced with the collagen. Without flow, the majority of them grow into an elongated shape, which is a phenotype called mesenchymal. We found that with a physiological relevant interstitial flow (3.5 µm/sec), the percentage of the mesenchymal cells is greatly reduced, and the ratio is further lowered with higher flow rate (11.5 µm/sec), as shown in Figure 2. The molecular mechanism is currently being investigated.

**Endothelium Layer for a Complete *in vitro* Model.** We also demonstrate that our system can be used for endothelium co-culture. With the two-line collagen configuration, we are able to grow a layer of HUVECs in the center channel. After two hours of incubation in a 37°C, 5% CO₂, and 90% humidity environment with no flow in the center channel, a slow flow of HUVEC growth media is applied to maintain the growth of cells for two days. The results are shown in Figure 3.

**References:**
Figure 1: Illustration of our contact line pinning device. (B) shows the area marked by the white dashed box in (A).

Figure 2: Interstitial flow prevents cells to form an elongated shape. Insets are typical amoeboid (round) and mesenchymal (elongated) cells.

Figure 3: An endothelial layer can be cultured so a vessel is next to our collagen matrix.
Reactivity of Monolayer CVD Graphene Imperfections Studied Using Scanning Electrochemical Microscopy

CNF Project Number: 1283-04
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Abstract:
We are interested in using scanning electrochemical microscopy (SECM) to study the chemistry of graphene. First we discuss the study of the local chemical reactivity of graphene. Then we show fabrication of a 500 nm SECM probe electrode to improve the resolution of SECM imaging.

Summary of Research:
We are using scanning electrochemical microscopy (SECM), a quantitative scanning probe technique that allows for the measurement of the (electro)chemical reactivity of substrates with spatial resolution. One key problem of chemical vapor deposited (CVD) graphene, yet to be solved, is the defective regions created during fabrication and processing. McCreery and co-workers reported that the edges and defects of highly oriented pyrolytic graphite (HOPG) are chemically more active when compared to the basal plane of HOPG [1]. The difference in the chemical reactivity of defects could hinder our understanding of graphene chemistry and creates a hurdle in the application of graphene. We have used scanning electrochemical microscopy to examine the local reactivity of graphene.

By biasing the probe electrode potential and the substrate electrode potential at reducing and oxidizing potentials, respectively, a redox-active molecule can be reduced and oxidized, generating a feedback current. The variations of the feedback current provide us information about the local reactivity. The SECM image in Figure 1 shows the spatially resolved reactivity of a mechanically induced defect. The feedback current of the edges of graphene is much higher than that of the general area of graphene. The projection of the SECM shows clearly the differences in feedback current between graphene (green), Si/SiO$_2$ (dark blue), and graphene edges (red). The chemically induced defect was created by oxidizing the graphene with a solution of NaOCl. The edges have higher feedback current than the general area of graphene as observed in the mechanically induced defect (Figure 2). The optical images show no obvious morphological features at the edges of the two kinds of defects.

The spatially resolved SECM images provide a direct way to understand the chemical reactivity of graphene, however, the resolution of the images is approximately 7 µm. The resolution of the SECM images is determined by the radius of the SECM probe electrode. In this case, we used a 7.5 µm radius Pt electrode.

In order to improve the resolution of the SECM images, a smaller probe electrode has to be fabricated. The thickness of the insulating glass has to be less than ten times the radius of the Pt in order to allow the diffusion of the redox mediator. Figure 3 shows an SEM image of the Pt probe electrode of a radius of 500 nm.

References:
Figure 1: Optical (left) and SECM (right) images of a mechanical defect. No obvious morphological feature was seen at the edge of the graphene in the optical image. The reactivity at the edge of graphene is higher (red) compared to the general area of graphene (green). The exposed Si/SiO$_2$ substrate is in blue. (See full color version on inside front cover.)

Figure 2: Optical (left) and SECM (right) images of a chemical defect. The reactivity at the edge of graphene is higher (red) compared to the general area of graphene (yellow). The exposed Si/SiO$_2$ substrate is in green. (See full color version on inside front cover.)

Figure 3: SEM image of a Pt probe electrode. The radius of the SECM probe electrode is approximately 500 nm.
Chemical Bonding across the Periodic Table at High and Ambient Pressures

CNF Project Number: 1371-05
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Abstract:
Part of our research is devoted to the properties of hydrogen-rich materials, especially under high pressure. In this area, we have built on previous work at the Cornell NanoScale Facility to further our understanding of the W-H phase diagram [1]. We also searched for new stable ice phases at very high pressures [2], and contributed to a deeper understanding of the physics and chemistry involved in some of the high-pressure phases of pure hydrogen [3]. We also studied the ternary phase diagram of the light elements Li-Be-B, which involved comprehensive studies of the binary phases Be-B and Li-B as well [4].

Summary of Research:
1. Molecular Models for Tungsten Hydrides. At atmospheric pressure, some tungsten hydrides exist as discrete molecular complexes in low temperature matrices, but are very reactive and cannot be isolated in bulk at room temperature. Under pressure, one observes WH in experiment at room T, and calculations indicate several other stoichiometries are stable, all extended and not molecular. This work aimed at constructing a bridge between discrete molecules and the pressurized extended structures, with the emblematic example of WH₆. The equilibrium structure, known as a distorted trigonal prismatic molecule, is likely to be a good acceptor. In numerical experiments, discrete WH₆ molecular complexes are allowed to interact with each other at T = 0K and P = 1 atm. They spontaneously rearrange to form 1D-chains, with the W centers adopting a tricapped trigonal prismatic environment. The electronic structure of these chains is examined and related to that of the monomer and extended solids [1].

2. High Pressure Ice Phases. From computational evolutionary structure searches, we find a sequence of new stable and meta-stable structures for the ground state of ice in the 10…50 Mbar regime, in the static approximation [2]. We find a new Pmc2₁ phase to be stable at P = 9.3 Mbar, followed by a transition to a high-coordinated P2₁ structure at P = 13 Mbar. These new structures are insulating — chemistry burns a deep and (with pressure increase) lasting hole in the density of states near the highest occupied electronic levels of what might be component metallic lattices. Metallization of ice in our calculations occurs only near P = 48 Mbar, where the intrinsically metallic C2/m phase becomes most stable. We analyze carefully the geometrical changes in the calculated structures, especially the buckling at the H in O-H-O motifs. In the Mbar pressure regime, zero-point energies much larger than typical enthalpy differences suggest possible melting of the H sublattice, or even the entire crystal.

Figure 1: One-dimensional chain of WH₆ stoichiometry, with a local tricapped trigonal prismatic arrangement around W atoms. The dashed lines indicate the frontiers of the unit cell [1].
3. Understanding Hydrogen Under Pressure. Dense hydrogen, despite its apparent simplicity, is a challenging system for experimentalists and theoreticians alike. But understanding this fundamental system remains an important area of study, as dense hydrogen comprises a large fraction of the universe in the form of stars and planets. Results of both experimental and theoretical studies of dense hydrogen reveal a surprising richness of behavior in this complex and intriguing system. Our results, published in a series of four papers [3], present a fresh look at molecular hydrogen under pressure and its transition from a molecular to a monatomic, metallic state. Using a numerical laboratory of the best static structures calculated for hydrogen, we find in it an approach to metallization that preserves some features of diatomic molecules as well as opposing effects influencing distances in the compressed solid — a physical one of containment, and an orbital/chemical one of essentially donor-acceptor bonding between hydrogen molecules (making a connection to the side-on bonded dihydrogen organometallics). We also explore a model with lovely symmetry that makes a single-parameter transition between a molecular solid and a highly compressed monatomic one.

4. Ternary Light Element Compounds. Beginning an in-depth analysis of binaries and ternaries in the Li/Be/B system, we examined the static structures and electronic properties of LiBeB (i.e., 1:1:1) over a range of pressures [4]. This as yet unknown compound is predicted to possess a stable ground state at P = 1 atm and some higher pressures. As the pressure rises, LiBeB goes through a diverse series of structures, beginning with metallic structures which feature chains and layers of atoms, progressing to structures built on “colorings” of the Laves phases, and containing helical arrangements of boron atoms, on to high pressure phases that are ternary variants of a bcc lattice. We find some curious features in the LiBeB structures, these include near-icosahedral coordination, independent of atom type; in a range of pressures a resemblance of the total DOS to that of metallic Be; and also a Dirac surface.

References:
High Temperature Micro-Reactors for Stable Isotopic Analysis

CNF Project Number: 1592-07
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Abstract:
Micro-reactors that combust organics at high temperature, were fabricated in various configurations in high purity fused silica using photolithography techniques and novel HF deep wet etching. These robust reactors were used to interface a gas chromatograph with a cavity ring-down spectrometer to enable analysis of the stable carbon and hydrogen isotopic composition of light hydrocarbons.

Summary of Research:
The measurement of the stable isotopic composition (i.e. of C, N, O, S, H) in compounds is used for sourcing their geographic, chemical, and biological origins in a variety of applications such as food authentication, geochemistry, and forensics. In order to measure the stable isotopes of individual compounds in mixtures, gas chromatography (GC) is coupled to isotope ratio mass spectrometry (IRMS) [1] or, more recently, to cavity ring down spectroscopy (CRDS) [2]. This technique requires compounds to be chemically converted to an inert gas, such as combustion of organic molecules to CO$_2$ for $^{13}$C/$^{12}$C measurement, using on-line reactors before the IRMS or CRDS. The conventional reactors, which are generally comprised of ceramic or fused silica tubes, are relatively fragile. In the CNF, we have been developing microfabricated micro-reactors (MFMR) as a more robust alternative for such systems [3] and potentially for coupling of IRMS to more advanced separation such as fast GC and comprehensive 2D GC (GC$x$GC) [4]. Coupling of GC to CRDS using MFMRs (GC-MFMR-CRDS) would also enable systems that are less expensive, simpler, and easily made portable.

In previous work, we developed the methodology to create MFMR with small (S-MFMR) and large (L-MFMR) inner diameter hot zone channels. S-MFMR were comprised of hot zone channel diameters of ~ 200 µm and tapers to port channels with diameters of ~ 400 µm, which would be suitable for fast GC and GC$x$GC peak shapes. As shown in Figure 1, L-MFMR were comprised of hot zone and port channel diameters measuring ~ 500 µm and ~ 750 µm, respectively, which are suitable for normal GC peak shapes.

The methodology generally consisted of photolithography techniques with isotropic HF deep wet etching in pure fused silica wafers, followed by thermal bonding. Cu/Ni metal wire was inserted into the hot zone region and then MFMR ports were sealed to GC fused silica (FS) tubing. The Cu/Ni was oxidized at 600°C, which was then used as a source of O$_2$ at 925-1070°C for combustion of organics.

A unique GC-MFMR-CRDS system for simultaneous C and H isotope measurements from CO$_2$ and H$_2$O vapor generated from combustion, which is not possible with an IRMS, was assembled and characterized by our collaborators at Stanford University, and Picarro, Inc. (see Acknowledgements). The MFMR was housed in a high temperature furnace held at 1070°C. A mixture of natural gases were analyzed due to their significance in exploratory and routine oil drilling and the suitability of the instrumental setup for potential field measurements.

For isotope ratio measurements, the $^{13}$C/$^{12}$C and $^2$H/$^1$H of a sample is measured and values are reported in δ notation with respect to an international standard, in units of per mil (∆‰)
and expressed as $\delta^{13}C$ or $\delta^2H = (R_{\text{spl}} - R_{\text{STD}})/(R_{\text{STD}}) \times 1000 \, \%e$, where $R_{\text{spl}}$ is the isotope ratio of the sample and $R_{\text{STD}}$ is the isotope ratio of an international standard. The measured $\delta^{13}C$ and $\delta^2H$ values resulted in precisions of $SD(\delta^{13}C) < 0.8\%e$ and $SD(\delta^2H) < 2.0\%e$, and deviation from true values no greater than $0.5\%e$ for $\delta^{13}C$ and $1.3\%e$ for $\delta^2H$. As shown in Figure 2, GC separated peak shapes for CO$_2$ were acceptable; however, those for H$_2$O vapor were a bit wide due to its sticky nature.

In early development, MFMR input/output port connections were sealed with low temperature sealant, where the port seals were stable at < 100°C and is sufficient for gases and volatile compounds and is useful for many applications. For applications involving low volatility compounds, the ultimate goal, port connection seals require to be leak free at temperatures > 400°C to allow the low volatile compounds to enter the MFMR by preventing condensation in “cold” regions. Through experience with high temperature applications, we discovered that port connections are the limiting factor in lifetime and robustness. In our investigations, high temperature ceramic sealants, rated to temperatures of up to 1650°C, were found to be too porous to prevent gas leaks. Some resins, epoxies, and silicone materials succeeded in making seals at room temperature; nevertheless, failed to retain hermetic seals at higher temperatures (> 100-300°C) at prolonged periods (few hours to three days), depending on the material. Further work is required to address this limitation.

Acknowledgements:

The CRDS work was done in collaboration with Nabil Saad, Bruce Richman, and Eric Crosson from Picarro Inc. and Douglas Kuramoto and Richard N. Zare from Stanford University.

References:


Figure 2: Chromatograms for a mixture of methane, ethane, and propane injected into the GC-MFMR-CRDS instrument showing the detection of (a) $^{12}$C$^{16}$O$_2$ and (b) $^1$H$_2^{16}$O.
**Single Molecule Catalytic Studies of Nanoscale Metal-Metal Junctions**

**CNF Project Number:** 1844-09  
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**Abstract:**

We aim to study catalysis at the interface of a heterogeneous nanoscale metal-metal junction, with the goal of determining what effect the junction has on activity compared with either metal, and also seeing how far the interface effect extends into either metal. The interface is made by electrodepositing striped gold and platinum rods into an alumina template. Alternatively, photolithography will be used to generate orthogonally intersecting gold and platinum stripes on a quartz surface. Catalytic reaction product formation will be detected and imaged using optical fluorescence sub-diffraction imaging.

**Summary of Research:**

In order to study the catalytic properties of two-domain, bimetallic nanostructures, we have been using the CNF to help fabricate a nanoscale gold-platinum interface in a couple of ways. The first way is based on alumina templated electrodeposition, in which a nanoporous alumina film is grown by anodizing a piece of aluminum. The alumina layer is removed and the pores are opened up from one end to the other by dissolving the thin barrier layer left at the bottom. An evaporator is used to put a thin silver film onto one side which serves as the working electrode for the electrodeposition of gold and then platinum. The particles are removed by selectively dissolving the alumina template and the silver layer. This method for making two-domain rods was pioneered by Thomas Mallouk and coworkers [1]. The second method to getting a nanoscale to microscale gold-platinum interface uses photolithography in which sub-micron width strips of gold and then platinum are evaporated onto a quartz surface in an orthogonally intersecting manner using lift-off lithography.

The bimetallic nanostructures are then coated in a porous silica shell to allow for optical detection of fluorescent product molecules using sub-diffraction fluorescence microscopy [2]. This technique allows us to capture a single catalytic turnover with spatial resolution of about 20 nm. We aim to quantify the catalytic rate in different domains within a single nanostructure, which will allow us to determine the effect on catalytic rate due to the presence of the junction.

**References:**


![Figure 1: SEM of nanoporous alumina used as a template for electrodeposition.](image-url)
Mid-Infrared Transparent Substrate for Alkylsiloxane Monolayers

CNF Project Number: 1936-10
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Abstract:
An infrared (IR) and visible transparent substrate compatible with alkylsiloxane monolayers was fabricated by coating a calcium fluoride window with SiO₂ by atomic layer deposition (ALD). Octadecylsilane (ODT) monolayers adsorbed to the SiO₂-covered CaF₂ substrates were found to be equivalent to ODT monolayers on glass in terms of water contact angle and vibrational spectra. The substrate was also used as a platform for thiocyanate, t-butyl ester, and hydroxyl-terminated monolayers.

Summary of Research:
Self-assembled monolayers (SAMs) can be used to create highly ordered surfaces having tunable chemical functionalities. Alkylsiloxane monolayers are simple to apply, stable in a wide range of physical and chemical environments, and do not degrade appreciably even over periods of months [1]. These attributes make them ideal platforms for probing the interaction of water with various surfaces. However, the most important substrates compatible with alkylsiloxane monolayers, the silicon oxides, are not transparent through most of the infrared region. Since water also has strong absorption bands in the IR region, spectroscopy of vibrational transitions at the water-alkylsiloxane SAM interface is greatly complicated in both transmission and reflection geometries.

In this project CaF₂ (transparent 0.2-10 μm) was coated with a thin film of SiO₂ to create an IR and visible transparent substrate capable of functionalization by alkylsiloxane monolayers. Clean, optically polished CaF₂ was coated with 10 nm of SiO₂ with the Oxford ALD FlexAL. Deposition of SiO₂ on bare CaF₂ results in a substrate with SiO₂ on both sides. To modify a single side of the CaF₂, a protective mask of photoresist was spin-coated onto one face. After ALD of SiO₂, the substrate was sonicated in a stripping solution to remove the mask. An octadecylsilane monolayer was then applied to the substrate by adsorption from dilute solution.

The stationary contact angle of water on OTS was found to be the same whether the SAM was deposited on glass or SiO₂-coated CaF₂. IR spectra of the modified CaF₂ show that the thin SiO₂ layer did not absorb in the IR significantly above 1250 cm⁻¹ (Figure 1). In addition, IR spectra of OTS on the modified CaF₂ substrate matched those of OTS on glass.

The SiO₂ deposited on the CaF₂ was later increased to a nominal thickness of 50 nm for a more robust film. X-ray photoelectron spectroscopy in conjunction with argon ion milling was used to determine the elemental profile of this substrate. It indicated a transition from SiO₂ to CaF₂ over ~ 10 nm, and a film thickness of ~ 39 nm (Figure 2).

The substrate was also used as a platform for thiocyanate, t-butyl ester, and hydroxyl terminated SAMs using silane chemistry. In the future new functionalities and mixed monolayers will be applied.

References:
Figure 1: Absorbance of 1 mm thick bare CaF$_2$ (dashed line), 1 mm thick SiO$_2$ (dotted line) and 10 nm SiO$_2$ on CaF$_2$ (solid line) in the IR region.

Figure 2: Elemental profile of SiO$_2$ film (nominal thickness 50 nm) on a CaF$_2$ substrate as measured by x-ray photoelectron spectroscopy and ion milling.
Formaldehyde Hydration Reaction and Transition State

CNF Project Number: 2067-11
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Abstract:
We use quantum chemistry methods available in the Gaussian09 package to study the properties of the transition state of the formaldehyde hydration reaction in water.

Summary of Research:
We have used the Gaussian09 program to find the enthalpy and energy of cyclic transition states for the dehydration of methanediol to formaldehyde. We have performed these calculations in a continuum dielectric with varying amounts of explicit water molecules. We note that cluster studies of water surrounded by a continuum description of water are difficult because the continuum waters are differently treated than the explicit water molecules, with no accounting for vibrational effects. For this reason, we include multiple water molecules in both the transition state cluster and the formaldehyde cluster to reduce these effects.

We have learned that both large numbers of waters, large basis sets, and coupled cluster singles and doubles are all necessary to produce results which approach experimental values for the enthalpy of the transition state. We also note that other attempts at finding the transition state have suffered from insufficient basis set size and level of theory [1], and yet other studies have focused on the gas phase reaction, without catalysis by water [2]. We find that the configuration space is large—there are a large number of local energy minima, and we are currently unsure as to whether we have found the lowest energy configurations for the transition state, the reactants, and the products.

However, we have identified a number of promising structures which give us insight into the actual transition state structure. The transition state appears to be methanediol with a ring of water molecules which exchange protons, and an additional water molecule hydrogen bonding to one of the alcohol groups of methanediol. Additionally, it appears that the transition state is closer in structure to the formaldehyde than to the methanediol. Further work will help to determine additional properties of this transition state.

References:
A Ferroelectric and Charge-Based Hybrid Nonvolatile Memory

CNF Project Number: 715-98
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Abstract:
We introduce a new one-transistor nonvolatile memory, which benefits from the two seemingly unrelated memory mechanisms; namely ferroelectric (FE) polarization and nonvolatile charge injection. The hybrid memory demonstrates larger memory window and reduced depolarization field during retention compared to conventional FE-FETs. This summary discusses the working principle, device fabrication and experimental results of the proposed memory device.

Device Concept:
Figure 1 (a) shows the hybrid memory cell with integration of charge storage layer above the ferroelectric (FE) [1, 2]. During program condition (Figure 1 (b)), negative bias at the gate aligns the FE polarization to the applied field and also initiates tunneling of electrons from the gate into the charge trap layer through the thin tunnel oxide. The stored charge reduces the depolarization field in the FE and the FE polarization reduces the field in the tunnel oxide during retention. This combined effect results in longer retention of the programmed state (Figure 1 (c)). Erase operation is performed by applying a positive bias the gate which reverses the polarization of the FE as well as removes electrons from the storage layer.

Device Fabrication:
One-transistor hybrid memory devices were fabricated with P(VDF-TrFE) as the FE and HfO2 as the charge trap layer by a modified gate-last process to accommodate the low processing temperatures after deposition of the polymer layer. The NMOS device comprised of SiO2 bottom and tunnel dielectric of 10 nm and 5.4 nm respectively, 2.5 nm thin charge trap layer and 35 nm thick P(VDF-TrFE) deposited by spin coating. Further details of the fabrication process may be obtained in [3].

Experimental Results:
Hybrid devices show larger $\Delta V_{fb}$ against FE-FETs for $V_{prog} < -10$ V due to the contribution of injected electrons as seen from Figure 2. The equivalent gate-inject Flash does not show $\Delta V_{fb} > 1$ V for all $V_{prog}$ due to the inability to store over $5 \times 10^{12}$ cm$^{-2}$ electrons in the storage node. Figure 3 shows the program efficiency comparison between the two devices. Hybrid device consistently shows higher $\Delta V_{fb}$ on account of injected charge for all pulse widths. It should be noted that the time scales for the PVDF switching and charge tunneling are comparable and therefore the two memory mechanisms cannot be distinguished from one another. However, hybrid devices show longer retention of programmed state due to the reduction in depolarization field in the FE, as seen from Figure 4. The trap layer also acts as an effective sink for electrons that are captured in the FE during program condition and which gradually leak out during retention thereby improving retention.
Summary:
In conclusion, we have demonstrated a novel nonvolatile memory technique that employs two independent memory mechanisms. The device operation was designed to offer mutually complementary interaction between them which results in larger memory window and longer retention times.

References:
Fabrication and Testing of Chemoreceptive Neuromorphic Neuron Transistors

CNF Project Number: 752-98
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Abstract:

Novel architecture and layout considerations were employed to fabricate chemical imaging devices on a complementary metal oxide semiconductor (CMOS) compatible platform. Arrays of Chemoreceptive Neuromorphic Transistors (CνMOS) were created, and silicon nitride was employed as a passivation layer. These devices will be used for a wide variety of spatially resolved biochemical sensing studies, such as cell monitoring and DNA detection.

Summary:

The idea of CMOS compatible chemical sensing was first conceived and demonstrated by Bergveld, et al., in 1970 [1]. Since then similar devices have been explored, demonstrating low limit of detection, as well as high spatiotemporal resolution [2]. Most of these devices employ an architecture identical to Bergveld’s original design, which suffers from problems such as sensor drift. This architecture also requires a reference electrode in solution, reducing the viability for long term in vivo monitoring. This work hopes to build on these previous studies, while making use of novel Chemoreceptive Neuromorphic Neuron MOS Transistor design.

CνMOS consists of a metal oxide semiconductor (MOS) transistor utilizing an extended floating gate. Two input gates; the control gate and the sensing gate couple to this floating gate, and establish its potential. While the control gate is driven by a voltage source, an electrically floating biological fluid covers the sensing gate. The resultant voltage on the floating gate, and thus the current in the channel, is related to the chemical conditions at the sensing gate-liquid interface.

While previous efforts in this area have focused on the operation of a single device [3, 4], this study hopes to focus on 2 × 2 sensor arrays. First, we have designed 2 × 2 arrays, which will fit entirely underneath one mammalian cell. This allows us to “image” a cell for electrochemical activity, and can give information related to the spatial nature of cellular signaling.

In addition, we have fabricated arrays with larger dimensions. By using these devices, we can obtain information related to the signaling patterns of cellular populations.

Passivation and chemical fouling degrade signal reliability, and are perennial problems in many CMOS chemical sensors. We attempted to overcome this problem by depositing a silicon nitride passivation layer covering most of the chip area. This nitride layer was subsequently masked and etched to expose the sensing gates to the ambient chemical environment. This protection layer, along with the lateral isolation between the transistor and the fluid, serve to effectively isolate the device from chemical contamination and fouling. While the process used to manufacture these devices is CMOS compatible, production in the Cornell NanoScale Facility (CNF) allows more fine control over fabrication conditions.

Recently, pH curves have been obtained on devices similar to those fabricated in the CNF. Due to the smaller size of the control gate compared to the sensing gate large shifts in threshold voltage were observed. However, when the capacitive anisotropy was corrected for, these devices were shown to have a nearly Nernstian response.

References:

Figure 1: Optical micrograph of CnMOS array.

Figure 2: The pH response of CnMOS devices.

Figure 3: CnMOS schematic.
Improving the GaSb/High-k Oxide Interface Using an InAs Capping Layer

CNF Project Number: 780-99
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Abstract:
A highly reactive GaSb surface was passivated by growing a thin epitaxial InAs capping layer to improve the GaSb/high-k oxide interface. MOSCap structures were fabricated with and without InAs to study improvements in C-V characteristics and interface trap state density ($D_{it}$). Al$_2$O$_3$ and Al$_2$O$_3$/HfO$_2$ high-k oxides were deposited using atomic layer deposition (ALD) in the Oxford ALD FlexAL and annealed in forming gas (FG) at 350°C and 450°C. Optimized processing with (NH$_4$)$_2$S pre-treatment and 350°C anneal resulted in low frequency dispersion, improved stretch-out and a large reduction in $D_{it}$ for both oxides. The interface with ALD oxides was thermally stable to 450°C.

Summary of Research:
Alternative p-channel materials are being investigated for their high hole mobility and low switching voltage potential in future CMOS circuits. III-Sb based heterostructures offer a promising replacement material that utilizes quantum confinement and bi-axial strain engineering to increase hole mobility [1]. The main challenge with GaSb is the high density of trap states formed at the III-V/high-k interface when exposed to ambient conditions. Terminated surface bonds react with oxygen and form states within the bandgap that cause Fermi level pinning. InGaAs III-V/high-k interfaces show a reduction in $D_{it}$ when using chemical pretreatments and ALD of Al$_2$O$_3$ with trimethylaluminum (TMA) as a precursor [2]. During TMA exposure to the native III-V surface, a ligand exchange process occurs which reduces oxygen on the surface. This forms a high quality interface with a low $D_{it}$ and an unpinned Fermi level.

This project implemented a thin 2 nm InAs interface layer on a thick 1 µm n-GaSb (n = 5 x 10$^{17}$cm$^{-3}$ by Te) MOSCap structure grown on a n-GaAs substrate using MBE (Figure 1). Chemical pretreatments were used at CNF immediately prior to ALD of high-k oxide. An HCl:H$_2$O pre-treatment was used to remove native oxides or (NH$_4$)$_2$S:H$_2$O was used to remove oxides and passivate surface dangling bonds. Quality gate oxide was needed to allow for proper gate control while maintaining a low leakage current. The Oxford ALD FlexAL tool at CNF was used with the guidance of Vince Genova to deposit 10 nm oxide after surface pretreatments. Al$_2$O$_3$ was deposited using 100 cycles of TMA and H$_2$O, and an alternative stack was deposited using ten cycles of Al$_2$O$_3$ and 100 cycles of TMAH and H$_2$O to deposit Al$_2$O$_3$/HfO$_2$. This gate oxide stack takes advantage of the initial TMA “self-cleaning” effect and the high permittivity of HfO$_2$. 

Figure 1: TEM image showing the GaSb/InAs/Al$_2$O$_3$ gate stack with EDS scan to confirm the InAs interface layer.

Figure 2: n-GaSb/Al$_2$O$_3$ MOSCaps without (a) and with (b) InAs showing improvement in C-V characteristics with the InAs interface layer.
These oxides were then annealed in FG at 350°C and 450°C, and 120 nm Ni gate metal was deposited using e-beam evaporation.

MOSCaps with InAs (HCl, no anneal) showed a significant reduction in C-V stretch-out (dC/dV)max at 500 kHz compared to bare GaSb surfaces (3.0%/dec to 2.2%/dec) (Figure 2). D_it extracted using the High-Low method [3] showed a 10x reduction throughout the GaSb bandgap using the InAs interface layer. XPS was performed on InAs/GaSb with 1 nm Al_2O_3 and showed a large reduction in metal-oxides at the III-V/Al_2O_3 interface after (NH_4)_2S compared to a standard HCl treatment. An (NH_4)_2S passivation before ALD and FG annealing at 350°C resulted in an optimized fabrication process showing minimized frequency dispersion (1 MHz to 1 kHz in accumulation) of 1.1%/dec with Al_2O_3 and the best stretch-out of 0.8 V^−1 using Al_2O_3/HfO_2. The D_it “hump” seen in Figure 2b was reduced after passivation and annealing in FG. Dit was extracted using High-Low, Conductance [4] and Terman [5] methods, and showed a large improvement compared to samples without InAs. Al_2O_3 showed a D_it ≈ 2-3 × 10^{12} cm^−2 eV^−1 near the GaSb conduction band edge. Low temperature measurements effectively slowed trap response to allow for probing D_it near the GaSb conduction band. Sub-threshold slope extracted from the conductance method resulted in 200 mV/dec showing an unpinned Fermi level suitable for MOSFET gate operation. P-GaSb/InAs MOSCaps were also fabricated, but showed fast generation/recombination through a high density of bulk defect states.

Annealing conditions of both ALD oxides were optimized using a FG anneal at 350°C. This anneal improved breakdown voltage (9V) and leakage current (down to pA) and led to improved C-V characteristics. Annealing Al_2O_3 at 450°C resulted in interface degradation. The breakdown voltage of these MOSCaps was 2V lower than un-annealed samples and showed poor gate control (Figure 4). A large degradation in both frequency dispersion (5%/dec) and stretch-out (0.2 V^−1) was seen after this high temperature anneal, showing the need for a low thermal budget process for antimonides.

InAs can also be used as etch stop layer in “gate-last” p-channel (In)GaSb MOSFET processing to avoid implantation and high temperature activation annealing. A pre-grown p++ GaSb top contact layer is selectively etched over InAs using aCl-based plasma due to the fact that In-Cl_3 is not volatile at room temperature.

Future work will include implementing the optimized GaSb/InAs/Al_2O_3 ALD gate stack process developed at the CNF into this “gate-last” process to ensure an unpinned Fermi level and proper gate control of the MOSFET channel.

References:
Memory Effects Induced by Space Charge Polarization in a SmNiO₃ Film

CNF Project Number: 804-99
Principal Investigator: Prof. Sandip Tiwari
Users: Sang Hyeon Lee, Moonkyung Kim

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Abstract:
This report presents the first characterization of a correlated oxide SmNiO₃ (SNO) used in electronic device structures. In a MOS capacitor with a gate insulator of a SNO film sandwiched between two SiO₂ layers, memory effects are observed. The response time of polarization is over a microsecond. This implies that space charge polarization is dominant. Due to the instability of Ni³⁺ valence state, oxygen vacancies appear to lead to a polarization effect. In a MOSFET, counterclockwise hysteresis is observed, which is consistent with polarization switching effect. The stored information decays gradually with the retention time of around ten seconds.

Summary of Research:
Rare-earth nickelates RNiO₃ (R stands for rare-earth elements such as Pr, Nd, and Sm) exhibit metal-insulator phase transition (MIT) as a function of temperature [1]. The MIT temperature increases with the size of the element [2]. At high temperature, the RNiO₃ compounds are metallic with orthorhombic symmetry. As temperature decreases, charge disproportionation occurs between Ni sites, reducing the symmetry to monoclinic and resulting in a transition to an insulating state [3]. SNO is of particular interest because it is the first nickelate with MIT temperature above room temperature. Owing to instability of 3+ oxidation state of Ni, oxygen vacancies are present in a SNO film [2]. They lead to space charge creation and influence the conduction properties of SNO thin films. Space charge can affect the dielectric property of a SNO film. In this work, the dielectric properties of SNO are examined and the possibility of a memory application is also investigated.

The MOSFET structure in the experiment is similar to that of a Flash memory and composed of a transistor with a SNO film employed as an intermediate gate insulator. The SNO film is sandwiched between thermal SiO₂ and ALD SiO₂ layers on a p-type silicon substrate. The MOS capacitor is the same gate structure as the MOSFET without Source and Drain (S/D) contacts except that it has an n⁺ silicon substrate.
Figure 1 shows the capacitance-voltage characteristics of a MOS capacitor at various temperatures. The dielectric constant of SNO can be extracted from the structural parameters and capacitance. It is ~ 26 at room temperature. The extracted dielectric constant is in good agreement with optical measurements of the closely related material NdNiO$_3$ [4]. As shown in the figure, the hysteretic behavior of capacitance is observed. Capacitance during a forward sweep increases initially and has a peak value at a certain gate voltage, around -1.6 V and then decreases. During a backward sweep, similar effect happens, but its magnitude is lower. Similar hysteretic behavior was also observed in a SrTiO$_3$ (STO) film. Buniatian, et al., explained that the hysteresis effects are caused by excess space charges, such as oxygen vacancies, and proposed a model based on the Poole-Frenkel charge trapping/de-trapping mechanism [5].

Figure 2 shows the results of peak capacitance variation as a function of frequency. As shown in the figures, the capacitance and its hysteresis window increase as the frequency decreases. This implies that the dielectric constant of SNO changes with the frequency. The permittivity of a dielectric material depends on the frequency of the applied field since there are various polarization mechanisms and each type of polarization has a different response time. For example, space charge polarization has a relaxation frequency of around 100 kHz. In case of SNO, the response time of polarization is over 1 µsec. Therefore, space charge polarization is dominant in the permittivity of the SNO film.

As expected from the results of a MOS capacitor, hysteretic behavior of capacitance between gate and S/D is observed as shown in Figure 3. As the gate voltage is cycled between -10 and 10 V, the capacitance cycles counterclockwise. This hysteretic behavior is consistent with polarization switching effect. Since the threshold voltage shift happens even in the absence of channel current, it is a gate field effect. In addition, it occurs at room temperature. Therefore, this effect is independent of the thermally-driven phase transition of SNO. With the decrease of frequency, threshold voltage decreases substantially, and hysteresis window and inversion capacitance increase slightly. This implies that polarization change makes an effect on the change of threshold voltage dominantly. These results, their existence with a gate field, their existence in the absence of heating, and frequency dependence, can be explained through space charge polarization mechanism in the SNO film.

In measurement of state retention characteristics, capacitance at zero gate bias is monitored after applying a gate pulse to suppress data interference. As shown in Figure 4, the state decays gradually. It is believed that the depolarization field degrades the polarization of a SNO film. The state retention time is of the order of ten seconds.

References:
Wirebonding and Packaging of Various Integrated Circuits

CNF Project Numbers: 1088-02, 2121-12
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- Integrated circuits (IC) fabricated in commercial processes require wirebonding for testing.
- Several IC designs are packaged and tested including radio designs, data converters, and biomedical monitors.

Figure 1: An ultra-low-power ADC for real-time neurochemical monitoring with fast-scan cyclic voltammetry wirebonded in CNF.
High Breakdown Voltage AlGaN/GaN HEMTs for Power Switching Applications

CNF Project Number: 1230-04
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User: Ekaterina Harvard

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Primary Source of Research Funding: Office of Naval Research
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Abstract:
The fabrication of an AlGaN/GaN high electron mobility transistor (HEMT) with small feature sizes and an off-state breakdown voltage of 440 V is presented. This breakdown voltage was measured on a 0.5 µm gate-length device with a small (5 µm) gate-drain spacing with a standard HEMT layout, without using any field plates. This is achieved through the use of AlSiN as a passivating dielectric. The device also had an 18.9 GHz $f_T$ and 40 GHz $f_{MAX}$.

Summary of Research:
Aluminum silicon nitride (AlSiN) is showing promise as a passivation material for AlGaN/GaN high electron mobility transistors (HEMTs) [1]. The negative fixed charge provided by the dielectric allows for controlled depletion of the ungated portions of the channel, which suggests that this passivation scheme would allow for higher operating voltages without the use of a field plate. The epitaxial structure of the devices consisted of an AlN nucleation layer, followed by a 0.5 µm GaN buffer, a 220Å AlGaN barrier with 24.5% Al fraction, and a 20Å GaN cap grown on semi-insulating SiC. The device active areas were isolated with a Cl2/BCl3/Ar reactive ion etch (RIE). A passivation layer of 35 nm of AlSiN was deposited by low pressure chemical vapor deposition (LPCVD) at 750°C using trimethylaluminum, dichlorosilane, and ammonia precursors. The ohmic source/drain contact windows were defined by electron beam lithography using the JEOL 9300 and etched using a CF4 RIE, and the V/Ti/Al/Mo/Au ohmic metal stack was deposited. A 20 nm cap of SiN was deposited by plasma-enhanced chemical vapor deposition (PECVD) to encapsulate the ohmic contacts before annealing at 850°C.

To create a T-gate, a stack of 75 nm of AlN and 120 nm of SiN was deposited by reactive sputtering and PECVD, respectively. The Schottky gate was defined by e-beam and the stack was etched with first SF6/Ar then BCl3/Ar RIE, then SF6/Ar again to cut through the AlSiN dielectric. The gate region was then annealed at 700°C, and the Ni/Au contacts were evaporated. The AlN sacrificial layer was then removed in 726 MIF developer. The final pad metal layer was defined by optical lithography and the ohmic cap was etched through with an SF6/Ar RIE and the Ti/Cu/Ti/Au pads were evaporated. See Figure 1 for a device cross-section.

The devices had a two-fingered “U” layout. The total gate width was 400 µm, the gate lengths were 0.5, 1, and 1.5 µm. The gate-drain spacings were 3 and 5 µm. These feature sizes are significant because most high breakdown voltage HEMTs have gate drain spacings in excess of 10 µm [2].

Figure 1: Schematic cross section of the device structure.
Figure 2 shows the combined DC measurements of the parameter space of a typical device, including the output curves, constant power measurements, and high voltage off-state breakdown measurement. The highest $f_T$ of 18 GHz and $f_{MAX}$ of 40 GHz was obtained on a 0.5 µm gate length device with a 5 µm gate-drain spacing. High voltage measurements were taken with the devices submerged in Diala XL oil to prevent air breakdown. With the gate biased at -7 V, breakdown was observed at 440 V for a 0.5 µm gate length device and 470 V for a 1 µm gate length device, both of which had a source-gate spacing of 1 µm and a gate-drain spacing of 5 µm (Figure 3).

To our knowledge, this is the highest recorded off-state breakdown voltage for an AlGaN/GaN HEMT device with a relatively small gate-drain spacing and no field plate, exceeding 320 V for a device with similar feature sizes with $\text{Al}_2\text{O}_3$ passivation [3] and 238 V with SiN passivation [4].

Continuous wave measurements were taken at 10 GHz on a load pull bench. The input was matched for reflection and the output was loaded for maximum efficiency. A typical device power sweep at $V_{ds} = 30$ V is shown in Figure 4. The device had 12.2 dB of linear gain, 4.9 W/mm of output power, and 41% peak power-added efficiency. In conclusion, we present an AlGaN/GaN HEMT which exhibits a high off-state breakdown voltage with small features and without a field plate, while maintaining high bandwidth.

References:
Low-Frequency Ultrasensitive Magnetic Detectors

CNF Project Number: 1236-04
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Users: Amol Gupta, Dr. Huanan Duan

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Abstract:
Low-noise magnetic sensors (noise spectral density ~ pT/Hz\(^{1/2}\)) based on magnetic tunnel junctions can be integrated to provide unprecedented clinical information, viz., room-temperature magnetocardiography [1]. The sensors comprise a tunnel junction, a “flux concentrator,” and a mechanism for chopping the input signal with a microelectromechanical actuator to reduce 1/f noise. An array of sensors assembled on a planar substrate along with suitable digital signal processing modules could yield a system that is capable of imaging, both in time and space, the magnetic field generated by depolarization/repolarization currents in the chest cavity during a cardiac cycle.

Summary of Research:
Magnetic tunnel junctions are routinely used as ultrasensitive magnetic sensors, for example in computer hard disk drives. However their low-frequency performance is limited by low-frequency noise, i.e., 1/f noise. We investigated 1/f noise in high-performance MgO magnetic tunnel junctions (MTJs) with a tunneling magnetoresistance (TMR) of 160%, and examined the influence of annealing and MTJ size on the noise. The results show that the annealing process not only dramatically improves the TMR, but can also strongly decrease the MTJ noise.

Junctions were prepared in a deposition system dedicated to synthesis of magnetic nanostructures. A typical MTJ stack had the structure Si/Ta(5)/Ru(15)/Ta(3)/IrMn(10)/Co\(_{60}Fe_{20}B_{20}\)(3)/MgO(2.2)/Co\(_{60}Fe_{20}B_{20}\)(3)/Ta(8)/Ru(7), where the thicknesses in parentheses are in nm. The films were annealed at various temperatures in order to improve the quality of the interface at the tunneling barrier and thereby maximize the tunneling magnetoresistance. Figure 1 shows the relation between TMR and anneal temperature, showing that a value as high as is achieved for \(T_{\text{anneal}} = 380^\circ\text{C}\) [2].

The low-frequency noise of these tunnel junctions is also reduced at the optimum annealing temperature. We observe a current-independent noise voltage that is not very frequency dependent, and find that the 1/f noise is approximately linearly dependent on current indicating an origin in tunneling fluctuations. Figure 2 shows the frequency and current dependence of the noise voltage for an unannealed and optimally-annealed device. The noise level is usefully characterized by the “Hooge parameter” defined as where \(A\) is the area of the tunnel junction. At 10 Hz, the Hooge parameter in the state with the magnetizations parallel (low resistance) decreases from \(3.8 \times 10^{-8}\ \mu\text{m}^2\) for the unannealed device to \(7.1 \times 10^{-9}\ \text{m}^2\) for the optimally annealed device—a fivefold decrease.

We attribute this decrease to a reduction in the number of imperfections within the barrier layer, reducing low frequency occupation variations that alter the tunneling probability roughly fourfold. The noise is found to depend on the applied magnetic field, indicating that magnetic noise also contributes somewhat to the overall noise.

The Hooge parameter indicates that the noise can be reduced by increasing the area.

Figure 1: Effect of annealing on tunneling magnetoresistance ratio.
Figure 2: Power spectral density of the noise observed in (left) an unannealed device and (right) a device annealed to achieve maximum TMR. The raw voltage noise is lower, and since the sensitivity is increased greatly (Figure 1) the effective magnetic noise is much lower.
A voltage spectral density was achieved at 1000 Hz in a junction with an area of 6400 µm². This value is consistent with the state of the art, but is not sufficient to achieve the pT sensitivity necessary to detect the magnetic signal associated with the human heartbeat. Furthermore, although the 1/f noise is fairly small, the increase in noise in the 1-100 Hz range makes the situation even less favorable.

Achieving a viable sensitivity requires two new ingredients: a method to increase the magnetic flux brought to the MTJ (a “flux concentrator”), and a method to modulate the flux threading the MTJ (a “chopper”). Flux concentrators are, at least in principle, straightforward to design—increases in flux by a factor of ~10 - ~100 have been reported in the literature for similar device configurations [3]. Essentially, the flux concentrator is a high-permeability magnetic material that provides a low-reluctance path for the flux to follow. A large cross-sectional area region intercepts magnetic flux (the magnetocardiogram signal) and guides it to a smaller cross-section region. Since the magnetic field adjacent to the smaller region is approximately equal to the flux density, the same flux in a smaller area yields a proportionally higher field.

Devising an effective chopper has proven much more difficult. The role of the chopper is to modulate the flux presented to the sensor at a high frequency. The relevant noise is then the noise level at the chopping frequency. By chopping at 1000 Hz, the effects of 1/f noise can be avoided. This technique is widely used in electronic instrumentation to avoid 1/f noise. We invented a new approach to chopping in which the flux concentrators and MTJ are stationary and a “shorting bar” is mounted on a MEMS comb drive [4]. This shorting bar comprises a small rectangle of high-permeability material. When brought close to the flux concentrator, it diverts the flux from the region where the MTJ is located. When the bar is far from the flux concentrator, a large air gap is introduced, and the high reluctance of the air gap ensures that the flux in the flux concentrator passes to the MTJ as desired. Figure 3 illustrates the concept.

A simple reluctance analysis was used to establish an optimum design, and finite-element modeling was used to confirm the design. Finally, a magnetic structure was fabricated to validate the design experimentally. The depth of modulation, or chopping efficiency, depends strongly on the distance of closest approach. This is because even a small air gap introduces a relatively large reluctance in the magnetic circuit. A value of ~ 2-5 µm represents a reasonable compromise between controllability and efficiency. The distance of furthest retreat is less critical, and a value of ~ 30-50 µm is adequate. The overlap of the shorting bar also has an effect, and there is a clear optimum.

Experimental results using AMR sensors with the optimized chopper design validated the concept and yielded an excellent sensitivity chopping efficiency of 72% with the chopper in the “unshorted” vs. “shorted” positions. The test was conducted using stationary shorting bars fabricated by photolithography, as illustrated in Figure 3. Development of a MEMS-driven shorting bar would be required to achieve a functional sensor, and optimized materials choices and flux concentrator design would be needed to achieve the required final sensitivity. Nevertheless, all the ingredients are now in place for development of a successful system.

References:
Optoelectronic Probes of Suspended Carbon Nanotube Transistors

**CNF Project Number: 1854-09**
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**Abstract:**
Suspended carbon nanotube transistors are used as nanoscale optoelectronic probes to investigate the electrical and optical processes in biological systems due to their high sensitivity, robust electrochemical stability, and high surface to volume ratio. Taking advantage of dual-optical trapping technique and scanning photocurrent microscopy, we are able to study the interface between carbon nanotubes and biomolecules such as DNA at the single-molecule level.

**Research Summary:**
Carbon nanotube-biomolecular hybrids have emerged as one of the most promising materials for biological and biomedical applications, such as biosensors, drug delivery, and imaging. Recently, carbon nanotubes (CNTs) have shown the ability to protect bound DNA cargos from enzymatic cleavage both during and after delivery into cells. This ability may result from the interaction between CNTs and DNA, which makes DNA unrecognizable to enzyme binding pockets. Therefore, it is important to study the interaction between CNTs and DNA.

Figure 1 shows the cross view of a CNT transistor. We etched a 5 µm wide and deep trench into a 170 µm thick fused silica substrate using the Oxford PlasmaLab 80+ RIE system. The source and drain electrodes (2 nm Ti, 40 nm Pt) were put beside the trench and separated by 8 µm. On the top of metal electrodes were the catalyst pads (10 nm of Al₂O₃, 0.2 nm of Fe) deposited by the evaporator. Using a “fast heating” chemical vapor deposition method, we then grew the carbon nanotube which connects the source and drain. A 100 by 60 µm microfluidic poly(dimethylsiloxane) (PDMS) channel was sealed over the CNT, and a gold wire in a reservoir on the end of the channel was used to set the electrochemical potential of the solution [1].

Figure 2 illustrates the manipulation of DNA in our experiment. Taking advantage of dual-optical trapping technique...
[2] and scanning photocurrent microscopy, we build up a nanoscale detection system using suspended CNT transistor which enables us to investigate the interaction between CNT and DNA at the single-molecule level. We tightly bonded both ends of a DNA molecule with microbeads, which could be held and moved by dual optical traps. The position of the CNT bridge is detected through the photocurrent measurement right before the DNA is moved to the target device. We have observed that when the DNA is pushed down to the target CNT device, there is a current shift which may indicate the interaction between CNT and DNA. We will continue to do more measurement regarding the changes of optoelectronic properties of the CNT transistor before and after the DNA molecule interacts with CNT.

This experiment will not only extend the understanding of the interactions between DNA and CNTs, but also provides a new platform to probe in real-time and thus understand biomolecular interactions.

References:


Implantable Device for Prevention of Late-Phase Hemorrhagic Shock Using a Novel Non-Enzymatic Fuel Cell

CNF Project Number: 1863-10
Principal Investigator: David Erickson
Users: Vlad Onescu, Seoho Lee

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Primary Source of Research Funding: Office of Naval Research (ONR)
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Abstract:
Here we present an autonomous implantable device for vasopressin monitoring and prevention of late-phase hemorrhagic shock powered using a novel non-enzymatic fuel cell unit. The device consists of a biosensor, drug delivery device and fuel cell unit. The innovative stacked single-layer fuel cell (SLFC) design allows to achieve small packaging volume and reduces oxygen cross-over at the anode resulting in a power density of 46 µWcm⁻³, a large improvement over the state of the art for non-enzymatic fuel cells [1]. In addition, it is demonstrated for the first time, that non-enzymatic fuel cell can be used to directly monitor changes in vasopressin levels by supplying current to an aptamer-based biosensor.

Summary of Research:

Fuel Cell. Glucose fuel cells produce electricity through the coupling of glucose oxidation and oxygen reduction reactions. The biggest challenge with non-enzymatic fuel cells has been the lack of selective catalysts for glucose oxidation in the presence of oxygen at the anode. Most groups have tackled this issue by having a stacked electrode design, meaning that a porous cathode is placed in front of the anode in order to reduce the amount of oxygen that interferes with the glucose oxidation reaction [2, 3]. This approach, although potentially useful as a coating layer for implantable devices, had the disadvantage being thick and impractical for low volume applications. Here we present a fuel cell unit composed of stacked SLFCs printed on each side of a 500 µm thick fused silica wafer. Each SLFC consists of a platinum nickel alloy anode surrounded by an oxygen-selective platinum aluminum alloy cathode. By itself, when tested in vitro at physiological levels of glucose and oxygen, the SLFC achieves low power output (0.6 µW), because of oxygen cross over at the anode, however when the layers are stacked 500 µm apart, the oxygen gets depleted at the cathode before it reaches the anode. Consequently, due to a reduction in oxygen cross-over, two stacked fuel cells in parallel can achieve 4.6 µW (or 46 µWcm⁻³ power density) — almost eight times higher power output than a SLFC (Figure 3a).

The fabrications steps for the fuel cells described here were performed at the Cornell NanoScale Facility. The single-layer fuel cells (SLFC) used in this paper are fabricated on fused silica substrates using multiple patterning steps (Figure 2). In order to achieve high surface area, a Raney-type alloy process is used for both the anode and the cathode. The process — first demonstrated by Gebhardt, et al. [1] — involves the annealing of a thin layer of platinum with a non-noble metal followed by the chemical etching of the non-alloyed outer metal layer. In this paper, nickel was used as the non-noble metal at the anode and aluminum at the cathode. We use a Ni/Pt alloy as anode for glucose oxidation because it has been shown elsewhere to exhibit greater selectivity towards glucose than other catalysts. 20 nm of Ti was used as adhesion layer followed by the deposition 100 nm of Pt and 300 nm of Ni. Experiments with several anneal temperatures in the range 400°C-600°C have demonstrated that the roughness that can be obtained using a Reney-type process peaks when the annealing temperature is 500°C. For the cathode, a high surface area Pt surface was obtained by annealing Pt/Al at a low-temperature of 300°C and subsequently etching the Al with NaOH.

Figure 1: a) Implantable autonomous device. b) structure of single layer fuel cell (SLFC) and SEM images of the electrodes. c) oxygen-glucose separation at the anode due to stacked SLFC design. d) biosensor for vasopressin detection.
Biosensor. Vasopressin in humans is correlated with blood pressure, and sudden drops in vasopressin levels can indicate a risk of late-phase hemorrhagic shock or other traumatic injuries. Figure 3b shows the voltage drop across the biosensor as current was varied between 0 and 20 µA using a current source. It can be seen that when 1 mM vasopressin is present, the voltage drop is higher by roughly 2.6% due to increasing resistance caused by vasopressin binding.

Integrated Device. In order to demonstrate the potential applications of the novel fuel cell unit, we developed an implantable device capable of monitoring and maintaining vasopressin levels in solution. The device in Figure 4a, composed of two stacked SFCLs and a biosensor, was used to demonstrate that this fuel cell unit can be used to detect changes in vasopressin (Figure 4b). It can be seen that the voltage drops by 2.56% when vasopressin is added, which is consistent with Figure 3b. The vasopressin drug delivery system has been presented in another paper [4].

References:
Tunable Capacitive Micromechanical Ultrasonic Receiver Array

CNF Project Number: 1969-10
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Abstract:
This project aims to develop custom ultrasonic microphones. It involves developing key processing steps to fabricate MEM devices. Once these processes have been characterized and optimized, we will proceed to complete first round prototype devices.

Summary of Research:
MEMS-based ultrasonic transducers and receivers are of current interest due to their ability to generate and receive acoustic wave, low cost, IC-based fabrication enabling integrated electronics, small size, high degree of reliability, and low power consumption. Capacitive micromachined ultrasonic receivers (CMURs) are favored utilized over piezoelectric MURs (pMURs) for their simpler designs and improved performance. Utilizing an array approach, large band width receivers with high sensitivity are realizable in compact formats. In addition, phased array detection can be utilized for source targeting. The frequency response (resonant frequency) and quality factor, \( Q \), of the individual CMUR cell scales with membrane geometry, whereas the overall sensitivity scales with cell number. In an effort to improve CMURs performance, a novel tunable CMUR device is proposed. The proposed device integrates an array of tunable CMURs to produce a highly sensitive receiver with tailored bandwidth. Tuning is obtained through an orthogonal electrostatic force which creates a variable biaxial residual stress which alters the spring constant shifting the natural frequency of the membrane. Through utilization of frequency tuning and multiple membrane geometries wide bandwidth receivers are possible.

Figure 1 illustrates a single element CMUR MEM device and a schematic of the fabrication steps used to process proto-typed devices. During this year first round prototype devices were fabricated. This process was delayed due to key fabrication equipment being off-line (deep RIE Si etcher, Karl Suiss wafer bonder and critical point dryer). This lead us to farm out to the wafer-bonding step to the University of Michigan in order to complete the first round proto-types. The device results for this batch of devices were poor. Most of the devices were shorted out due to clamped membranes (resulting in direct electrical shorts in the devices). The small percentage of devices not shorted exhibited low \( Q \) values and resonant frequency twice the design value. Several months were required to remodel and redesign the MEM device design and rethink the fabrication process (making it more robust). Second round proto-type devices were completed, but during the lapping phase (devices are sent out to a third party for lapping), the devices were over thinned, destroying them.

Third round prototypes were started in April, 2012. The progress of this round has also been delayed due to the unavailability of the Heidelberg mask machine (delaying the fabrication of the optical masks required for photolithographic steps). Last week, the masks were made, coinciding with the repair of the Heidleberg, and proto-type development has resumed.

We hope to complete the proposed devices this year with improved performance, and results will be published.
Electro-statically Tunable Mems Ultrasonic Transducer

Disk or Ring Membrane
Electrostatic “tuning” Contact pair
Support Arm

Highly Doped SI substrate for back side bottom Capacitive Electrode

Top Capacitive Electrode

Key Processing Step

- Highly Doped SI Wafer (n+)
- PCVD Silicon Nitride (or other etch stop layer)
- Amorphous Si deposition (or other sacrificial layer)
- Pattern and etch amorphous Si
- PCVD Silicon Nitride (or other membrane layer)
- Via and sacrificial etch (side supports not shown)
  optional nitride support for ring membrane
- Top and bottom electrodes

Figure 1
Laser Crystallized Thin Film Silicon Transistors

CNF Project Number: 1990-11
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Abstract:
This report describes the fabrication of thin film Si transistors using a laser crystallization method. The laser crystallization method is performed at room temperature, which enables the use of these devices on lower temperature substrates. Transistor performance is shown to achieve mobilities of 400 cm²/Vs. Anticipated applications of these transistors are monolithic integration with compound semiconductor substrates or with conventional CMOS for 3D integrated circuits.

Summary of Research:
Hybrid integration of different material systems is an area of intense research for combining systems with different advantages. One such example is the integration of compound semiconductor optoelectronics with high performance switching and amplification transistors. III-V optoelectronics form the highest efficiency light source known, the light emitting diode (LED), with internal quantum efficiencies near 100% and luminous efficacy of over 100 lm/W. However, materials that are good light emitters do not form high quality transistors. There are many ongoing projects investigating this integration of incompatible material systems including heterogeneous growth [1], shape self-assembly [2], wafer bonding [3], and solder-bump bonding [4]. Although some of these techniques have shown promise, many are limited by the reliability and minimum size of the bonding/integration technique.

To overcome these problems, our process starts with high quality optoelectronics devices. The compound semiconductor wafers are sourced commercially, and are then patterned to form LED devices. Using this as a starting material allows us to obtain the highest efficiency possible for LEDs. The integration with silicon is then performed monolithically by depositing a 1 µm film of SiO₂ and 100 nm of amorphous Si. The amorphous silicon is then irradiated with a 308 nm excimer laser to melt and allow to cool and form polycrystalline silicon, Figure 1a. This polycrystalline layer is the active layer of our transistor and is then ion implanted, at the Cornell NanoScale Science and Technology Facility, to define the source and drain areas, Figure 1b. The contact dopants are activated using an RTA process, Figure 1c. The transistor area is then defined and source drain electrodes are deposited, Figure 1d, e. Lastly, a Parylene-C dielectric layer and a gate metal is deposited and patterned, Figure 1g. Figure 2 shows a top-view micrograph of a completed device.

Current-Voltage characteristics of these transistors were measured using a semiconductor analyzer shown in Figure 3. These laser crystallized polycrystalline silicon thin film transistors showed a mobility of 400 cm²/Vs, a value much greater than most thin film transistors and approaching values typically only found on single crystal silicon.

Future work includes lowering the contact resistance of the source and drain contacts, improving the process techniques to include a laser anneal for dopant activation, and monolithic integration with LED devices.

References:
Figure 1: Cross-sectional schematic of transistor process flow. 
(a) Laser crystallization of silicon. (b) Ion implantation. (c) Dopant activation. (d) Active pattern. (e) Source and Drain contacts. (f) Gate dielectric. (g) Gate metal.

Figure 2: Top-view micrograph of fabricated devices.

Figure 3: Current-voltage characteristics of silicon thin film transistor devices.
Organic Permeable Gate Transistor for Digital Printing Applications

CNF Project Number: 2077-11
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User: Christopher Heidelberger

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Primary Source of Research Funding: Xerox Company
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Abstract:
A novel permeable gate transistor was designed for use in high-resolution, high-voltage transistor backplanes for digital printing applications. While standard designs for digital xerography require thin-film transistors (TFTs) capable of switching over 500-1500V, with this improvement, TFTs would only be required to switch over 5V, improving resolution and ease of manufacturing. Conducting polymers used in the device are incompatible with polar solvents and thus most conventional photoresists, and were consequently patterned using a fluorinated resist system designed for patterning organic films. The finished devices were electrically tested and show behavior consistent with expectations.

Summary of Research:
Current laser printers use xerography for rapid and inexpensive printing. Xerography is essentially a photographic process by which a charged surface is selectively discharged using light from a laser, creating a pattern that can be transferred onto paper via charged toner particles [1]. This process can be fully digitized through the use of a backplane of addressable thin film transistors (TFTs), which replace the laser in selectively compensating the surface charge [2]. However, this requires TFTs that are capable of switching over 500-1500V, which would have large footprints and be difficult to fabricate [3].

Our solution to this problem is illustrated in Figure 1. Holes are injected from a hole-injection layer into a charge transport layer (CTL), which is hole-transport only. A grounded metal mesh in the CTL collects some of the injected holes, but allows others to pass, and get collected by the top electrode. In this configuration, the TFTs are only required to actuate over 5V to turn the device on and off. This allows the TFT design to be borrowed from current display technology [4].

Devices were fabricated on oxidized 4-inch Si wafers. Figure 2 shows the film stack, in the order of deposition and patterning. Poly(3,4-ethylenedioxythiophene) (PEDOT:PSS), which is known to form a diode with the CTL material, was chosen as the hole-injector, while aluminum was chosen to form the gate and collector. The CTL material is a proprietary conducting polymer from Xerox. Metals were deposited by thermal evaporation and patterned using lift-off. The PEDOT:PSS and CTL material were spun on to the wafer in solution and patterned using an O2-plasma etch.

The key challenge to the fabrication of these devices was the incompatibility of the PEDOT:PSS and CTL material with conventional photoresists. The electronic properties of the
conducting polymers are negatively affected by polar solvents such as those used in developing and stripping conventional resists [5]. To avoid this problem, a fluorinated resist system, provided by Orthogonal Inc., was used. The Orthogonal resists and associated developers and strippers are mutually immiscible with polar and nonpolar substances, limiting their interaction with the PEDOT:PSS and CTL material [5].

An image of a finished device with an emitter width of 100 µm is shown in Figure 3. Features larger than 10 µm resolved well, while features smaller than 10 µm became rounded and resolved differently depending on the underlying material (e.g., Si, Au, conducting polymer). This can be observed in the 2 µm holes in the gate mesh, and was not expected to interfere with the operation of the device. There is a high amount of foreign material present (black spots), which was likely introduced during non-cleanroom processes.

The intended functionality of the device is demonstrated in Figure 4 at reduced voltages. The curve on the left shows the diode behavior of the device (collector at -15V, emitter swept from -25V to 5V) with the gate left floating. To turn off the device, the emitter must be brought from 0V to between -10V and -15V. When the gate is grounded (right curve), the turn-on voltage shifts by about 10V, reducing the voltage shift needed to turn the device off to about 5V. Future iterations of the device will have a larger CTL layer to be able to accommodate the higher voltages required for the xerographic process.

References:
Fabrication of Silicon on Insulator Device for Characterization of Thin Film Solar Cell Surfaces

**CNF Project Number:** 2099-12  
**Principal Investigator:** Radhakrishna Sureshkumar  
**User:** Miriam Israelowitz

**Affiliation:** Department of Chemical Engineering, Syracuse University  
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**Abstract:**
Thin film silicon photovoltaic cells offer the potential for efficient solar energy harvesting. Understanding how the surface architecture/morphology of the silicon thin film cells affects photocurrent is essential to improving cell efficiency. In this project we fabricate a silicon-on-insulator (SOI) device that would emulate the characteristics of a thin film solar cell surface when a voltage bias is applied to it. Using this device, the voltage characteristics as a function of wavelength are measured.

**Summary:**
Silicon is one of the most commonly used materials in the creation of solar cells. Thin film solar cells, cells on the order of 500 to 1000 nm thick, have become favored area of research because of the reduced amount of silicon needed for device creation. Characteristics of thin film solar cells include features such as transmittance, absorbance, and electrical conductivity. In the characterization of silicon thin film surfaces, a SOI device with applied artificial electrical gradient can be used to approximate the effects of a thin film silicon solar cell surface [1, 2].

In this work, we develop a device that can be used in research of thin film solar cell surfaces and surface enhancements. The device structure is composed of a bonded SOI wafer and aluminum contacts. The bonded SOI wafer used consists of a thick undoped silicon substrate, 2 nm of a thermal grown oxide layer, and 1.5 nm of a bonded n-doped amorphous silicon top layer. The surface of the SOI wafer is cleaned using a buffered oxide etch (BOE) to remove the native oxide growing on the top n-doped layer. Aluminum contacts (30 nm) are masked off and deposited via thermal evaporation on the top n-doped layer.

The electrical characteristics of the device were measured by applying a 0.05V bias to the contacts to emulate the internal electrical gradient of a solar cell. When light is shined between the contacts, a current is induced. The voltage response is measured as a function of the light wavelength using a halogen lamp and tunable monochromator between the spectrum of 400 and 1100 nm. Figure (1) shows the voltage response to the SOI wafer with applied bias device. As expected, the voltage drops suddenly to zero near 730-800 nm, the band gap frequency of amorphous silicon. The contacts show good stability and ohmic properties to allow bidirectional flow of current and voltage. The device can be used in future work of characterizing the surface of thin films and any effects of surface enhancements to the thin films.

**References:**
Investigation of Acid Diffusion During Laser Spike Annealing with Systematically Designed Photoacid Generators

CNF Project Number: 386-90
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Users: Marie Krysak, Byungki Jung

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Abstract:
The post-exposure bake in the lithographic process has a primary influence on resist performance as the time/temperature profile controls both the diffusion of photogenerated acids and the deprotection of the resist backbone. We utilize a laser (CO2) based scanned heating system to achieve sub-millisecond heating durations with temperatures up to the thermal decomposition limit of the resist. This research is aimed at using synthetic techniques to vary the structure of the photoacid generator (PAG) in order to learn about the role of PAG size and structure on acid diffusion during sub-millisecond heating.

Summary of Research:
The requirement for smaller feature dimensions continues to drive lithography research forward. In order to meet the requirements of the ITRS roadmap, it is necessary to investigate not only resist materials, but every step of the lithography process as well. By optimizing processing steps, it may be possible to push materials past their current limit on state of the art lithography systems.

The post exposure bake (PEB) step in the lithography process has the most effect on the shape and size of the patterns produced [1]. The heat catalyzes the deprotection or cross-linking of chemically amplified resists by mobilizing the PAG in the resist matrix. Some diffusion of acid is necessary to start the reaction, however, uncontrollable acid diffusion has been held accountable for limiting resolution and increasing line edge roughness (LER) of patterned resists [2,3]. Controlling acid diffusion by altering resist chemistry has been studied extensively with the creation of polymer-bound PAGs, where the PAG is chemically bonded to the resist chain, thereby limiting its mobility [4,5]. These resists have shown reduced LER, but at the expense of sensitivity [5].

Our research is associated with changing the method of the PEB to improve resist performance. Resists baked on a hotplate are heated to a modest temperature, in the range of 100°C to 150°C for seconds to minutes. By heating the wafer with the laser, the film is subject to a high rate of heating and cooling, in the sub-millisecond timeframe. We have shown that laser-post exposure bake (l-PEB) has resulted in improved LER, along with greater sensitivity, demonstrating the excellent performance capability of the laser [6].

We have synthesized a set of PAG molecules with differing anion sizes, shown in Figure 1. The anion structure has been systematically varied to investigate the effects of anion size on diffusion behavior. The acid diffusion length was measured using a bilayer technique that has been previously reported [7, 8]. The resist on the PDMS stamp was exposed using an ABM contact aligner with dose steps from 0-20 mJ/cm², and tested at a variety of bake temperatures with both the hotplate and laser. Figure 2 shows the acid diffusion length of the various photoacids as a function of bake temperature. The acid diffusion length is shortened with l-PEB treatment, even though those wafers were brought to a higher temperature than those tested on the hotplate.

Figure 1: Structures of PAG molecules.
Figure 2: Acid diffusion behavior of the PAGs with hotplate and l-PEB.
By reducing the timeframe of the past exposure bake, we can see a distinctive change in the diffusion characteristics of the PAGs. There is also a definite trend in diffusion length with varying the anion size. As the anion gets larger, the diffusion length decreases. The patterning properties of the PAGs were tested with a JEOL 9300 e-beam lithography system. Figure 3 shows patterns achieved with both hotplate and l-PEB. The l-PEB method is shown to reduce the high-frequency line edge roughness (LER) of the patterns, from 12 nm to 9 nm, calculated by SumMIT software. This study has helped us identify PAGs for use with our new l-PEB system, with optimal diffusion behavior and promising lithographic properties.

References:
Room-Temperature Nanoimprint Process for Patterning Biomolecules

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Abstract:

Fluorinated solvents and photoresists have been found to be non-damaging to biomolecules such as proteins and DNA. We have developed a novel room-temperature nanoimprint patterning process using these materials. This process can be used at room temperature because it relies on controlling adhesion forces rather than polymer flow. Clean, reproducible features from 100 µm to 1 µm have been imprinted. This process has other advantages including not requiring a descum etch after imprint and also being capable of patterning large-area features. Finally, we have demonstrated the use of the patterns formed to pattern proteins via a lift-off step.

Summary of Research:

Conventional nanoimprint lithography (NIL) is incompatible with patterning biomolecules for two reasons. First, they are solution processed in harsh organic solvents, such as acetone, which damage biomolecules. Second, conventional nanoimprint relies on heating the imprint resist above its glass transition temperature (Tg) or melting temperature such that it can flow to take the form of a template mold. But, biomolecules are damaged if heated above 40°C, which is below the Tg of most resists.

We have developed a novel nanoimprint process to address both these issues and successfully pattern proteins by nanoimprint lithography. To avoid using harsh organic solvents a fluorinated polymer, poly (1H,1H,2H,2H-decafluoromethacrylate), is used for the nanoimprint resist. Highly fluorinated materials are soluble in fluorous solvents such as hydrofluoroethers. The Ober group has published previously on the use of Orthogonal Processing for patterning of organic electronic materials [1, 2]. These materials have recently been shown to be non-damaging to biomolecules [3].

To avoid heating the resist during imprint we spun-coat a thin 200 nm-thick layer of the fluoropolymer, then pressed a rigid quartz template into this layer under 300 psi using the Nanonex 2500 tool. Although the resist doesn’t flow during this step, some areas of the resist are compressed while others aren’t. This sets up shear forces at the edge of features causing micro-cracks through the resist. Then when the pressure is removed and the template lifted off, the resist that was compressed is adhered to the template and removed with it. Figures 1 and 2 show demonstration patterns. For the resist to be successfully removed the adhesion between the resist and the template must be greater than the adhesion between the resist and the silicon substrate, as illustrated...
in Figure 3. These adhesion forces can be tuned by using surface treatments such as molecular vapor deposition of self-assembled monolayers, APTMS treatment, oxygen plasma cleaning or Piranha cleaning.

If the adhesion between the resist and substrate is sufficiently weak, it will be removed cleanly, so no etch step is required to remove any remaining ‘scum’ layer. Also, since the imprint process does not rely on the flow of polymer, the size of features patterned is not limited by the polymer flow distance. Large features can also be patterned right next to small features, and variation of fill factor across the wafer is not an issue.

To demonstrate the utility of this nanoimprint process, patterns in the resist were transferred into protein patterns by first treating the exposed silicon with APTMS to present an -NH group on the surface for the protein to bind to. Proteins were then put down from an aqueous solution. This was followed by fluorescent label that binds to the protein in order that the patterns can be imaged. The final step was to lift off the remaining parts of the resist in hydrofluoroethers, leaving well-formed protein patterns. The whole of this process can be repeated with a different protein to achieve patterns of more than one protein on the same substrate as illustrated in Figure 4.

References:


Development of an Organic-Inorganic Hybrid Nanoparticle Photoresist for Next-Generation Lithography

CNF Project Number: 386-90
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Abstract:
We have developed a transparent, high refractive index inorganic photoresist with significantly higher etch resistance than even the most robust polymeric resist. As patterned feature sizes continue to decrease, film thickness must be reduced in order to prevent pattern collapse. Normally thinner films prevent sufficient pattern transfer during the etch process, creating the need for a hardmask, thus increasing production cost. Compared to PHOST, we have shown over six times better etch resistance. Organic ligands have been attached to a zirconium oxide (ZrO₂) nanoparticle core to create an imageable photoresist. This resist has shown superior resolution with e-beam lithography, producing 30 nm features.

Summary of Research:
The semiconductor industry is currently patterning at the 45 nm node with 193 nm immersion lithography (193i). 193i has extended the lifetime of 193 nm exposure tools by introducing an immersion fluid in the gap between the resist and the lens of the tool [1]. This will expand the depth of focus, as well as increase the numerical aperture (NA), which will increase resolution beyond what was possible with dry lithography. Water has had success as an immersion fluid, enabling pattern dimensions of 45 nm with an NA of 1.35. Other high index fluids are currently in the development stage that can produce 30 nm feature sizes and below with an NA of at least 1.50 [2]. Next generation techniques such as EUV lithography are currently under investigation to push resolution limits to sub-30 nm and beyond. Regardless of the exposure tools that will be used, new resist platforms need to be developed to address current patterning issues as well as those that will arise with new technologies.

As pattern sizes reach the sub-30 nm range, it is necessary to use thinner films to prevent pattern collapse from high aspect ratios. An aspect ratio of 2:1 would require films in the 40-50 nm range. However, resist films are usually comprised of organic materials and cannot sufficiently resist the etch process as a thinner film [3]. Therefore, extremely high etch resistant structures must be studied and developed to allow pattern transfer to the underlying substrate. This study examines hafnium oxide based nanoparticles as a next generation photoresist material. This material provides benefits beyond the excellent etch resistance of a metal oxide. Metal oxides are extremely thermally stable, and because ZrO₂ cannot oxidize, it is chemically stable as well. It is possible to control the size of the nanoparticles through synthetic techniques [4], allowing the creation of small particles with a narrow size distribution, thereby increasing homogeneity of the film. We have previously studied hafnium oxide as a core for nanoparticle photoresists, which has shown excellent sensitivity and resolution with EUV patterning, as well as unprecedented etch resistance compared to state of the art resists [5]. ZrO₂ has been shown to be low absorbing at both 193 nm and EUV wavelengths (13.5 nm) [6].

ZrO₂ nanoparticles with methacrylic acid (MAA) ligands were synthesized and patterned using e-beam lithography and photolithography. ZrO₂ is used as a core to increase the etch-resistance of the resist. Besides the nanoparticles, a photoactive compound was added into the resist formulation to generate a photochemical reaction upon exposure and change the solubility of the resist. An organic developer is used to develop the resist using an ABM contact aligner at 254 nm and using the JEOL 9300 e-beam lithography system. High-resolution negative-tone patterns have been achieved (Figure 1) using e-beam radiation with feature size as small as 30 nm.

One of the most important aspects of this photoresist is etch resistance. A strong advantage of having a metal oxide core is its extreme resistance to plasma etching. To determine etch rates of our resist, etching studies were carried out by first flood exposing a film and then measuring the film thickness with a Woollam spectroscopic ellipsometer. In order to fully exploit the high etch resistance of the metal oxide, an oxygen plasma pre-treatment was applied to the resist before etching. The chemical stability of the ZrO₂ core enabled an O₂ RIE process for 30 seconds. The film was then...
etched for time intervals of 30 seconds with an Oxford 81 etcher, with film thickness measurements being performed between each interval. From these data points, an etch rate of the resist was determined and compared to the etch rate of poly(hydroxystyrene) (PHOST). The etch rate comparisons of ZrO$_2$-MAA and PHOST (polyhydroxystyrene) for CF$_4$ and SF$_6$/O$_2$ are shown in Figure 2 and Figure 3.

With CF$_4$ etch gas, the etch resistance of ZrO$_2$-MAA is six times better than PHOST as shown in Figure 2. The etch resistance of ZrO$_2$-MAA is fourteen times better when etched under SF$_6$/O$_2$ in Figure 3.

The e-beam patterning performance and etch resistance of zirconium oxide nanoparticles functionalized with organic ligands has been reported. Further research is being conducted to fully exploit this material, including different core/ligand variations.

References:


Hafnium and Zirconium Oxide Nanoparticles as Novel Photoresist Materials

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Abstract:
Performance requirements for next generation lithography will necessitate the development of entirely new resist platforms. The new resists for extreme ultra-violet (EUV) lithography must show high etch resistance and high sensitivity. We have developed a novel inorganic photoresist platform to be applied in advanced lithography. These inorganic photoresists are made of metal oxide nanoparticles stabilized with organic ligands and have shown etch resistance that is 25 times higher than polymer resists. This will enable the processing of very thin films (< 40 nm) and will push the resolution limits below 20 nm without pattern collapse.

Summary of Research:
Currently, extreme ultra-violet (EUV) lithography is considered the upcoming next generation patterning technology [1]. Despite remaining challenges with the EUV system, alternative lithographic techniques, such as double patterning, nanoimprint and self-assembly, either cannot achieve the required resolution or have other issues that limit their applicability. As a result, EUV lithography and improved photoresists for this patterning technology are important goals for the semiconductor industry.

As pattern sizes reach the sub-20 nm range, it is necessary to use thinner films to prevent pattern collapse. However, photoresist films that are comprised of organic materials cannot sufficiently resist the etch process as thinner films [2]. Therefore, extremely high etch resistant structures must be studied and developed to allow pattern transfer to the underlying substrate. Additionally, the relatively low intensity of current EUV sources requires photoresists that demonstrate high sensitivity and optimum absorbance. At 13.5 nm, which is the wavelength used in EUV lithography, the absorption of all materials is very strong and only dependent on their atomic composition and density [3]. For example, elements that are commonly used in photoresists at other wavelengths, such as fluorine, are highly absorbing at 13.5 nm making them problematic for EUV applications. Other elements including carbon, silicon, zirconium or hafnium show lower absorption.

In order to fulfill the requirements that were mentioned above, in this study we present a next generation photoresist material based on hybrid organic/inorganic nanoparticles. The nanoparticles are comprised of a hafnium oxide or zirconium oxide core that is surrounded by organic ligands. Previous work at Cornell University has studied the application of these inorganic photoresists in DUV, 193, and e-beam lithography [4]. These studies also revealed that the nanoparticle films exhibit exceptionally high etch resistance due to their thermal and chemical stability [5].

Hafnium oxide nanoparticles stabilized with carboxylic acid ligands were prepared by a controlled hydrolysis reaction. Hafnium or zirconium isopropoxide was dissolved in an excess of the carboxylic acid (methacrylic acid, MAA or isobutyric acid, IBA) at 65°C followed by the slow addition of water. After stirring for 21 hours, the product is precipitated and dried. Photoresist solutions were prepared by adding proper amounts of the nanoparticle powder to PGMEA followed by the addition of the photoactive compound or any other additives. Initial DUV (254 nm) testing was carried out at the Cornell NanoScale Science and Technology Facility (CNF), using an ABM contact aligner equipped with a mercury lamp UV source. EUV experiments were carried out at Lawrence Berkeley National Laboratory (LBNL) using the SEMATECH BMET EUV tool.

The Hf-methacrylic acid (HfMAA) and Zr-methacrylic acid (ZrMAA) inorganic photoresists can be patterned at high resolution using either a photoradical initiator (2,2-dimethoxy...
acetophenone, DPAP) or a photoacid generator (PAG). Additionally, the same films can be patterned either in positive tone or in negative tone depending on the film processing and the choice of developer. This versatility of the inorganic photoresist is demonstrated in Figure 1.

The isobutyric acid nanoparticles can also be patterned at both tones with either DPAP or PAG as the photoactive compound. This is demonstrated in Figure 2 for the HfIBA inorganic photoresist.

The same resist formulations that were patterned using DUV at CNF were then tested under EUV exposure at the LBNL. The ZrMAA nanoparticle photoresist was patterned at 26 nm half-pitch using an exposure dose of 4.2 mJ/cm². The results demonstrate that the inorganic photoresist has a remarkable sensitivity and the capability for high resolution patterning.

References:


Nanocalorimetry Study of Metastable Au-Si Alloy

CNF Project Number: 522-94
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Abstract:
This project focuses on the applications of the thin film nanocalorimetry device [1, 2] developed using CNF. We investigate interesting thermodynamic properties that are unique to nanometer size materials including size-dependent melting point depression and magic number size formation in metal nanoparticles [3], thickness-dependent glass transition in polymer thin films [4], phase transitions in self-assembled monolayers of alkanethiols (SAMS) [5], and the melting of lamellar crystals of silver alkanethiolates (AgSR) [6, 7]. In this report, we discuss the formation of a unique metastable Au-Si alloy (δ₁) through the in situ nanocalorimetry measurement during physical vapor deposition (PVD) of gold (Au) and silicon (Si) [8]. The δ₁ melts 60K lower than the eutectic alloy and has a composition of 74 ± 4 at. % Au and a b-centered orthorhombic structure (a = 0.92, b = 0.72, and c = 1.35 nm; body-center in the a-c plane), which grows heteroepitaxially to Au.

Summary of Research:
We have studied the formation of a unique metastable phase (δ₁) which melts ~ 60°C below the eutectic melting point (305°C vs. 363°C) in the gold-silicon (Au-Si) system using in situ nanocalorimetry measurement during the deposition of Au and Si. This is a rare case of a direct thermal analysis of a metastable alloy and to our knowledge the first measurement in the Au-Si system. Figure 1 shows the schematic of nanocalorimetry device and the formation of δ₁ on a nanoparticle gold substrate wherein the metastable alloy grows heteroepitaxially with gold and wets the surface. The nanocalorimetry result shows the melting peak at 305°C, which is lower than the γ phase, the eutectic, or the 2D surface crystal.

The results of the in situ nanocalorimetry during the deposition of Au and Si are shown in Figure 2. Figure (2a) shows the result when the sample is initially silicon rich where Si is deposited first and then Au is added while the nanocalorimetry measurement is being taken at the same time with the resolution of submonolayer of added Au. The results show the initial formation of the eutectic, but after more Au is added, the δ₁ phase dominates. Both the eutectic and δ₁ show size-dependent melting point depression. Figure (2b) shows...
the opposite case wherein Au is deposited first and then Si is added. Both the eutectic and $\delta_1$ form, and no significant melting point depression is observed at the initial stages of the reaction due the difference in the wettability of $\delta_1$ on Au and Si. The $\delta_1$ does not wet a Si surface, so it forms small 3D particles — while it wets an Au surface, so it forms a uniform 2D surface layer (confined liquid). Geometrical considerations explain why the size dependent melting point depression is more significant in a 3D compared to a 2D structure.

The schematic on Figure 3 shows the key learnings from this investigation. The metastable $\delta_1$ alloy grows heteroepitaxially to a gold substrate. Upon melting, the liquid phase features a high degree of ordering of the atoms. This ordering is brought about by the 2D crystalline surface layer, the layered liquid induced by this surface ordering, the ordering at the interface of the liquid to the Au substrate and the layered liquid induced by this interfacial ordering. The metastable solid $\delta_1$ phase is the frozen state of this highly ordered liquid.

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References:
Patterning of Polymer Brushes as Membranes and Investigating the Effects

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Abstract:
Patternning of polymer films has been an area of great interest due to the broad range of applications including bio-related and medicinal research. Consequently, the nature of patterned polymer brushes as removable thin films was explored. Both an etching and polyvinyl alcohol dissolution process has enabled us to lift off very thin membranes for further characterization with the potential of using them as Janus membranes for biological applications. Furthermore, the repolymerization of monomer on brushes that had previously been exposed and patterned was explored.

Summary of Research:
Polymer brushes can be formed by densely attaching polymer chains to a substrate. They have garnered much attention due to the need for systems in the fields of microelectronics, nanofluidic devices, biosensing, and other areas of nanotechnology [1-4]. Another application of interest is the production of Janus membranes. We have begun to explore different means for detaching polymer brush membranes from a surface. Previous work on polymer brush nanochannels led us to question the robustness of the bridging polymer film that stretched over distances ranging from 100 nm to a few microns. The first method involved a silicon oxide layer upon which the polymer brushes were grown from via atom transfer radical polymerization (ATRP) to thicknesses ranging from 30 to 70 nm. The polymer brushes were then patterned and crosslinked by a UV source and etched with HF to produce very thin membranes. HF was chosen because it etches only the silicon oxide and does not harm the polymer brush membranes, and because silicon oxide is a conventional surface for polymer brush growth. Thus we are not limited to the types of brushes membranes that can be produced.

Membrane characterization included TEM and optical microscopy (Figure 1). Results indicate the membranes retain a significant amount of strength even at very thin thicknesses. Folding, wrinkling, and bending are commonly observed instead of tearing or falling apart and therefore imply that membranes are robust enough to undergo further functionalization to produce Janus membranes. Additionally, this technique can provide information regarding ATRP initiator immobilization as well as the brush polymerization process to answer questions about the nature of brush films.

A second method for the controlled release of polymer brushes from a silicon substrate involves crafting the polymer film from a sacrificial layer of poly (vinylalcohol) (PVA). For this, thin layers of PVA were spin-coated on bare silica and were subsequently functionalized with an ATRP starter in a gas phase reaction. ATRP polymerization of monomers like styrene or methylmethacrylate yielded homogenous polymeric films with thicknesses between 20 and 200 nm linked to the PVA. Subsequent polymerization of a second monomer allowed producing Janus films consisting of two different brush layers. After patterning these films into isolated squares with lateral dimensions of 10 × 10 µm using photolithographic techniques, the PVA layer was dissolved in water. Thereby, the patterned polymer brushes were lifted.
off the substrate and formed a dispersion in the water. Due to entanglements between remaining PVA chains and the polymer brush, the resulting particles were stable without further crosslinking. They were characterized using optical-, fluorescence- and electron microscopy (Figure 2). Currently, we are investigating the possibility to use these nanofilms as encapsulation agents.

In order to expand our range of patterning techniques, we investigated the possibility of patterning positive tone polymer brushes using deep-UV photolithography. In comparison to recent experiments using e-beam lithography [5], this approach allows patterning larger brush areas at lower costs. In first experiments, we exposed brushes from several materials like poly(methylmethacrylate), poly(isobutylmethacrylate) and poly(neopentylmethacrylate) at different doses of 220 nm irradiation. After development, optical and scanning probe microscopy proved that the patterning was successful. Additionally, we investigated the effect of irradiation on the brush’s ability to reinitiate ATRP polymerization. For this, we polymerized styrene on deep-UV patterned brushes. We found that the ATRP polymerization runs much slower in exposed areas of the brush, thus drastically increasing the contrast of the first pattern. Consequently, this new patterning technique allows the patterning of negative tone materials like polystyrene without the application of photoresists. Finally, applying a gray-scale exposure gradient enabled us to produce interesting thickness gradients in polymer brushes.

Acknowledgements:
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References:
Characterization of Molybdenum Disulfide Devices

CNF Project Number: 804-99
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Abstract:
We theoretically and experimentally investigate molybdenum disulfide (MoS₂) for transparent electronics. Using a first principles approach, we determine the plane where charge transport is occurring. Next using optical spectroscopy we determine the number of layers in preparation for fabrication of Hall and field effect devices. Finally we measure the fabricated devices and estimate their properties.

Summary of Research:
Recently there has been increasing interest in flexible transparent electronics in order to maintain industry growth following the theme of “More than Moore.” However getting transparent high performance devices on transparent substrates remains a challenge due to non-ideal transport properties of organic electronics. Here we propose using the recently rediscovered transition metal dichalcogenides for this purpose, of which MoS₂ is one example. These materials are transparent due to their inherent thickness. Furthermore they are semiconducting and have a direct bandgap. We can then think about scaling these devices further than 3D materials since the electrostatic integrity can be maintained for shorter channel lengths. Finally by having a direct bandgap we can think of on chip optical devices. Although MBE technology for these materials is still in its infancy we can investigate their properties to first order using exfoliation techniques and first principles calculations.

We first determine where the charge transport is occurring in the plane of MoS₂ as this has significant implications for device performance and reproducibility. In our calculation we assume the LDA approximation with 30 by 30 k-point grid sampling and an energy cut-off of 100 Rydbergs. We use the quantum espresso solver to solve for the DOS and band structure. Based on our calculations we observe a direct bandgap of 1.78 eV and the density of states near the Fermi level is peaked at the p orbital of the Mo atom as shown in Figure 1. We can conclude that charge transport is occurring in the plane of the Mo not at the surface of the sheet.

Next we wish to fabricate some devices to investigate the carrier type and concentration. It is important to ensure we have a monolayer because the bandgap is indirect for bulk MoS₂ so the properties will be qualitatively different. To this end we perform Raman, AFM, and photoluminescence scans of the exfoliated flake before fabrication. The thickness we obtain for our exfoliated flakes is around 1 nm. The difference between the Raman (Figure 2) and photoluminescence scan (Figure 3) is that while Raman is a resonant process, photoluminescence is not. The photoluminescence spectra for a bilayer has a peak due to a nearby band valley as well as a reduced photon count due to indirect bandgap.

In contrast MoS₂ monolayer has a higher peak due to the direct bandgap and no secondary peak. For the Raman spectra, the phonon modes corresponding to the peaks can be found in Ref. 1. In this case the difference between the layers are harder to discern because the layers are only weakly coupled and each layer has similar phonon modes. Nonetheless the in-plane E₂g and out-of-plane A₁g modes stiffen as additional layers are added, and this shows up as a shift in the Raman spectra as shown.

Figure 1: MoS₂ projected density of states.
After ascertaining the number of layers, we employed e-beam lithography to fabricate our devices because of the size of the flake we needed the resolution and overlay afforded by e-beam lithography. The substrate is 285 nm SiO$_2$ and the contacts are 5 nm Ti and 50 nm Au. We fabricated a Hall device and measured the source drain current across it with gating.

Based on our estimate, our device impedance to be 0.8 MΩ for a 1 µm device. This implies that our device is largely limited by the contact resistance. We plan to try different contact materials as well as anneal the contacts to reduce the contact resistance in our current runs.

In conclusion we have performed preliminary studies of MoS$_2$ both from first principles and experimentally. We find that MoS$_2$ might have potential for transparent electronics but needs further investigation of electrical transport.

**References:**
Structure and Motion of Stacking Faults in CVD-Grown Bilayer Graphene

CNF Project Number: 900-00
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Abstract:
We use diffraction-filtered darkfield transmission electron microscopy (TEM) to study the structure and motion of stacking faults in Bernal-stacked bilayer graphene. We find that corrugations in the original copper growth-substrate correlate closely with AB-BA stacking faults and that additional faults occur at what are presumed to be high stress regions, perhaps during the process of transferring these to TEM grids. We observe the dynamics of these AB-BA boundaries and find that they have nanoscale fluctuation at room temperature on a time-scale of minutes.

Summary of Research:
Monolayer and bilayer graphene—one and two atom-thick sheets of graphite, respectively—have received much attention over the past decade as materials exhibiting superlative electronic and mechanical properties. Part of their appeal to physicists has been in their simplicity as prototypical two-dimensional materials. However, the fact that these materials are often grown and/or studied on a three-dimensional substrate adds a complexity to the system which, while undesirable for many device applications, yields a fascinating and not-yet exploited opportunity for the study of the structure and motion of defects in a simple and easily visualized system. Here we use darkfield transmission electron microscopy to image the structure and motion of stacking faults in bilayer graphene and relate these to both the original growth substrate and transfer process.

Large-grain (30-100 µm) graphene was grown on copper foil (Alfa Aesar Cat#13382), by chemical-vapor deposition (CVD), using the Ruoff group’s enclosure method [1]. In this method, the copper growth foil is enclosed in a second copper foil to limit exposure to growth gases, and placed in an evacuated furnace (base pressure 10^-4 torr). Methane is then flowed at 3 sccm, and hydrogen is flowed at 60 sccm at 980°C for two hours, then cooled. The resulting graphene is predominantly monolayer, with ~ 10 µm six-fold symmetric star-shaped bilayer and multilayer patches at many of its nucleation sites.

Figure 1 shows one of these bilayer regions as imaged by scanning electron microscopy (SEM), and atomic force microscopy (AFM). In the SEM image on the left, the darker hexagon corresponds to bilayer on copper, while the rest of the visible surface is covered in monolayer graphene. As can be seen in the topographic AFM image on the right, the copper surface underneath these bilayer patches tends to be more corrugated than the surrounding monolayer.

On this sample, bilayer corrugations are ~ 10 nm high with a ~ 200 nm separation, while monolayer corrugations are ~ 3 nm high with a 100 nm separation. Due to the alternating positive and negative curvature across these corrugations, we might expect bilayer graphene to have AB-BA stacking faults parallel to these corrugations.

To investigate this, after imaging the graphene on copper by SEM, we spun 50 nm of PMMA on it, etched away the copper, transferred the PMMA and graphene to a carbon...
Quantifoil TEM grid, and baked it at 300°C for three hours to remove the PMMA. By using darkfield TEM, with an aperture in the diffraction plane selecting electrons diffracting from the graphene lattice in a particular direction, we are able to distinguish the two different mirror-symmetric stackings of bilayer graphene (AB and BA) [2].

Figure 2 shows the results of such a darkfield TEM measurement on a region of bilayer graphene that we had previously imaged by SEM while on its original copper growth substrate. In the upper left of the bilayer region, we notice vertical AB-BA stacking faults which seem to correspond to the vertical corrugations in the copper surface in the upper-right SEM image. Additionally, we see that near the indentations in the star-shaped bilayer which are expected to correspond to regions of high stress (marked by S’s), there is a high concentration of stacking faults that propagate inwards to the center of the bilayer patch in the bottom right. In this and many other samples, it seems that the stacking faults observed in CVD-grown and subsequently-transferred bilayer graphene samples are a combination of faults caused by stress during transfer, and those that occurred due to interaction with the original growth substrate.

To investigate how stable these boundaries are at room temperature, we zoom in on a bilayer region that has a high density of faults. Figure 3 shows a series of darkfield TEM images taken over the course of 43 minutes. By filtering for one of the outer diffraction spots, we are able to distinguish the AB-BA transition boundaries (faults), which appear black, from the brighter Bernal-stacked (AB or BA) regions on either side. Over the course the measurement, all of these boundaries fluctuate back and forth on a minute time-scale, and a few nanometer length-scale. The white arrows indicate a few boundaries that have shifted by more than 10 nm.

In this work, we have presented a few measurements demonstrating the origin and dynamics of faults in bilayer graphene, and have highlighted bilayer graphene as a model two-dimensional system for studying the structure and dynamics of crystalline faults.

References:
Design and Validation of High-Efficiency Chopper for Magnetoresistive Sensors

CNF Project Number: 1236-04
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Abstract:
Low-frequency (1/f) noise severely limits the low-frequency performance of magnetoresistive sensors. Based on reluctance analysis and finite-element simulations, this paper reports a high-efficiency chopping system consisting of a flux concentrator and chopper to mitigate the effect of 1/f noise and improve the field sensitivity for magnetoresistive sensors. Experimental results based on the optimized design using an anisotropic magnetoresistance sensor validate the concept. The sensitivity of the sensor with the chopper at OFF and ON positions is determined to be 0.009 and 0.067%/Oe, respectively, which yields a high sensitivity chopping efficiency, namely 72%.

Summary of Research:
Ultra-high-sensitivity magnetic sensors with a magnetic field detection limit in pT/Hz\(^{1/2}\) region have aroused renewed research interest in recent years due to the discovery of novel device structures with large magnetoresistance (MR), such as magnetic tunnel junctions (MTJs) [1, 2] and spin valves [3]. Intrinsic noise is an important property to obtain high signal to noise ratios (SNR). While the frequency-independent white noise sets the ultimate limit to the SNR, 1/f noise dominates in the low frequency region (< ~1 kHz), and severely inhibits the potential application of MR sensors in these regions. In this paper, we report a novel chopper design to suppress the 1/f noise, in which the only moving part is a small, low-mass bar that acts as a low-reluctance magnetic short. We validate the optimized design by experiments using anisotropic-MR (AMR) sensors and demonstrate a chopping efficiency of 72%.

AMR stacks were deposited on 3-inch SiO\(_2\)-coated Si substrates using DC magnetron sputtering with a base pressure below 5 × 10\(^{-9}\) Torr. The stack consists of Si/SiO\(_2\)(500)/Ni\(_{0.81}\)Fe\(_{0.19}\)(25)/Ta(8) (thicknesses in parentheses are in nm). A micrometer thick Py layer was electrodeposited on an area defined by photolithography to make the FCs and chopper elements [4]. The geometry of the chopping system is shown in Figure 1. The distances between the FCs and the shorting bars were set to be 2 \(\mu\)m (OFF position) and 80 \(\mu\)m (ON position). AMR sensors are 20 \(\mu\)m wide and 100 \(\mu\)m long.

The as-fabricated devices were annealed at 240\(^\circ\)C (heating rate: 20\(^\circ\)/min) in Ar flow (flow rate: 80 sccm/min) for 30 minutes and cooled down under a magnetic field of 150 Oe to set the easy axis perpendicular to the long side of the shorting bar [5]. AMR sensors were measured using a multimeter with 1 mA current bias at room temperature in a magnetic field ranging from -40 ~ 40 Oe. The MR ratio was defined as \((R_l-R_t)/(1/3R_l+2/3R_t)*100\), where \(R_l\) and \(R_t\) are the longitudinal and transverse resistances, respectively.

In Figure 2a, the MR of the sensor with and without FCs is recorded as a function of the external magnetic field applied parallel to the short axis of the AMR sensor. Clearly, the MR curve is symmetric and hysteresis free. Figure 2b shows the effect of FCs on the MR curves. Here the sensors are 45\(^\circ\) with respect to the direction of the external field.
magnetic field, which leads to decrease of the MR value from 0.76% to 0.23% and asymmetry of the MR curves.

Figure 3 presents the effect of FCs and chopping system using the optimal design parameters on the MR-H curves of AMR sensors. The field sensitivity of the AMR sensors is defined as MR/H and can be obtained directly from the MR-H curves [6]. From Figure 3a, the field sensitivities of the AMR sensor with and without FCs, namely S_{FC} and S_{AMR}, can be determined to be 0.081 and 0.008%/Oe, respectively, which yields a gain of 10. This confirms the function of FCs to enhance the magnetic flux.

In Figure 3b, the MR-H curve is steeper with the chopper in ON position compared to that with the chopper in OFF position. This implies that with the same external magnetic field change the chopper in OFF position results into smaller resistance change and subsequently smaller sensitivity due to the shunting effect of the chopper. We can define a sensitivity chopping efficiency, \( \eta_s \), as \( \frac{(S_{ON} - S_{OFF})}{S_{FC}} \times 100\% \), where \( S_{ON} \), \( S_{OFF} \) and \( S_{FC} \) are sensitivity with the chopper in ON position, OFF position, and with no choppers, respectively. The sensitivities for OFF and ON positions, \( S_{OFF} \) and \( S_{ON} \), are determined to be 0.009 and 0.067%/Oe, respectively. The sensitivity with the FCs only (\( S_{FC} \)), by contrast, is 0.081%/Oe. So \( \eta_s \) is determined to be 72%.

Our results herein present the best case scenario and they validate the high-efficiency chopper design.

References:
Colloidal Self-Assembly-Directed Laser-Melt-Induced Crystalline Silicon Nanostructures

CNF Project Number: 1356-05
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User: Kwan Wee Tan

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Abstract:
We describe a rapid, facile technique coupling colloidal crystal templating with laser thermal processing to fabricate crystalline Si nanostructures with deterministic non-close-packed symmetry. Two- and three-dimensional (2D, 3D) colloidal crystals are grown as porous templates on a single-crystal Si substrate. Pulsed excimer laser irradiation is used to induce a transient melt-crystallization conversion of the amorphous silicon filled in the templates to the crystalline phase. The pattern transfer yields are high and long-range hexagonal order is maintained. The method is highly adaptable as an alternative strategy to generate silicon nanostructures of other symmetries for use in applications such as energy generation and storage.

Summary of Research:
Ordered nanostructures based on silicon grown by colloidal self-assembly have been extensively studied and widely adapted for nanofabrication and many potential applications, for example, photonic crystals and solar cells [1-7]. In particular, non-close-packed crystalline silicon nanostructures are highly desirable for their enhanced electrical conductivity, photonic and other surface properties [5-10].

In this work, we combined colloidal crystal template formation with laser thermal processing to generate crystalline silicon (c-Si) nanostructures with non-close-packed symmetry [11]. Figure 1 schematically shows the experimental procedure.

We began by growing a hexagonally arranged close-packed (hcp) monolayer colloidal crystal of 350 nm or 530 nm polystyrene (PS) spheres surrounded by silica (SiO₂) using the vertical deposition method. The latex beads were calcined in air at 500°C, leaving behind a highly ordered SiO₂ inverse monolayer. The SiO₂ template was subsequently filled with a 100 nm thick amorphous Si (a-Si) overlayer and irradiated with a 40 ns full-width-half-maximum pulsed XeCl excimer laser (308 nm wavelength) in air to induce the transient melt conversion of a-Si to the crystalline phase (~ 20-100 ns).

Finally, the template was removed in concentrated hydrofluoric (HF) acid solution, leaving hexagonally arranged non-close-packed (hncp) Si nanostructured array as seen in Figure 1c.

Using the 530 nm PS colloidal template with a single laser irradiation pulse at 600 mJ/cm², we grew 2D hncp arrays of c-Si nanostructures with an average width and height of around 410 and 300 nm, respectively.

Figure 2 shows the hncp c-Si nanostructures generated from a smaller, 350 nm PS colloidal template and four sequential
laser pulses at 500-700 mJ/cm², with an average width of 170 nm and height of 60-70 nm height. Atomic force microscopy analysis indicated a ~ 100% hexagonal pattern transfer from the (350 nm) cp SiO₂ inverse colloidal template to the ncp c-Si nanostructures.

The method is easily adaptable to generate more complex 3D ordered macroporous c-Si inverse opals with hncp symmetry. We began with a ~150 nm thick plasma enhanced chemical vapor deposited (PECVD) α-Si in a (350 nm) multilayered SiO₂ inverse colloidal crystal template. The SiO₂/α-Si core-shell composite inverse opal was subsequently irradiated with a total of 43 sequential laser pulses at fluences from 5-550 mJ/cm². Selective HF etching of the SiO₂ template results in a ~ 1.3 µm thick c-Si single interconnected network of hncp inverse opal morphology, see Figure 3.

In conclusion, we have demonstrated a rapid and highly versatile method using colloidal crystal templates coupled with pulsed excimer laser-induced melting to obtain ncp c-Si arrays of varying scales at ambient conditions. In analogy to results on block copolymer templates, this approach may also be applied to form not only single-crystal homoepitaxial but also heteroepitaxial ncp nanostructured arrays on Si, enabling new functionalities and the engineering of novel device prototypes [12].

References:
Abstract:
We have demonstrated the validity of high-throughput measurements of ionic conductivity in oxygen-ion conductors. Y-Zr-O composition-spread thin films were prepared by reactive RF co-sputtering. Lithographically-defined planar interdigitated electrodes allow us to infer the conductivity with good accuracy. The intragrain oxygen ion conductivity of thin film was systematically measured as a function of yttria concentration over the range 2 mol % to 12 mol %; the results are consistent with measurements on bulk materials reported in the literature. Validation of this technique means that it can now be applied to novel chemical systems for which systematic bulk measurements have not been attempted.

Summary of Research:
The development of solid oxide fuel cells (SOFCs) into a common commercial technology remains limited by the elevated operating temperatures required for ionic conductivity in the cell electrolyte. Two classes of cells are considered: conventional large-scale (megawatt-range) devices operating at 600-1000°C, and small thin-film-based (watt-range) devices operating at < 500°C. In either case, operation at lower temperature is desirable, but not feasible because the thermally-activated motion of O\textsuperscript{2-} ions becomes too sluggish. We are developing the techniques needed to explore new materials for these applications.

Our approach is to prepare compositionally-graded thin films and characterize the bulk (intragrain) ionic conductivity as a function of position. In a single experiment we can potentially determine the ionic conductivity of hundreds of compositions. This project focused on validating the technique—demonstrating that we can reliably measure conductivities in a well-documented model chemical system, Y-Zr-O. Addition of sufficient Y to monoclinic ZrO\textsubscript{2} stabilizes the cubic phase, hence this system is referred to a “yttria-stabilized zirconia” (YSZ).

YSZ composition-spread thin films were deposited on 75 mm Φ Si or SiO\textsubscript{2} substrates by reactive RF co-sputtering using elemental Zr and Y targets [1]. The composition gradient was roughly 0.25-1 at% per mm and the films are roughly 150 nm thick. The films were annealed in air at 1000°C for one hour soak to achieve good crystallinity and to preclude significant structural changes during conductivity measurements at ~ 500°C. For electrical measurements, two electrode configurations (see Figure 1) were tested, namely the out-of-plane and in-plane (interdigitated electrode, IDE) geometries. Counterelectrode dots (Figure 1, left) were 200 µm Φ Pt deposited by evaporation using a metal shadow mask. IDEs were created using photolithography with finger length, width and interelectrode spacing set to 750 µm, 5 µm and 5 µm, respectively. Devices were created on 1 mm centers covering the substrate.

Devices were measured in a custom-built high-temperature probe station with better than ± 3°C uniformity, using micromanipulators to position Pd-needle probes on the contact pads. The ionic conductivity was evaluated using electrochemical impedance spectroscopy (EIS) measurements in the air ambient using an LCR meter at frequencies ranging from 100 Hz to 1 MHz. The complex impedance, Z, is then fitted to a model to extract the ionic conductivity. Figure 2 illustrates typical data obtained for Zr\texttextsubscript{0.96}Y\texttextsubscript{0.04}O\textsubscript{1.98} at 440°C using a Nyquist plot (-Im(Z) vs. Re(Z)) with frequency as
a parameter. Parallel-plate capacitor (“out-of-plane”) and IDE (“in-plane”) devices yield values for Z due that are different not only in scale because of the different geometric proportions, but also in shape due to the difference in electrode spacing. The solid lines in Figure 2 represent model fits, where the circuit model is indicated in the inset. It is readily apparent that the IDE device fits the model well, while the parallel-plate device evidently requires a more sophisticated model. The resistances $R_1$ and $R_2$ correspond to the ionic motion associated with intragrain and intergrain conductance, respectively. The circuit elements denoted $Q$ represent “constant-phase behavior” used to model the distribution of activation energies associated with ionic motion.

Figure 3 shows the intragrain O-ion conductivity inferred for our YSZ composition spread film as a function of composition (open symbols), as measured at 440°C. For comparison, measurements reported for bulk single-crystal [2] and polycrystalline-ceramic [3] samples are shown as solid symbols. The agreement is excellent. Our measurements represent only the first step in identifying a good oxygen-ion conductor: follow-up studies would be needed to confirm that the conductivity is due to ions rather than electrons, to characterize the temperature dependence of the conductivity, to characterize the intragrain conductivity, etc.

This work [4] demonstrates that reliable measurements of ionic conductivity can be made using codeposited composition-spread thin films, in that the measurements yield values that are consistent with the intragrain conductivity reported for bulk samples. This approach allows high-throughput measurement of behavior over a wide range of compositions with 0.5 mol% resolution in multication oxide systems. We used YSZ as a model system, varying the yttria concentration over the range 2 mol% to 12 mol% $Y_2O_3$. The in-plane electrode configuration was found to be more suitable than out-of-plane configuration measurements on 150 nm films. The intragrain ionic conductivity of the YSZ thin films is essentially identical to that of bulk specimens, with a peak conductivity value of about $3 \times 10^{-4}$ S cm$^{-1}$ at 440°C for the optimum $Y_2O_3$ concentration near 8 mol%.

References:
Electrical Characterization of Nanocrystal Arrays

CNF Project Number: 1645-08
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Abstract:
Notwithstanding the rapid progress in nanocrystal-based prototype devices, significant knowledge gaps concerning the electronic structure and charge transport in nanocrystal thin films remain. Phenomenological models adopted from transport in bulk semiconductors have enabled much of the progress to date; however, these models are less satisfying with regards to mechanistic clarification as they do not adequately address interfacial transport in coupled quantum confined systems. This knowledge gap is likely to impair future advances in nanocrystal based devices. We apply advanced optical and electrical characterization techniques to achieve a deeper understanding of charge transport in nanocrystal films.

Summary of Research:
We have studied charge transport in PbS NC films that coupled via chemical treatment with ammonium sulfide. Temperature-dependent mobility measurements reveal decreasing mobility with increasing temperature. In contrast to thermally activated hopping, this trend has previously been interpreted as ‘band-like’ charge transport through delocalized states. However, the electronic disorder in NC films is too large to support the interpretation as in terms of band conduction.

The effect of temperature on carrier concentration on the other hand is not yet well understood and is likely to impact the negative temperature coefficient of the charge mobility. Our current experiments aim to clarify this effect.

Another critical unresolved question is: ‘What limits the mobility of charges through highly coupled nanocrystal films?’

The answer to this question has important implications both on understanding the basic charge transport mechanism as well as on the performance limits of nanocrystal thin film in photovoltaic devices.

Charge trapping is commonly cited as the cause of low mobility for localized carriers and hopping transport, but this does not adequately describe the limitation in ‘band-like’ transport. We have adopted a model of charge transport in polycrystalline thin films to describe the charge transport limitation in context of grain boundary scattering. This model predicts charge transport limited by acoustic phonon scattering at high temperatures and a \(\mu \sim T^{-5/2}\) temperature dependence; at low temperatures grain boundary scattering dominates.

Our current work focuses on refining this hypothesis by testing NCs with controlled superstructure and degree of interconnection; this builds on recent advances in our group to create superstructures with controlled symmetry and degree of connection.
Development of a Monochromatic Phonon Source for Studying Nanoscale Phonon Heat Transport

CNF Project Number: 1746-09
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Affiliations: 1. Materials Science and Engineering, Cornell University; 2. Cornell NanoScale Science and Technology Facility, Cornell University

Primary Source of Research Funding: Start-up Funding, KAUST-Cornell Center for Energy and Sustainability, National Science Foundation Agreement No. DMR-1149036, DOE Office of Basic Energy Science under Award Number DE-SC0001086

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Abstract:
Thermal transport by acoustic phonons in nanoscale dimensions differs markedly from behavior in bulk materials because of the phonons’ interaction with surfaces and interfaces. To improve our understanding of such effects, we are developing a microfabricated phonon spectrometer. We have demonstrated a prototype of such a device, generating a controllable, non-thermal distribution of acoustic phonon modes ranging from ~100 to ~800 GHz and measuring its transmission through silicon microstructures and nanostructures at a temperature of 0.3K. To generate and detect the non-thermal phonons, we exploit excitation and decay processes in superconducting tunnel junctions (STJs) attached to the micro/nanostructures. By these studies, we seek to understand thermal transport at the nanoscale in order to improve the efficiency of nanostructured thermoelectric materials.

Summary of Research:
Phonon spectroscopy using superconducting tunnel junctions (STJs, consisting of two superconducting metal films separated by a tunnel barrier) has been described by Kinder, et al. [1]. To probe ballistic phonon transport in silicon, we construct devices in which an STJ phonon generator and detector face each other across a microstructure (Figure 1). We first perform shallow KOH etch of a <100> silicon wafer to form ‘mesa’ type microstructures 0.8 μm high, 200 μm long and 7 to 30 μm wide (Figure 2). To attach STJs in contact with the mesa sidewalls, we perform photolithography using the GCA 6300 DSW 5X g-line stepper, followed by deposition of two thin films of aluminum separated by an in situ-grown oxide barrier (performed in a dedicated evaporating chamber outside of CNF), followed by lift-off of the photoresist. We form 112 chips per wafer and twelve devices on each chip.
We form the phonon detectors in a split ‘SQUID’ type geometry (Figure 2) to enable suppression of Josephson currents during measurement.

We have tested the devices at a temperature of 0.3 K in a helium-3 refrigerator [2]. The emitter STJ is biased at voltages greater than twice the aluminum superconducting bandgap of 170 microeV, thereby injecting free electrons into excited states of the superconductor. Decay of these excited states emits a highly non-thermal distribution of phonons, which the STJ radiates into the adjacent silicon. The phonon spectral distribution comprises a monochromatic component at an energy of ~ 340 micro eV plus a broad distribution having cutoff energy determined by the bias voltage. Modulation of the bias voltage therefore allows the phonons at this cutoff energy to be isolated in the measurement, offering spectral resolution as low as ~ 10 GHz over a range of hundreds of gigahertz. The detector STJ functions by collecting incident phonons and measuring a tunneling current of the locally-excited electrons. We confirm the non-thermal nature of the phonon distribution by observing a step-change in signal level at a generator voltage four times the superconducting bandgap, and confirm direct ballistic transport by selectively blocking the direct path through the silicon (Figure 3).

In upcoming measurements, we seek to force the phonon flux to traverse silicon nano-channels in order to reach the detector (Figure 4). We form the nano-channels using electron-beam lithography (JEOL JBX-6300FS) followed by DRIE plasma etch (Unaxis 770). By observing the dependence of the transmission on phonon frequency, we will determine the mean free path of the phonons in such nanostructures and relate this to both phonon wavelength and nanostructure geometry, thereby improving fundamental understanding of phonon-mediated thermal transport in nanostructures.

References:
Vertically Aligned Carbon Nanotube Membrane for Solar Hydrogen Generation

CNF Project Number: 1782-09
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User: Gregory Pilgrim

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Primary Source of Research Funding: US Department of Energy
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Abstract:
Vertically aligned carbon nanotube (VA-CNTs) membranes for an artificial photosynthetic device were synthesized. Fabrication of VA-CNTs proceeds by chemical vapor deposition (CVD) at 800°C. Following nanotube growth, an epoxy is spin-coated onto the array to make a self-supporting, chemically resistant, and electrically conductive membrane.

Summary of Research:
The development of new energy sources is a necessity for satisfying the increasing worldwide demand for energy. Out of the possible energy resources, the sun is the most widely available and most abundant source for long-term carbon free energy. However there are still many challenges in making solar energy viable economically and socially [1, 2]. In this project, we are developing vertically aligned carbon nanotube membranes for a bio-and nano-inspired integrated system for producing hydrogen from solar energy. VA-CNTs will act as a linkage between a light harvesting bio-molecule and hydrogen producing catalysts. Due to their exceptional electrical properties, NTs can efficiently accept and separate electrons from the photo-excited donor and transport them to the catalyst even over long distances. At the same time, the impermeable VA-CNT membrane can provide a physical barrier between the light harvesting and hydrogen producing components, separating them into different chemical environments.

Dense forests of vertically aligned carbon nanotubes were synthesized by chemical vapor deposition (CVD). Catalyst layers of 5-10 nm of Al₂O₃ followed by 3-12 nm of Fe are deposited on a Si wafer by e-beam evaporation. The thin iron layer is annealed at 800°C under a hydrogen flow [3]. Al₂O₃/Fe form catalyst particles that are 8-12 nm as analyzed with atomic force microscopy (AFM). These particles seed CNT growth at 750-800°C when in an environment comprised of a carbon source (ethylene) and a carrier gas (argon).

The CNT forming reactions are carried out in a single joint tube furnace [4] where the VA-CNTs grow to heights of 100-150 μm (Figure 1). After growth, the VA-CNTs are coated with a commercially available two part epoxy (Figure 2) such that the bulk NT array is completely coated, while ~ 10 μm of CNT tip are left exposed (Figure 3). The exposed NTs are therefore available to conduct electrons, and in fact, we verified through current-voltage spectroscopy that electron transport through the array was Ohmic. The resulting self-supporting membranes exhibit a trans-membrane resistivity on the order of 0.1Ω/μm. As CNTs have already been shown capable of transporting ions [5] future work is aimed at facilitating proton conduction, as a material capable of transporting electrons and protons would be a significant step towards solving the water splitting problem [6].

References:
Figure 1: SEM image of vertically aligned carbon nanotubes.

Figure 2: SEM of the epoxy penetrating fully into the NT array.

Figure 3: SEM image of carbon nanotubes exposed above the epoxy-coated array.
Calculation of the Temperature Dependent Superconducting Superheating Field

CNF Project Number: 1959-10  
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User: Mark K. Transtrum

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Primary Source of Research Funding: National Science Foundation Grant No. DMR-0705167  
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Abstract:
Modern accelerators use superconducting radio-frequency (RF) cavities to accelerate particles. These cavities are typically operated well above the critical magnetic field at which it is thermodynamically favorable for the flux to penetrate the superconductor. However, an energy barrier to flux penetration allows cavities to operate in a meta-stable, superheated state. The superheating field is the point at which this barrier vanishes, allowing flux to enter the superconductor, and represents a fundamental limit to the operation of superconducting RF cavities. This work is a numerical calculation of the superheating field using the semi-classical theory of Eilenberger, which accounts for the temperature dependence of the superheating field.

Summary of Research:
Superconductors are an essential component of modern accelerators, used both as magnets to bend the path of the particles and as resonance cavities to accelerate them. While the former are pushed to their limits in the type-II regime where magnetic flux lines penetrate and are pinned to impurities, the latter must change the sign of the induced electric field in gigahertz synchrony with the electron packets. The time-varying electric-fields induce magnetic fields which must not penetrate the superconductor; otherwise their motion would dissipate large amounts of heat and quench the superconductivity.

Thermodynamically, it is energetically favorable for magnetic fields to penetrate the superconductor for fields larger than that \( H_c \) (for type-I materials) or \( H_{c1} \) (for type-II materials).
However, in practice, there is an energy barrier to the penetration of magnetic flux at the superconducting surface, allowing for the operation of accelerating cavities with fields much larger than either $H_c$ or $H_{c1}$. In this regime, the superconductor operates in a meta-stable, superheated state. In principle, this state can persist with magnetic fields up to the so-called superheating field at which point the energy barrier vanishes and magnetic flux enters the material. The superheating field therefore represents a fundamental limit in the operation of superconducting accelerator cavities.

Calculating the superheating field is a linear stability problem complicated by a pre-existing spatial in homogeneity in the superconducting order parameter and vector potential. Previous estimates of the superheating field have been made within the relatively simple Ginzburg-Landau theory [1]. However, the predictions of Ginzburg-Landau theory are quantitatively only accurate for temperature very near the critical temperature. Niobium, the most common material used in cavity construction, has a critical temperature of about 9 K. In contrast, the cavities are operated at about 2 K, in a regime where the Ginzburg-Landau theory requires significant corrections. These temperature dependent corrections have been calculated using Eilenberger theory in the limit of large Ginzburg-Landau parameter [2]; however, these calculations also have limited applicability since niobium has a Ginzburg-Landau parameter of about one. The current work goes beyond previous results by using the Eilenberger formalism—involving self-consistent solutions with anomalous Greens functions, Matsubara frequencies, and integrals over the Fermi surface, to calculate the temperature dependence of the superheating field over a wide range of Ginzburg-Landau parameters and temperatures.

In addition to the added complexity of the Eilenberger formalism, the present calculation is numerically challenging due to the necessity of including a large number of Matsubara frequencies and Gauss points on the Fermi surface to obtain reasonable convergence, as shown in Figure 1. Fortunately, solving the Eilenberger equations for the anomalous Greens functions at different Matsubara frequencies and Fermi surface points is trivially parallelized. For solutions near the operating temperature, we found it necessary to solve the equations at several thousand Fermi surface points for about ten values of the Matsubara frequencies. To complete the linear stability calculation, it is also necessary to calculate the sensitivity of these solutions to infinitesimal fluctuations in both the superconducting order parameter and vector potential. In all, this process required the solution of over two million independent differential equations at each iteration of the search. By distributing this problem over tens of cores at the CNF computing cluster, we were able to obtain solutions within a few weeks for a single value of the temperature and Ginzburg-Landau parameter.

We repeat the calculation for several values of the temperature and Ginzburg-Landau parameter. Some preliminary results are given in Figure 2 for several values of the Ginzburg-Landau parameter at the operating temperature of niobium. Although the results agree qualitatively with those of Ginzburg-Landau theory, we find that the predictions of Eilenberger theory are about 7% higher for niobium at operating temperature.

The Eilenberger formalism opens the possibility of even more precise predictions about the superheating field. In particular, it is also possible to account for anisotropy in the Fermi surface and for impurities in the niobium, both of which could be significant corrections. It may also be possible predict the dependence of the superconducting gap on the induced magnetic field, a quantity important for measuring the quality of a superconducting cavity. Each of these calculations could be performed with the numerical framework developed here.

References:
Fabrication of Nanoscale Silicon Fracture Test Specimens and Calculation of Ideal Strength of Silicon

CNF Project Number: 1982-11  
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Web Site: https://confluence.cornell.edu/display/frac/Strength+of+Nanoscale+Systems

Abstract:
This work is an extension of that performed by Alan, et al. [1, 2], investigating the effect of surface characteristics on the strength of nanoscale silicon (Si) structures. Progress, utilizing CNF, during the 2011/2012 year has been focused in two main areas. We have replicated the process of Alan, et al., to fabricate nanoscale Si beams with current equipment and techniques, and have made progress in modeling Si fracture at the atomic scale using molecular dynamics. The molecular dynamics calculations have been completed using CNF’s Nanolab computing cluster.

Summary of Research, Test Specimen Fabrication:
Alan, et al., developed a method to fabricate nanoscale Si beam structures based on work by Wang, et al. [3]. We spent a large amount of effort during 2011/2012 to update Alan’s procedure with new equipment and techniques. Samples are fabricated from <111>-oriented Si wafers. The fabrication involves two sets of photolithography and reactive ion etching (RIE) steps. The first etch depth is done to a depth of about 200 nm and roughly controls the thickness of the final beam structure. Before the next round of lithography and etching, a 100 nm thermal oxide layer is grown. The second RIE step is done to a much greater depth of about 10 µm and forms the trench above which the final beam structure will be suspended. At this point the beam is still connected to the substrate along its entire bottom side. Before removing the Si from under the beam, the devices are cleaned with a modified RCA process [4]. The beams are then anisotropically etched using KOH and tetramethyl ammonium hydroxide (TMAH). This etch terminates on Si {111} surfaces. Because the bottom side of the beams are <111>-oriented, and the top and sides are protected by the thermal oxide, the beams themselves are not etched away. Finally, the thermal oxide is removed with a buffered oxide solution. The resulting beam structure is shown in Figure 1.

Failure on Si {111} planes under mixed loading using molecular dynamics: Work has also been done on calculating the strength of Si {111} planes under mixed mode loading conditions. This is being done using the molecular dynamics code Large Atomistic and Molecular Massively Parallel Simulator (LAMMPS) and the modified embedded atom method (MEAM). Roundy and Cohen have calculate the theoretical strength of Si {111} planes under pure tension and pure shear in a <112> direction using density functional theory [5]. Because the top and bottom of the nanobeam shown in Figure 1 are <111>-oriented, the {111} fracture planes are not orthogonal to the long axis of the beam. Even though the middle of the beam is under approximately pure tensile loading at fracture, the {111} planes are under a mix of tensile loading as well as shear in both directions. For this reason, the Roundy and Cohen results cannot be directly compared to fracture data obtained from such beams.

Figure 1: Nanoscale Si fracture test specimen fabricated at CNF.
Thus far, the MEAM calculations have successfully reproduced the failure behavior reported by Roundy and Cohen for the pure tension and pure shear cases. The stress-strain behavior for the pure shear case is shown in Figure 2. MEAM calculates a stress and strain at failure of 35 GPa and 17% in tension, and 9.25 GPa and 28% in shear. Roundy and Cohen report 22 GPa and 17% in tension, and 6.8 GPa and 30% in shear [5]. The failure strains produced by MEAM agree with the results of Roundy and Cohen. The failure stresses are higher in the MEAM calculations because Roundy and Cohen allow all strains in the plane of the {111} fracture surface to relax. The periodic boundary conditions of the MEAM calculation do not allow for this relaxation, and thus a higher stress is required to reach the strain at which failure occurs. Work is currently in progress to extend these results to mixed mode loading cases.

References:

Figure 2: Stress-strain data calculated using MEAM for an infinite and idea Si crystal.
Bismuth Electrodeposition on GaAs <110>

CNF Project Number: 2036-11
Principal Investigator: Joel D. Brock$^{1,2,3}$
Users: Xin Huang$^{1,2}$, Manuel Plaza$^{1,2}$, J. Y. Peter Ko$^{1,2}$


Primary Source of Research Funding: Energy Materials Center at Cornell
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Abstract:
We evaporate Au$_{88}$Ge$_{12}$/Au on GaAs wafers at the Cornell NanoScale Science and Technology Facility (CNF) to fabricate an ohmic contact on the back side of GaAs wafers.

Summary of Research:
We use in situ x-ray diffraction to characterize the structural evolution of the thin-film during electrochemical deposition.

In order to use GaAs <110> as an electrode for electrochemical reactions, we need to fabricate an ohmic contact on the back side of the wafers. To achieve this, we evaporate an Au$_{88}$Ge$_{12}$/Au bilayer. First, we thermally evaporate 80 nm of Au$_{88}$Ge$_{12}$ eutectic alloy at a rate of 0.1 nm/s at 10$^{-6}$ Torr. Immediately after, we deposit 200 nm of Au at 0.1 nm/s at 10$^{-6}$ Torr by e-beam evaporation. After that, we anneal the GaAs wafers at 380° in Ar/H$_2$ atmosphere for 90 seconds.

In Figure 1, we show a schematic representation of the final GaAs/Au$_{88}$Ge$_{12}$/Au structure.
Origin of the Strain Sensitivity of Superconducting Triniobium-Tin

CNF Project Number: 2053-11
Principal Investigator and User: Matthias G.T. Mentink

Affiliation: Superconducting Magnet Group, Lawrence Berkeley National Laboratory, Berkeley, USA
Primary Source of Research Funding: Lawrence Berkeley National Laboratory, under Department of Energy contract nr DE-AC02-05CH11231
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Abstract:
The effect of axial strain on the electron DOS and phonon DOS of Nb$_3$Sn is investigated, in order to explain the origin of the strong effect of strain on the superconducting properties of Nb$_3$Sn.

Introduction:
The superconductor niobium-tin or triniobium-tin (Nb$_3$Sn) is a widely used “workhorse” superconductor for high magnetic field applications, such as the Iter fusion project [1]. Due to thermal contraction differences and Lorentz forces, the superconductor is exposed to considerable deformation during operation. The superconducting properties of Nb$_3$Sn are quite sensitive to strain, impacting the performance of applications using this superconductor [2].

As Nb$_3$Sn is a low temperature superconductor, the circumstances leading to formation of the superconducting state are fairly well understood. In particular, the critical temperature is related to the electron density of states at the Fermi level and the phonon density of states [3], which implies that strain either affects the electron DOS, or the phonon DOS, or both.

Progress:
DFT calculations are performed using Quantum Espresso [4]. Structural optimizations are performed on a deformed cell, after which the electron DOS at Fermi level and the phonon DOS is calculated. Preliminary electron DOS results indicate a large change in electron DOS with strain (see Figure 1). Furthermore, a calculated phonon spectrum is compared to a measurement and found to be quantitatively consistent (see Figure 2).

Outlook:
Looking forward, emphasis will be placed on calculating the phonon DOS at various strain states. Using the calculation results, the strain sensitivity of the superconducting properties of the material will be calculated and compared to experimental results.

A brief overview of this work with the CNF computing facility was given in a DOE review poster session as part of a general overview of my Ph.D. research.

Summary of Research:
The calculation results indicate that the electron DOS at Fermi level of Nb$_3$Sn as strongly affected by strain. The effect of strain on the phonon DOS of Nb$_3$Sn is ongoing, but preliminary results are quantitatively consistent with experimental data.

References:
Figure 1: Electron DOS as function of strain along <100> direction.

Figure 2: Calculated phonon DOS (at -1.5% axial compression) in light grey versus measured phonon DOS in black [5].
Mass Production of Patterned Nanowires via Electroplate-and-Lift Lithography

CNF Project Number: 2079-11
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Abstract:
Electroplate and Lift (E&L) Lithography [1-4] has been developed as fast, simple, scalable technique for the controlled, solution-based, electrochemical synthesis of patterned metallic and semiconducting nanowires. E&L lithography is the only known process that allows patterned nanowires to be mass-produced, without requiring any additional vacuum or cleanroom processing after the initial fabrication of the patterned template. The present work aims to automate both the electroplating and the liftoff steps through roll-to-roll processing. Patterned templates with over two million features have been produced, which should enable the production of nanowires at a rate of approximately four hours per gram.

Summary of Research:
An overview of the E&L lithography process is shown in Figure 1 [1]. E&L employs a reusable, non-sacrificial, multi-layered, lithographically patterned ultrananocrystalline diamond (UNCD)™ template for electrodeposition. The electrodeposition itself may be performed from either an aqueous or ionic liquid solution. To date, micro- or nanowires composed of 26 distinct elemental metals, alloys, compound semiconductors, and even conductive polymers have been deposited [2]. Following electrodeposition, the nanowires may be removed by mechanically lifting them away from the template with scotch tape or other adhesive polymers, thus regenerating the template surface for subsequent depositions. Wires of the same or a different diameter, composed of any desired electrochemically depositable material, may be electroplated.

As is diagrammed in Figure 2 [3, 4], the classic E&L template is made from alternating layers of intrinsic UNCD, which is insulating, and nitrogen-incorporated UNCD (N-UNCD), which has nearly metallic conductivity. The diamond layers are lithographically patterned and etched in oxygen plasma to expose only edges of the N-UNCD layer, which is 80 nm thick. Intrinsic UNCD is used to isolate the top and bottom surfaces of the N-UNCD layer from the electrochemical bath, thereby confining the initial nucleation of the nanowire to the patterned edges of the N-UNCD. The thickness of the conductive N-UNCD layer establishes the minimum achievable wire diameter. The maximum wire diameter is determined by the deposition time, independent of feature sizes in the template [3]. In alloy systems such as copper-tin bronze, the composition of the wires may be controlled through control of the composition of the solution [4].
Unlike all other known methods of patterning nanowires, E&L lithography allows patterned nanowires to be inexpensively mass-produced, without requiring any additional vacuum or clean room processing after the initial fabrication of the template. Although at present the setup of each electrodeposition bath and the wire removal are both performed manually, it is easy to envision the automation of both the electroplating and the liftoff steps through roll-to-roll processing, as depicted in Figure 3. A silicon wafer, patterned with a UNCD-based E&L template, would be attached to the wheel on the left, and rotated through the electrochemical bath. The wheel on the right would be coated with an adhesive polymer, to remove the newly produced nanowires and regenerate the template. This pickup wheel would rotate through a rinse bath, where wires would either be removed from the wheel by sonication, or transferred to a final collection spool coated with an even stronger adhesive.

This nanowire mass production instrument may be operated in either of two modes, interchangeable simply by changing the template. For the mass production of patterned nanowires, one may use a wafer with millions of patterned features arranged in a ring, as shown in Figure 4. This prototype template was made from a mask of ~ two million flashes. Assuming all lateral dimensions and spacings are greater than 2 µm, a 100 mm wafer can typically accommodate 1-5 million patterned features within the deposition ring. The rate of nanowire production with the template in Figure 4 is calculated to be approximately one gram every four hours. Gram quantities of shaped nanowires may have many applications, such as use in RFID taggants, control of viscosity in polymeric and colloidal materials, and the development of self-sharpening tools. Alternately, if the template pattern were a continuous circle, with its circumference positioned to coincide with the deposition ring in Figure 4, the single edge could be used to produce a continuous wire with a diameter between 100 nm and 1 µm, at a rate of approximately one kilometer per day. If made of a thermoelectric material such as bismuth, a nanowire with this ~ 109:1 aspect ratio should provide unambiguous experimental evidence related to a theoretical prediction by Dresselhaus, et al. [5], that thermoelectric materials which are nanoscale in one dimension and macroscopic in another are expected to display an enhanced Seebeck Effect.

References:
Disappearance Mechanisms of Sub-100 nm Diameter Aluminum Dots on Sapphire during Annealing

CNF Project Number: 2089-11
Principal Investigator: Prof. Richard P. Vinci
User: Jeffrey Biser

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Primary Source of Research Funding: National Science Foundation
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Abstract:
It has been observed by the authors that aluminum nano-dots on single-crystal sapphire substrates will disappear entirely when annealed between 450°C and 1200°C, provided they are ~ 100 nm in diameter or smaller. Such dots have been fabricated at the CNF by electron-beam lithography and liftoff of thermally evaporated metal for a series of annealing experiments to identify the mechanism(s) responsible.

Motivation:
A novel and cost-effective technique for improving the efficiency of compound semiconductor devices built on sapphire substrates has been developed [1] and patented [2] at Lehigh University. This technique requires the fabrication of large arrays of nanoscale “dots” on the substrate in order to improve the quality of the grown semiconductor, leading to substantial gains in final device efficiency. To avoid the hazards inherent in fabricating nanostructures in oxides, we have made use of an annealing process for converting metallic aluminum films on single-crystal epitaxially polished sapphire substrates into sapphire that is epitaxially commensurate with the original substrate [3]. Applied to pre-existing metal nanostructures, the result is the same. Little is known about the behavior of nanoscale aluminum objects under the combined conditions of size scale, temperature range, and chemical environment which are required for the conversion process, as these conditions are extreme as compared to those typical of integrated circuit aluminum conductor structures.

We have regularly observed that during the conversion process, metal dots smaller than a certain threshold diameter (~100 nm) tend to either dramatically deform or disappear entirely during the high-temperature step of 24 hours at 1200°C, yet all dots remain unchanged after the first step of 450°C for 24h. A systematic study of aluminum nanodots annealed in the 450°C to 1300°C range was deemed necessary in order to identify the mechanism responsible for this disappearance and to determine an appropriate technique to prevent such failures in the future. The knowledge obtained in this experiment will also prove instrumental in refining the times and temperatures used in the conversion process, which had been arrived at empirically and originally tailored for use with blanket films as opposed to isolated nanostructures.

Experimental Summary:
The suspected mechanisms for material transport in the relevant temperate range are evaporation, surface diffusion, fracture at the metal/oxide interface leading to wholesale removal of the dot, or extrusion through cracks or flaws in the native oxide shell. It is possible to use the distinct temperature ranges at which each of these mechanisms are expected to be active as a diagnostic tool. In January of 2012, we used the electron-beam lithography capabilities at the CNF to fabricate suitable arrays of aluminum nano-dots for such a study. Dots in the 50-200 nm diameter range were formed by exposure of poly-methymethacrylate on sapphire (JEOL JBX-9300FS) and subsequent deposition and liftoff of 100 nm of thermally evaporated aluminum (CHA thermal evaporator). These dots were then transported to Lehigh University for characterization by scanning electron microscopy (Hitachi 4300 SEM) at normal incidence and 45° tilt, annealing, and re-characterization post-anneal.

At the time of writing, the following conditions have so far been studied: (A) 700°C for one hour, (B) 900°C for one and (C) 12 hours, (D) 1000°C for one hour, and (E) 1200°C for one and (F) 24 hours (six samples total).

Dots of all sizes remain present, even if deformed, for all of the temperatures tested below 1000°C. Complete disappearance of dots has only been observed for diameters less than 70 nm for samples (D) and (E), and in dots 80 nm or smaller for sample (F). Spreading (“slumping”) of dots of all sizes is indicated by increased diameter and changed shape at temperatures above 1000°C, and to a smaller degree in sample (C). Slumping in sample (F) is severe, leading to almost complete disappearance of dots up to 100 nm in diameter and nearly 50% increases in diameter.
and extrusion is seen at 900°C and below for dots larger than ~ 100 nm. Faceting is observed at 1000°C and higher, and is universally hexagonal and commensurate with the c-plane orientation of the substrate.

These results are all in agreement with preliminary observations made in the course of previous work.

**Interpretation of Results:**

The temperature threshold for disappearance at ~ 1000°C suggests that metal evaporation alone is not the mechanism responsible. The rate of metal evaporation at 900°C [4] would lead to removal of the entire volume of a 100 nm diameter dot in several minutes. Cross-sectional transmission electron microscopy of selected samples will be performed to determine at which point the dots have fully converted to oxide. Further experimentation with annealing temperatures and times and modeling based on these results is ongoing in order to conclusively determine which of the remaining mechanisms are responsible for dot disappearance. Quantitative study of dot spreading rates may also prove useful in separate studies of metal/oxide wetting and/or oxide surface diffusion.

**References:**

Optomechanical Experiments with Ultra Thin Mechanical Membranes

CNF Project Number: 762-99
Principal Investigators: Paul McEuen², Jeevak M. Parpia², Harold Craighead¹
Users: Vivekananda P. Adiga¹, Rob Barton¹, Isaac Storch², Rob Ilic³, Christopher Wallin¹

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Abstract and Introduction:
Manipulation and detection of the mechanical degree of freedom with light in connection with nanomechanical resonators is an emerging field with applications in the control of quantum states of macroscopic systems [1], sensing, and detection [2]. We demonstrated that a Fabry-Perot cavity between a flexural resonator and a reflecting mirror can be used to detect the motion of these resonators and a pulsed laser can be used to actuate the motion of the resonator (Figure 1) [3-6]. Detection of the resonant motion by optical means also offers advantages in terms of both optomechanical coupling experiments and sensing. In this regard there are advantages associated with using very large lightest possible membranes as the mechanical elements to achieve low spring constants and resonator mass [1, 2]. Two dimensional materials like graphene, boron nitrene and ultrathin silicon nitride membranes show promise of being used as mechanical membranes whose oscillations can be readily detected by optics [1-5].

However achieving high mechanical quality factors in these high surface-to-volume ratio resonators has been a challenge. Recently it has been observed that the mechanical quality factor of the resonators can be significantly improved by choosing appropriate resonator geometry, stress and improved fabrication techniques [2, 3, 5].

Summary of Research:
We have measured extremely high quality factors (up to 4,000,000 for 15 nm thin silicon nitride membranes [4] and up to 5,000 for graphene [6]) for large area (up to 1 mm for high stress nitride and up to 100 µm self-tensioned graphene drums, Figure 1) at room temperature. These findings pave the way for identifying optimum size and modes for achieving high mechanical Q oscillators for applications in mass sensing and next generation of optomechanical coupling experiments [1, 7].
Results and Conclusions:

Preliminary results on suspended graphene resonators show that it is possible to use lasers to cool or heat a 2D material like graphene in a simple low finesse Fabry-Perot cavity formed by a graphene membrane suspended over a prefabricated trench [7] or a movable metallic mirror. An electrically driven graphene membrane’s resonant motion can be amplified or suppressed by moving the membrane within the cavity (Figure 2), wherein the resonator can be moved from regions of higher absorption to lower absorption. A phothermal back action results in resonator motion being amplified or cooled depending on the direction of the force with respect to the membrane’s motion. The strong optomechanical coupling observed in these membranes is partly due to the low mass, spring constant of these structures. The implication is that this should significantly reduce the difficulty to cool these devices to their quantum ground state from some readily accessible starting temperature or to perform sensing with self-oscillating membranes at room temperature.

References:

Graphene Optomechanical Systems

CNF Project Number: 762-99
Principal Investigators: Harold G. Craighead, Jeevak M. Parpia, Paul L. McEuen
Users: Robert A. Barton, Isaac R. Storch, Vivek P. Adiga, Reyu Sakakibara, Benjamin R. Cipriany, B. Rob Ilic, Si Ping Wang, Peijie Ong

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Abstract:

By virtue of their low mass and stiffness, atomically thin mechanical resonators are attractive candidates for use in optomechanics. Here, we demonstrate photothermal back-action in a graphene mechanical resonator comprising one end of a Fabry-Perot cavity. As a demonstration of the utility of this effect, we show that a continuous wave laser can be used to cool a graphene vibrational mode or to power a graphene-based tunable-frequency oscillator. In addition to enabling studies of fundamental physics, the remarkable sensitivity of graphene optomechanical resonators and their ability to operate over a broad range of wavelengths and mechanical frequencies makes them attractive for technological applications.

Summary of Research:

Optomechanics [1] is a research field that uses the forces of light to manipulate mechanical objects. In many cases, it involves the use of feedback forces to alter the damping in a microelectromechanical systems or nanoelectromechanical systems (MEMS / NEMS) resonator. In much the same way as laser-cooled atoms are reduced to temperatures approaching absolute zero, the effective temperature of a resonator’s vibrational mode can be “cooled” by using optomechanical feedback to reduce its Brownian motion. This cooling can be so extreme that recent experiments have managed to laser-cool NEMS resonators to their quantum ground state, meaning that – to name one bizarre example – the resonator can be vibrating by two different amounts simultaneously [2].

Investigating the physics of MEMS and NEMS resonators in the quantum regime is expected to allow physicists to test the laws of motion on new size scales and help us to understand quantum mechanics [3].

To accomplish these goals, physicists are testing extremely small resonators, because resonators of smaller mass are more sensitive to the weak forces associated with light. The ultimate limit would be resonators with atomic thinness, but it is challenging to make a mechanical device one atom thick, especially when it must also couple to light. Surprisingly, mechanical resonators made from graphene [4] have the potential to overcome these challenges with excellent electrical and mechanical properties and a strong interaction with light over the entire visible range. We proposed and demonstrated a mechanism for optomechanical coupling to a graphene resonator. Our results open new avenues for research in optomechanics, photonics, and atomic physics.

The resonators used for this experiment are suspended single-layer graphene clamped on all sides to a silicon dioxide substrate with source, drain, and gate electrodes (Figure 1). The resonators were batch-fabricated from chemical-vapor-deposited (CVD) graphene following procedures developed in the CNF [5, 6]. To monitor mechanical resonance, a continuous wave (CW) laser impinges on the cavity and reflected laser light is modulated by an amount proportional to the amplitude of graphene motion. The reflected light is monitored by a fast photodiode connected to a network analyzer. Motion is actuated capacitively by applying a modulated voltage, $V_g$, between the graphene and the gate. The graphene device is placed inside of a vacuum chamber in which the pressure is less than 10^-6 Torr.

We showed that the damping in the resonator depends on incident laser wavelength and power, consistent with an optomechanical effect. Based on the magnitude and sign of the effect, we conclude that the important force providing the optomechanical coupling is the photothermal force arising from thermal expansion of the graphene membrane upon changes in laser-induced heating. When the damping from the
light field is negative, sufficiently high laser powers will cause regenerative self-oscillation in the graphene membrane – that is, it will vibrate strongly without any time-varying drive force. When the damping from the light field is positive, the laser can be used to cool the thermal motion of the membrane. At \( l = 718 \) nm, the area under a device’s Brownian motion peak decreases by about a factor of two when laser power increases from 1 mW to 2 mW (Figure 2a). We estimate based on the width of the driven peak (Figure 2b) that the effective temperature at \( P = 1 \) mW is 210 ± 60 K, and the temperature at \( P = 2 \) mW is 100 ± 40 K. Laser cooling of a graphene membrane could have applications in the aforementioned investigations of nanomechanical systems in the quantum regime.

References:
Resonant Properties of High-Stress Silicon Nitride Membranes

CNF Project Number: 762-99
Principal Investigators: Harold G. Craighead¹, Jeevak M. Parpia²
Users: Vivek P. Adiga¹, Roberto De Alba², Nikolay Zhelev²

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Abstract:

Micromechanical resonators have been studied for some time, with modern applications ranging from molecular-resolution gas sensors to micron-scale structures for the study of mesoscopic quantum physics. Of all possible resonant media, stressed silicon nitride (SiN) is among the most desirable due to anomalously large quality factors (reaching up to $10^7$), and relatively large device dimensions. We have fabricated circular SiN membranes with diameters of 20 µm-1 mm, and thicknesses of 15-75 nm, in order to study their intrinsic energy dissipation mechanisms as functions of resonator size, vibrational mode, and environmental temperature. These efforts have lead to a more complete understanding of these systems, and ultimately higher-quality devices.

Summary of Research:

Stoichiometric SiN films (with 1.2 Gpa tensile stress) were deposited on a silicon substrate with 600 nm of thermal silicon dioxide. Millions of well defined, 100 nm diameter release holes were then patterned on the SiN films using electron beam lithography; these holes served as entry points for hydrofluoric acid during etching of the silicon dioxide, and led to suspended SiN membranes of uniform thickness. This procedure was used to make devices of varying diameters and thicknesses. Membrane thicknesses were measured by peeling of the suspended SiN and imaging with atomic force microscopy. Such an image can be seen in Figure 1.

Membrane motion was actuated physically through mounting of the samples on a vibrating piezoelectric disk. Surface motion was detected optically through the reflection of an incident laser beam (visualized in Figure 2), as in previous experiments [1-3]; in this regard, the SiN membrane and silicon backplane acted as a Fabry-Pérot interferometer. Using this setup, we measured device dissipation as a function of membrane size, thickness, and vibrational mode. Observed quality factors were strongly dependent on mode shape for thicker membranes, whereas those of thinner devices were more size-dependent (shown in Figure 3). These results indicate that vibrating membranes can reach high qualities with the appropriate choices of dimensions, stress, and mode number; this applies equally well to graphene and other stable membrane materials.

Another area we have explored is the temperature dependence of membrane dissipation. Stressed SiN deviates strongly from the “universal behavior” of other amorphous materials, resulting in devices with room temperature quality factors that are orders of magnitude larger than expected values [3]. Other glasses are known to have a sharp decrease in internal friction (meaning enhanced quality factor) below a characteristic temperature [4]. We have attempted to observe such behavior in our stressed SiN membranes using a liquid helium flow cryostat; we have not yet detected such a change in the 300 K - 10 K range. Work is currently underway to extend these measurements to ~ 20 mK in a dilution refrigerator.
References:


Figure 2: Visualization of incident detection laser beam on finished device.

Figure 3: Dissipation (inverse quality factor) for 15 nm thick devices of various diameters. Each curve displays measurements for multiple vibrational modes. For larger devices, dissipation is shown to depend heavily on mode number. There is also a strong dependence on membrane diameter.
Silicon Nanowire Atomic Force Microscopy Probes for High Aspect Ratio Geometries

CNF Project Number: 804-99
Principal Investigator: Sandip Tiwari
Users: Brian A. Bryce, B. Rob Ilic, Mark C. Reuter

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Primary Source of Research Funding: Cornell Center for Nanoscale Systems
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Abstract:
We have developed silicon nanowire based atomic force microscopy probes combining vapor-liquid-solid growth in a custom UHV-CVD with a traditional MEMS processing backend performed at CNF.

Summary of Research:
Atomic force microscopy (AFM) is now an in-line tool for nanoscale process characterization. Standard probes work well for planar forms but fail with high aspect ratio structures. For this, one needs a probe of equal or higher aspect ratio. We have created wafer-scale silicon vapor-liquid-solid (VLS) nanowire based AFM probes using site controlled growth that are enormously effective for this challenging characterization. The process creates silicon very high aspect ratio nanowire AFM probes while requiring no lithographic features smaller than 1 µm. The maximum aspect ratio achievable is limited by the ratio of the bulk chemical vapor deposition and catalyzed VLS growth rates. Probes with aspect ratios as high as 90:1 (Figure 1f) have been created on <111> silicon-on-insulator wafers using Au as the growth catalyst. The Au tip offers a useful biological sensing platform for functionalization using thiol chemistry. The Au can also be removed chemically and the wire thinned sacrificially via oxidation to a desired final diameter. This diameter sets the probe’s lateral resolution, while the wire length determines the maximal vertical travel in the AFM. Control of the geometry of the wire via growth and chemical thinning also allows the stiffness of the probe to be tuned. Specifically, for a cylindrical wire, the Euler buckling force will scale as \( r^4 \). Taking Young’s modulus to be 185 GPa for a 8 µm long wire the bucking force ranges from 9 nN at \( r = 25 \) nm to 11 µN at \( r = 150 \) nm. We have successfully used both thinned and as grown silicon nanowire AFM probes for non-destructive topographic imaging of high aspect ratio features (Figure 1a, d).

Figure 1, opposite page: (a) The performance of the nanowire tip is compared to a conventional TESP tip, tilt-corrected FIB milled tip, and an SEM cross section of the same 1.95 µm deep, 900 nm wide trench. The symbols are plotted every 40th point for the nanowire and TESP AFM data and every 20th point for the SEM data and FIB milled AFM data. The nanowire tip significantly outperforms the TESP tip and agrees very well with the SEM data. The nanowire tip is comparable to the FIB milled AFM tip. The 90 degree image is scanned with the short axis of the cantilever orthogonal to the trench’s long axis and the 0 degree image is scanned with the long axis of the cantilever orthogonal to the trench’s long axis. The depth data are averaged along the trench’s long axis to produce the plotted traces. The encroachment seen at the left and right side of the 0 degree scan from the SEM data indicate the tip diameter at the time of imaging. The nanowire diameter at the time of imaging in (a) was 244 nm; in (c) the diameter was thinned to 84 nm. The commercial FIB milled tip had an 800 nm long chisel shaped tip terminated in a nominally 25 nm point attached to a nominally 5 µm long 200-500 nm wide shaft. This finer point allowed the FIB milled tip to probe channel on the negative X-axis but not the positive X-axis as the tip chisel shape is asymmetrical. On the positive X-axis the shaft diameter causes encroachment similar to the nanowire probe. The SEM data shown (a) is calculated from edge of the cross section shown in (b). (c) The probe as used during the scans shown in (a). It is possible to reach the bottom of a 2.05 µm deep, 300 nm wide trench by thinning the nanowire (d), although it does contain ringing artifacts that cannot be suppressed with the feedback settings on the Bruker Icon AFM used. The trenches being imaged in (d) are show in (e). The probe at the time of imaging (d) is shown in (f). The scale bars for (b), (c), (e), and (f) are 2 µm, 5 µm, 1 µm, and 5 µm respectively.
Figure 1
A Single Lithography Self-Aligned Vertical NanoRelay

CNF Project Number: 804-99  
Principal Investigator: Professor Sandip Tiwari\textsuperscript{1}  
Users: Joshua Rubin\textsuperscript{1}, Ravishankar Sundararaman\textsuperscript{2}, Moonkyung Kim\textsuperscript{1}  

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Abstract:

We demonstrate the use of torsion in nanorelays to achieve low voltages, high speeds, single lithography step construction, and a form useful for configurability and electronic design enhancements in three-dimensional integrated implementations. The combined bending and torsion of self-aligned nanopillars facilitates the first top-down fabricated vertical three terminal nanoscale relay. Experimental devices, even at 500 nm features, operate at \(~ 10 \) V and \( \mu \)s. Scaling suggests operation down to unit volts.

Introduction:

Nanorelays are potentially relevant devices for low standby power and energy exchanging computing \[1\] and for dynamic or non-volatile configurability. To date, nanorelay implementations have used processes that combine single crystal materials and layered structures, thus permitting ultra-small gaps needed for actuation. This allows electronics compatible voltages to be achieved at the expense of complex processes with high temperature deposition and selective removals.

Although recent planar structures based on CMOS-compatible top-down approaches have exhibited promising switching behavior \[2, 3\], they require either more real-estate or more aggressive scaling to achieve the high-aspect ratios necessary for low voltage operation. Vertical switches employing growth approaches based on nanotubes \[4\] have also been successfully demonstrated at low voltages with small device footprint. However, they require elaborate fabrication processes, use exotic materials and suffer from substantial variability in placement and size. We show here an appealing alternative, leveraging torsion, that has the potential to alleviate the need of single crystal deposition processes, and that can also be used in interposers for 3D integrated programmability. Using a single step of optical lithography, the device combines the benefits of a top down approach with the area savings of a vertical structure to achieve self-aligned nanopillars with aspect ratios up to 100:1.

Design:

Figure 1(a) shows vertical device which uses a combination of bending and torsion to physically connect S and D electrodes via a “channel.” With S/D grounded, \( C_{\text{SC}} \) and \( C_{\text{DC}} \) are in parallel with \( C_{\text{PC}} \), for a total equivalent capacitance \( C_{\text{PC}} \). Therefore, the gate to “channel” capacitance \( (C_{\text{GC}}) \) in series with \( C_{\text{PC}} \) form a voltage divider which determines the potential of \( C \) and the electrostatic force on the channel is proportional to \( V_\text{GC}^{-2} \). Electrostatic actuation of the “channel” results primarily in bending of the pillar until pull-in occurs.
Torsion allows self-correction for S/D offsets arising from line edge roughness or metal grain size. This is critical to achieving reproducible contacts in small geometries, an issue with conventional nanorelays.

Initial pull-in converts the device to a simple parallel plate configuration (Figure 1(d)) with new gap size determined by a predefined offset between a recessed gate and S/D, ~ 100 nm for current device. The recessed gate prevents gate–channel contact. This “new” gap is much smaller, thereby strongly enhancing torque on the channel. The device achieves an “ON” state when torsion overcomes the small S/D offset, establishing a low resistance metal to metal contact.

**Fabrication:**

The device is fabricated with a single step patterning that precisely places source, drain and gate perfectly aligned with the nanopillar. Devices employ a variety of tricks to decrease the critical dimensions (Figure 2). Stiction-free processing can be achieved using an isotropic SF$_6$/O$_2$ etch. A key feature of the fabrication is the thin Si$_3$N$_4$ insulating spacer. The Si$_3$N$_4$ enables low voltage operation by making possible independent optimization of gap and pillar sizes. Therefore, pillars can be reduced while maintaining small gap sizes.

Gaps were a conservative 200-350 nm for our devices. Starting with larger gaps enables deeper etching for taller pillars and reduces the likelihood of stiction during wet processing. Gap reduction is easily achievable by longer blanket evaporation of metal in the final process step. Electrical isolation is maintained between the body and the other electrodes by virtue of the silicon undercut beneath the Si$_3$N$_4$.

**Results:**

Operation of a typical device at 10V in air is shown in Figure 3. It has a gap of ~ 230 nm measured from the S/D, and non-uniform pillar thickness ~ 130 nm at the bottom and ~ 180 nm at the top. Figure 4 shows the measured device after pull-in is achieved.

**References:**


SQUID and XPS Analysis of Thin Nickel Films

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Abstract:
In previous reports, we described the fabrication of nickel nanomagnets on attonewton-sensitivity silicon cantilevers for use in magnetic resonance force microscopy (MRFM) experiments. Although we demonstrated a method for the successful high-yield fabrication of these tips by (1) separately batch-fabricating blank cantilevers and magnet-tipped silicon microchips, and (2) serially combining them using focused ion beam manipulation, the magnets were observed to magnetically saturate below the expected value of 0.6 T. In this report we detail our work to determine the root cause of this low magnetization by employing superconducting quantum interference device (SQUID) magnetometry and x-ray photoelectron spectroscopy (XPS) depth profiling.

Summary of Research:
Detecting single electron spins by MRFM requires the fabrication of attonewton-sensitivity cantilevers with attached nanomagnet tips. These nanomagnet-tipped cantilevers are brought to within a few nanometers of a sample surface to detect the spins in resonance in the sample film. Because the cantilevers have spring constants that are less than 1 mN/m, the long axis of the cantilever is aligned perpendicular to the sample surface.

Processes for fabricating these magnet-tipped cantilevers have been developed [1, 2], but the methods have been observed to produce magnets with a lower saturation magnetization than bulk nickel material when studied by frequency-shift cantilever magnetometry [1-3]. Obtaining a high signal-to-noise ratio in an MRFM experiment requires that the nanomagnet have a high tip field gradient; thus it is critical that the magnets have a high saturation magnetization and minimal damage layers, especially at their leading edge (the edge closest to the sample surface). Here we report analysis of the magnetization and elemental composition of thin nickel films to determine the damage mechanism of the nickel nanomagnets. Since few techniques have the sensitivity needed to characterize individual 100 nm × 100 nm × 1500 nm nickel nanomagnets, we studied large-area thin films using SQUID magnetometry and XPS depth profiling.

SQUID magnetometry was used to determine whether thin nickel films saturated at the expected value of 0.6 T when deposited using the same technique as for nanomagnet deposition in Ref. 1. Circular samples with diameters of 1.69 mm were patterned in a bilayer resist of LOR 10A and SPR 220-3 on a 0.5 mm thick fused silica wafer, and after development, they were oxygen-cleaned for one minute in an Oxford PlasmaLab 80+ RIE system. Two types of samples were evaporated using a CVC SC4500 e-gun evaporation system: (1) 82 nm of Ni with a 5 nm Ti adhesion layer; (2) 43 nm of Ni with a 5 nm Ti adhesion layer and a 10 nm Pt capping layer. Following lift-off, the wafer was diced into 4 mm × 4 mm squares using a KS 7100 dicing saw such that each chip contained one circular film of nickel. All samples were handled with plastic tweezers after dicing to avoid contamination with extra ferromagnetic material.

The samples were loaded into a Quantum Design MPMS-XL SQUID magnetometer using a plastic drinking straw attached to the loading rod. The SQUID was cooled to 4.0 K and allowed to equilibrate for about 30 minutes before the field was ramped between +1.5 T and -1.5 T to collect magnetization data as a function of external field. The background-subtracted data is displayed in Figure 1.

Magnetization was converted to saturation magnetization by accounting for the precise magnetic volume. The saturation magnetization was determined to be 0.61 T and 0.54 T for the TiNi and TiNiPt samples shown in Figure 1, respectively. Overall, the average saturation magnetization for four TiNi films was calculated to be 0.60 T, which agrees well with the
expected saturation magnetization for nickel and indicates minimal degradation of the film.

To assess the extent of surface oxidation, we also conducted XPS depth profiling measurements. Blanket-deposited films of Cr(5 nm)/Ni(80 nm) and Cr(5 nm)/Ni(80 nm)/Pt(10 nm) were evaporated in the same manner as for the SQUID samples. To collect the XPS depth profiles, (1) a chemical spectrum was recorded, (2) the surface was sputtered with argon at an accelerating voltage of 4 kV for approximately 30 seconds, (3) another spectrum was recorded, and (4) the process was repeated until the silicon substrate was reached.

After the data set was complete, the depth of the resultant crater was measured using profilometry to linearly convert the etch time to depth; this approximation yielded results that agreed with the expected thicknesses of the deposited layers. XPS data for both the uncapped and Pt-capped films are shown in Figure 2. For the uncapped film, oxygen content was observed within 20 nm of the nickel surface and was primarily concentrated in the first 5 nm. The platinum-capped sample was observed to successfully prevent the formation of any nickel oxide.

The SQUID magnetometry and XPS depth profiling results presented here indicate that our nanomagnets are damaged primarily by surface oxidation. Since the magnets that we employ in MRFM experiments have critical dimensions of 100 nm, we find that it is thus essential to develop new techniques to fully encase all exposed nickel surfaces.

References:
The Nanoaquarium: 
A Device for in situ Electron Microscopy of Processes in Liquids

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Abstract:
The nanoaquarium is a nanofluidic platform for in situ electron microscopy of processes in liquid media. The nanoaquarium consists of a hermetically sealed thin (~ 100 nm tall) liquid cell sandwiched between two electron-transparent silicon nitride membranes. The device has been used to image electrochemical deposition and etching, growth and dissolution of nanoparticles, and interfacial phenomena, and to assess various beam effects. This report will focus on the experiments pertaining to the aggregation of nanoparticles at interfaces.

Summary of Research:
The nanoaquarium is a microfabricated, nanofluidic device for in situ electron microscopy of processes taking place in liquids and of samples submerged in liquids. The device sandwiches a thin liquid layer, ranging in thickness from tens of nanometers to a few microns, between two thin, electron-transparent silicon nitride membranes. The liquid cell is hermetically sealed from the vacuum environment of the electron microscope. The thin liquid layer scatters few electrons and allows one to image objects suspended in the liquid with high resolution in a transmission electron microscope or scanning transmission electron microscope (TEM or STEM). The nanoaquarium also contains embedded electrodes for sensing and actuation. In situ (S)TEM with the nanoaquarium allows one to image dynamic processes in liquid media such as electrochemical deposition and etching, aggregation and assembly of colloidal crystals and metamaterials, growth and dissolution of nanocrystals and bubbles, interfacial phenomena, and biological interactions.

Fabrication of the device is accomplished using direct wafer bonding of silicon wafers coated with silicon nitride. One of the wafers also contains a thin film of patterned silicon oxide that defines the shape and height of the chamber and conduits. The direct wafer bonding approach reduces the possibility of contamination from glue, epoxy, and other sealing materials. Use of a dielectric material as the spacer allows electrodes to be directly integrated into the device. A schematic of the nanoaquarium is pictured in Figure 1 and a detailed description of the fabrication process has been previously reported [1]. Devices from the first generation of the nanoaquarium were used to study nanoparticle aggregation and colloidal crystal growth dynamics [2]. Some of the improvements in the latest version of the nanoaquarium include pillars in the window region to anchor the top and bottom membranes to each other to mitigate outward bowing of the membranes in the vacuum of the electron microscope chamber; changing the electrode material from gold to platinum to correct a problem with patchy electrodes after various heating steps in the fabrication process; and wafer bonding in the SB8e instead of manual hand bonding to improve bond uniformity, quality, and yield.

Figure 1: A schematic of the nanoaquarium’s cross-section.
Nanoscale investigation of the solid-liquid-vapor interface has remained a significant experimental challenge, but such studies are possible in the nanoaquarium [3]. The nanoaquarium was filled with an aqueous suspension of gold nanorods (20 nm in diameter, 40 nm in length) stabilized with surfactant cetrimonium bromide (CTAB). Imaging was performed in a 30 kV STEM (FEI Quanta 600 FEG Mark II). Electrical potential of ~ 15 V was applied across the embedded electrodes to generate a bubble that displaced liquid (and nanorods) to the perimeter of the observation chamber. We focused our observations at the interface between the bubble and the “bulk” liquid around the perimeter of the imaging window, which we refer to as the contact line. When the electron beam was focused onto the interface, the contact line moved (sometimes receding, sometimes advancing, and sometimes oscillating).

At a receding contact line, nanorods were propelled away from the “bulk” liquid (opposite from the direction of contact line movement). Interestingly, initially stationary particles did not move significantly until the contact line had passed by the particles by a distance of tens of nm. At an advancing contact line, nanorods were aligned and pushed into aggregates. Surprisingly, the initially stationary nanorods were not engulfed by the advancing contact line but were instead pushed ahead and formed a line that was parallel to the moving interface (Figure 2).

Movies of these phenomena are available at http://arxiv.org/abs/1110.3273. The experimental observations are consistent with a mathematical model that estimates the fluid velocity in the thin film by considering the surface tension force and disjoining pressure in the thin liquid film [4].

Acknowledgements:

Gold nanorods were provided by Dr. Christopher B. Murray and Mr. Xingchen Ye of the Chemistry department at the University of Pennsylvania. Dr. Frances M. Ross of the IBM TJ Watson Research Center provided encouragement and advice and collaborated on electrochemical experiments. Electron microscopy was performed at the Penn Regional Nanotechnology Facility at the University of Pennsylvania.

References:
Batch Compatible Integration of Nanoscale Samples and MEMS Devices

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Abstract:
We report on a micro tensile testing system that characterizes the mechanical behavior of nanometer scale specimens (~ 40 nm thick, ~ 200 nm wide, and ~ 7 µm long). The integrated sample/MEMS devices are made in batches with batch-compatible microfabrication techniques. Hence, the specimens and the associated MEMS devices are nominally identical. Nominally identical “nano sample-MEMS device” pairs will let us probe the statistical distribution of the mechanical properties at the nanometer scale, without the influence of random attachments of the nanowires to the MEMS device.

Summary of Research:
Current State of the Studies: Currently, we are characterizing the mechanical behavior of different types of metals at the nanometer scale regimen. Once the batch-compatible integrated “nano sample-MEMS device” pairs (please see Figure 1) are used to extract the mechanical behavior of the specimens, the specimens attached to the MEMS devices are removed from the environment, and the remaining MEMS devices are used for further mechanical characterization of externally integrated single crystal gold (please see Figure 2, and Figure 3), and single crystal gold-silver alloy specimens at the nanometer scale regimen. This design is the first in its field with such small samples (~ 40 nm thick, ~ 200 nm wide, and ~ 7 µm long) that are integrated with batch-compatible integrated specimens.

Recent Achievements: Recently, we were able to develop a technique that allows us to use digital image correlation (DIC) directly (without any image post processing) with SEM micrographs in order to extract the mechanical behavior (stress-strain diagram) of the tested nanoscale samples. Furthermore, using DIC, voltage-displacement behavior (please see Figure 4) of the “nano sample-MEMS device” pair are extracted. With the addition of recent achievements, the design became a complete setup that has the capability apply tensile forces on the test specimens in order to extract the mechanical behavior of the nanoscale specimens.

Acknowledgements:
National Science Foundation, Cornell NanoScale Science and Technology Facility (CNF) Staff, Center for Functional Nanomaterials (CFN) Staff.
Figure 1: Overall view of the “nanoscale sample-MEMS device” pair that is microfabricated with batch-compatible techniques.

Figure 2: Externally integrated single crystal gold specimen after the micro tensile test is completed.

Figure 3: The stress-strain diagram of the externally integrated single crystal gold specimen shown in Figure 2.

Figure 4: Voltage-displacement behavior of the “nano sample-MEMS device” pair. Experimental data is extracted from DIC on SEMs.
Non-Linear Dynamics of Coupled MEMS Oscillators

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Abstract:
An optically thin resonator suspended over a substrate, and illuminated with a laser, forms an interferometer, which couples deflection of the resonator to absorption within it. For high enough laser power, such resonators may self-oscillate due to feedback between deflection and absorptive heating. Bridges and cantilevers were fabricated, and the threshold power for self-oscillation measured as a function of beam geometry, and laser placement. Stiffness non-linearities lead to wide frequency tunability in bridges. Self-oscillating devices are inertially driven and regions of primary, sub- and superharmonic entrainment measured. Frequency tunability facilitates high order entrainment, large regions of entrainment, and frequency noise.

Summary of Research:
Applications of resonant MEMS in filtering and mass sensing require highly stable sharply peaked resonant curves. Our work focuses on achieving periodic motion through thermo-optic feedback. Bridges (doubly-supported beams) and cantilevers (singly-supported beams) are fabricated out of single crystal silicon using a silicon-on-insulator (SOI) process. Released beams are nominally 2 µm wide, 200 nm thick, and 7-40 µm long, with a 400 nm gap-to-substrate below. Devices are mounted on a piezoelectric driver in a high vacuum system and a continuous wave (CW) laser is focused to a 6 µm diameter spot on the resonator using a 50x objective lens. Device motion is inferred by measuring the modulation of the reflected laser in a high speed photodiode.

Transition to Limit Cycle Oscillation:
To observe the transition to limit cycle oscillations (LCOs), the piezoelectric driver is turned off, and the laser power on sample (P) increased until the onset of large amplitude self-oscillations at \( P = P_{\text{Hopf}} \). This critical power is measured as a function of beam geometry and location of illumination. Optical profilometry indicates that bridges 15 µm long and longer are buckled due to compressive pre-stress.

Stress-gradients and surface effects cause initial out-of-plane deflection in cantilevers too. As a result, the gap-to-substrate varies along the length of a beam, altering the properties of the interferometer, and the measured values of \( P_{\text{Hopf}} \). Note from Figure 1 that the amplitude of oscillation continues to increase with laser power for \( P > P_{\text{Hopf}} \).

Frequency Tuning:
Membrane stresses in bridges are a source of non-linearity which allow for tuning of the small amplitude frequency of oscillation, and give rise to an amplitude-frequency relationship. The frequency of oscillation of our buckled bridges increases with compressive stress due to further arching, and decreases with amplitude due to amplitude-softening. Increasing the laser power on sample increases compressive (thermal) stress in our bridges and increases the amplitude of oscillation.

For \( P < P_{\text{Hopf}} \) the frequency of oscillation increases with power due to the former effect. However, for \( P > P_{\text{Hopf}} \) the frequency decreases with power due to latter. In our bridges, amplitude softening dominates leading to frequency detuning as high as 25% (see Figure 2). Considerable frequency noise (\( \Delta f/f \approx 4 \times 10^{-5} \)) is observed in bridges due to instability in the laser power (\( \Delta P/P \approx 2 \times 10^{-5} \)). Less than a 0.03% change in the LCO frequency with laser power is seen in the cantilevers which do not support membrane stresses and are thus highly linear. As a result they are less affected by instability in the laser power and giving measured frequency noise as low as \( \Delta f/f \approx 3 \times 10^{-5} \).

Entrainment:
To study entrainment, the laser power is increased beyond \( P_{\text{Hopf}} \) and the self-oscillating devices are inertially driven. A function generator is used to create a swept sine wave which drives the piezoelectric element on which the chips are mounted. Thus the device has a preferred frequency, \( f_{LCO} \), and is forced at a separate drive frequency, \( f_D \), with a variable drive amplitude, \( A_D \). The spectral content of the device motion is observed on the spectrum analyzer, a frequency counter is
used to accurately track the forcing frequency, \( f_D \), and the forcing amplitude, \( A_D \), is measured on an oscilloscope. For hard forcing (\( A_D >> 1 \)) close to the limit cycle frequency (\( f_D \approx f_{LCO} \)) the limit cycle may be entrained to respond at the drive frequency, \( f_D \). When entrained, a single stable peak is seen on the spectrum analyzer at \( f_D = f_{LCO} \). For low forcing amplitudes at frequencies well separated from the limit cycle frequency, entrainment is not possible. A noisy peak is seen on the spectrum analyzer at \( f_{LCO} \neq f_D \) and a second steady peak is usually seen at \( f_{D} \), though it may disappear below the noise floor.

Primary entrainment is observed for a narrow band of frequencies (\( \Delta f / f_o < 0.3\% \)) in cantilevers, and over a large range of frequencies (\( \Delta f / f_o < 15\% \)) for bridges. Sub- and superharmonic entrainment is also demonstrated, where \( f_D \) is near a whole multiple or fraction of \( f_{LCO} \) – see Figure 3 for an illustration of 1:3 superharmonic entrainment where the limit cycle goes through three periods in the time the drive goes through one period. Sub- and superharmonic entrainment are observed for the bridges only. We see superharmonic entrainment at integer ratios up to 1:7 (drive:response frequency) and sub-harmonic entrainment at integer ratios up to 3:1. The width of the entrainment regions is dependent on the laser power and the amplitude of the inertial drive.
MEMS-Based Piezoelectric Vibrational Energy Harvesters

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Abstract:
Fixed-free cantilever microelectromechanical systems (MEMS) vibrational energy harvester structures have been fabricated and tested. These structures consist of a molybdenum-aluminum nitride-molybdenum (metal-piezoelectric-metal) stack with a proof mass at the free end of the cantilever. Based on the material and mechanical properties, the cantilever structure will resonate at a particular frequency. As the cantilever resonates, it creates a strain on the piezoelectric material within the structure and generates a charge. These devices are fabricated using standard micro-fabrication techniques. Summary structure design, theory of operation, process flow and final device characterization is presented.

Summary of Research:
Motivation. Energy harvesting devices are targeted for use in a variety of applications where they will, along with thin film batteries or supercapacitors replace typical batteries (such as “coin cell”) in systems. This is motivated by both environmental impact of disposal of batteries and the labor and cost associated with the battery replacement and disposal. The energy harvester is used to replenish the charge in the battery as the battery charge is depleted in use. Although other forms of energy harvesting exist such as solar, thermal, wind and electromagnetic, these devices tend to be large and bulky and not cost efficient. A MEMS process based energy harvester, once in production, will benefit from the economy of scale of standard micro-fabrication techniques where each silicon wafer will have several thousand energy harvesters and therefore be less costly that traditional batteries.

Structure and Theory of Operation. Figure 1 shows an illustrated view of the piezoelectric vibrational energy harvester (PZEH) structure. The structure shown is a “monomorph” — that is, a single layer of piezoelectric material between two metal electrodes. The structure is a cantilever (fixed on one end and free on both sides and the opposite end) with the free end also having a “proof mass.” Overall device size including the outer supporting frame is about 1 cm x 1 cm. Typically the cantilever is designed to resonate at a specific frequency and when it does, the free end will flex upward and downward (see Figure 2c). When this flexing motion happens, it creates a strain on the piezoelectric material which creates an equal and opposite charge on the top and bottom electrodes. This charge reads out as a voltage differential between the top and bottom electrodes. The output is a sinusoidal signal due to the upward and downward motion causing the charges on the electrodes to change signs. Excitation of the MEMS resonant...
A vibrational energy harvester for this particular design could be vibration of industrial motors and other equipment that runs on 60 Hz, 120V alternating current (AC) source. The cantilever design can be optimized to other target frequencies by varying the overall length of the cantilever, the change in size and placement of the proof mass and overall stiffness of the cantilever structure in general.

Summary Process Flow. The overall process flow can be lengthy to describe and cannot be sufficiently covered in this short report. An overall summary is shown in Figure 3. All processes are standard microfabrication techniques and are completely complementary metal-oxide-semiconductor (CMOS) compatible. This is a very important feature of the process flow and allows the PZEH technology to be transferred to a commercial fab with minimal process changes. A substantially more in-depth and complete report on the process flow, theory of operation and performance analysis can be found in other published documents [1, 2].

Of particular note, a dry deep reactive ion etch (DRIE) was used, instead of a more traditional wet potassium oxide (KOH) etch, that yielded controlled smooth edges, and also was used in die segmentation, which minimized die handling and potential yield loss in the final segmentation (dicing) step (typically performed with a mechanical dicing saw).

Performance. Singulated (individual PZEHs separated from the silicon wafer) packaged PZEH die in “quad” architecture (Figure 2a) were mounted to a test mount on a shaker. The shaker is interfaced to computer controlled electronics and vibrates (shakes) at a precisely controlled frequencies. In this report, testing was performed in open air (no vacuum). If tested in a vacuum, at similar g-forces, cantilever deflection, and therefore power output will be greater due to the minimization of squeeze film damping. The output of the PZEH quad chip is measured at the two output terminals (upper and lower electrodes as shown in Figure 3, lower right, final step) as an AC voltage. Coupled output power is optimized when the impedance of the load place across the PZEH electrodes matches the impedance of the electrically active PZEH structure. Figure 4 shows graphically the output power as a function of load impedance and G-force excitation.

For this design, maximum power output is at 85,000-ohm impedance. It should be noted that actual open circuit voltage output (no impedance load attached to the PZEH electrodes) of the PZEH is approximately twice the loaded circuit voltage as shown in Figure 4.

References:
Fabrication and Characterization of All-Thin-Film Multiferroic Cantilevers for Magnetic Energy Harvesting

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Abstract:

The electromagnetic energy harvesting properties of all-thin-film magnetoelectric (ME) heterostructures are measured. The devices are built on a silicon oxide/nitride/oxide (ONO) stack, and the ME layers consist of a magnetostrictive Fe$_{0.7}$Ga$_{0.3}$ thin film and a Pb(Zr$_{0.52}$Ti$_{0.48}$)O$_3$ piezoelectric thin film. The $Q$-factor of the mechanical resonance of the cantilevers is $\approx 2000$ in vacuum. The device resonant frequency displays a pronounced DC bias magnetic field dependence. The harvested peak power at 1 Oe is 0.7 mW/cm$^3$ (RMS) at the resonant frequency (3.8 kHz) with a load impedance of 12.5 kΩ.

Summary of Research:

In the emerging hybrid field of spintronics and straintronics, the multiferroic materials have attracted significant interest due to coexistence of ferromagnetism and ferroelectricity as well as their coupling. In laminated heterostructure systems, strong magnetoelectric (ME) coupling was observed due to strain-mediated interaction across the interface between magnetostrictive and piezoelectric phases. Such multiferroic heterostructures were used in bulk (centimeter-sized) devices for low magnetic noise measurements at room temperature and magnetic energy harvesting. Due to their low quality factor, $Q$, and low ME coefficient, $\alpha_{ME}$, as well their large size with unsuitability for integration with microfabricated peripheral circuits, the multiferroic bulk devices need to be improved by miniaturizing them on microelectromechanical systems (MEMS) platforms or magnetomechanical microsystems.

We have developed multiferroic sensors that consist of free-standing Pb(Zr,Ti)O$_3$ (PZT) magnetostrictive cantilevers (Figure 1). The thin-film heterostructure is on a Si substrate with a plasma-enhanced chemical vapor deposited (PECVD) oxide/nitride/oxide (ONO) stack. A 20 nm/100 nm Ti/Pt layer is sputtered at 430°C to form the bottom electrode of the piezoelectric part of devices. A ~ 500 nm PZT (e.g., Pb(Zr$_{0.52}$Ti$_{0.48}$)O$_3$) layer is spun by a sol-gel process. The PZT layer is covered by another 35 nm Pt buffer layer by sputtering at 305°C. A 500 nm Fe$_{0.7}$Ga$_{0.3}$ layer is then sputtered at room temperature.

Figure 1: A SEM micrograph of multiferroic harvesters. The devices are 950 nm long and 200 nm wide released cantilevers. The contact pads of the device are located at the base of the cantilever. Inset: Detailed SEM micrograph of the device heterostructure.

Figure 2: Energy harvested RMS voltage at bias field $H_{DC} = 66.1$ Oe and corresponding resonant frequency of 3833.1 Hz. Also, one displays the raw power measured as a function of loading impedance at resonance. The harvested AC magnetic field is $H_{AC} = 1$ Oe RMS.
temperature. The Pt buffer layer between PZT and Fe$_{0.7}$Ga$_{0.3}$ films has the role to improve the adhesion of the films.

A very important issue in making the cantilever-based sensors is the film-stress engineering of the heterostructure. The design allows a strategically vertical shift of the silicon ONO stack in the cantilever by modifying the thickness of the first and last layers of the silicon oxide. This will also alternate the stress distributions in the stack ensuring a final planar structure of the beam. The first silicon oxide layer grown on the silicon substrate has a thickness of 400 nm. Then, a 75 nm low stress tensile-type silicon nitride and a 100 nm silicon oxide are grown and so on up to six similar layers of silicon nitride. This structure with individual thinner silicon nitride layers instead of one single thick layer helps avoiding film cracks during the subsequent rapid thermal annealing (RTA) processes. The last oxide layer is deposited to complete a 3.8 µm thick ONO stack (Figure 1).

Figure 2 shows the experimental results for energy harvesting from a single multiferroic device. The AC magnetic field was maintained at 1 Oe RMS. The power output has a peak that occurs for a load impedance of 12.5 kΩ. To determine the power density of the multiferroic harvester, an effective volume (950 µm × 200 µm × 0.5 µm) must be taken into account that involves the freestanding length of the cantilever and the thickness of the poled (PZT) piezo-film. In this respect, the measured peak power density is 0.71 mW/cm$^3$. This value is close to other reported harvested power densities at 1 Oe RMS for bulk devices.

We have found that the resonant frequency of the present ME devices exhibits strong dependency on the magnitude and the sweeping direction of the DC bias field (H) (Figure 3). This fact indicates that the resonant frequency of the first-order flexural mode of the devices is determined not only by the mechanical properties of the devices, but also by the magnetic properties. The discontinuity in the DC field dependence of the resonant frequency occurs at 77 Oe (Figure 4). This value agrees with the coercive field of the 500 nm thick Fe$_{0.7}$Ga$_{0.3}$ films measured by VSM (vibrating sample magnetometry). A similar behavior was previously observed both in NEMS (nano-electromechanical systems) based devices and in larger clamped beams.

An interesting consequence of the field dependent $f_R$ is that as seen in Figure 3 the device can be operated at zero DC magnetic bias field with a relatively high ME coefficient as long as the AC field frequency is at the $f_R$ (H = 0). Removing the necessity to apply DC bias significantly simplifies the operation setup of these devices for both magnetic field sensing and energy harvesting schemes.

References:


Squeeze Film Damping in Microsystems

CNF Project Number: 2046-11  
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Abstract:
Squeezed film damping (SFD) is a common phenomenon that occurs in many microdevices when a surface moves normally to another solid surface in close proximity. In microelectromechanical systems (MEMS), SFD is often one of the largest sources of parasitic losses. Therefore, an accurate evaluation of hydrodynamic forces due to the SFD effect is critical in the design and optimization of the MEMS. In this work, experiments are implemented to investigate SFD effects on the dynamic response of a micro-plate compared to theoretical solutions. The test specimen is fabricated using standard microfabrication techniques.

Summary of Research:
In the recent past, a few studies have attempted to quantify the damping coefficient associated with beams and plates oscillating in a fluid environment, however, most of them show discrepancies from classical hydrodynamic lubrication theory in the relationship between damping coefficient and gap height [1-5]. The objective of this work is to identify the reasons for these discrepancies. To achieve this goal, SFD effects were first theoretically investigated employing a dynamic system consisting of a micro-plate separated from a parallel substrate by a thin fluid layer and undergoing normal oscillations. A compact expression for hydrodynamic forces was developed by employing the classical perturbation theory for small Reynolds number [6]. To validate the theoretical results, experiments were designed to investigate the same dynamic system.

The schematic of experimental setup is shown in Figure 1. The micro-plate is clamped together with spacers on a polycarbonate substrate. The gap between the plate and substrate can be varied by adjusting the number of thin glass spacers. A piezo stack, which provides the vibration motion, is glued on the bottom surface of the substrate. The piezo stack is actuated by a sinusoidal voltage signal generated by a lock-in amplifier. The lock-in amplifier serves as a function generator here and the signal is then amplified by a linear piezo amplifier which is connected with the piezo stack. The entire set up is mounted on an optical table. The motion of the plate is measured by a laser Doppler vibrometer. Usually, for a fixed input voltage, the measurement starts with a small frequency (e.g., 200 Hz) which will then be increased gradually until the resonance is found and amplitudes in each step is recorded.

The vibrating micro-plates were fabricated via a single mask lithography process that starts with a <100> double-side polished silicon wafer with thickness 400 µm; 2 µm of silicon dioxide was thermally grown on both sides of the wafer. After photolithography, the pattern was then transferred to silicon dioxide layer by reactive-ion etching (RIE). The Si wafer was etched deep reactive-ion etching (DRIE) and back side silicon dioxide acted as an etch stop. Finally, a buffered oxide etching (BOE) 6:1 etching is performed to release the plate structure by etching the silicon dioxide layers. The thickness of the silicon wafer determines the thickness of the vibrating plate and beams. The plate size is chosen to be 1000 µm × 1000 µm square and the width of the beams is 1/20 ~ 1/5 of the plate’s width.

Figure 1: Schematic experimental set up.
The length of the beams can be varying from two to five times longer than plate’s length. Several dozen vibrating plates were fabricated per wafer and each wafer was diced into chips of $25 \times 20 \text{mm}^2$ with one plate per chip. Figure 2 gives the SEM image of one of the chips.

The preliminary results of the experiments tested in air are presented in Figure 3. It is shown that by reducing the gap from 50 $\mu$m to 30 $\mu$m, the resonant frequency shifts to the left and amplitude reduces. It is because both viscous damping and fluid inertia have inverse relationships with the gap; therefore reducing the gap will increase both the inertia and viscous forces. While viscous damping effects reduce the magnitude of the system, fluid inertia acts as an added mass, shifting the natural frequency of the system to a lower range.

For the future, we will continue the experimental measurement and test structure with different areas, beam widths in various media.

References:

Spraycoat Lithography onto Silicon Carbide-Aluminum Nitride Wafers

CNF Project Number: 2058-11
Principal Investigator and User: Richard Mlcak

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Primary Source of Research Funding: National Science Foundation
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Web Site: www.bostonmicrosystems.com

Abstract:
Boston MicroSystems, Inc., used CNF to develop a photoresist spray-coating process used to pattern electrode and packaging metal layers for fabricating piezoelectric microelectromechanical resonators built from silicon carbide substrates and aluminum nitride piezoelectric films.

Summary of Research:
The spray-coated resist was exposed using CNF’s Karl Suss MA6 aligner and developed, to prepare the wafers for metal deposition and liftoff. Key process parameters were studied and optimized, including resist dilution, resist flow rate, number of passes, exposure time, softbake temperature, softbake time and develop time. The spray-coat process parameters and patterned liftoff resist were qualified by subsequent metal deposition and liftoff (both performed outside of CNF), which was successful and resulted in properly functioning devices and high manufacturing yield.
Microscale Electrostatic Energy Harvester

CNF Project Number: 2074-11
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Primary Source of Research Funding: National Science Foundation
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Abstract:
Electrostatic power generators employ micro-fabricated variable gap capacitors to harvest mechanical vibration energy and have applications in wireless sensors networks. Prototype designs with in-plane gap-closing topology were fabricated with silicon-on-insulator (SOI) technology. Mechanical tests show the devices resonate at approximately 100 Hz, as designed, when a two gram mass is attached to the shuttle electrode.

Summary of Research:
MEMS electrostatic energy converters use the relative motion between two independent electrodes of a variable capacitor, to convert kinetic energy into electrostatic energy. Energy is gained from the MEMS variable capacitor as its capacitance fluctuates between a maximum and a minimum in either voltage-constrained or charge-constrained power conversion cycles [1].

There are many possible energy sources for micro-power harvesters, nevertheless low frequency ambient vibrations are of particular interest due to their ubiquitous nature. To optimize MEMS harvesters and produce an increased power output, the devices must be designed to resonate at the available ambient frequencies. However, resonant frequencies of MEMS devices are typically much higher than ambient vibration levels due to their negligible mass.

One method to overcome this challenge is to add a high density mass to the moving electrode. Stiffness of the beam supporting the mass has to be carefully considered to ensure stability at resonance.

MEMS energy converters prototypes with gap-closing topology were designed and fabricated on SOI wafers with p-type doped, 200 µm thick device layers (Figures 1 and 2). A two gram mass of steel was attached to the shuttle electrode to add mass to the system. The device was the tested using an electromagnetic shaker operating at 100 Hz with 0.25g, 0.5g, 0.75g and 1g of acceleration. The minimum gap between the electrodes observed at 1g has been approximately 2 µm and the response was stable. Electrical testing of the device integrated with a power control circuit is in progress.

References:
Figure 1: MEMS electrostatic energy converter with 2g mass of steel mass attached to the free electrode.

Figure 2: Optical microscope image of a part of the device showing the comb-like electrodes and the suspension beam (top view).
Large Bandwidth Continuously Tunable Delay Using Silicon Microring Resonators

CNF Project Number: 980-01
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Users: Jaime Cardenas\textsuperscript{1}, Kevin Luke\textsuperscript{1}, Lian Wee Luo\textsuperscript{1}, Carl B. Poitras\textsuperscript{1}

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Primary Source of Research Funding: Morton Photonics under DARPA SBIR contract # W31P4Q-09-C-0298
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Abstract:
We demonstrate etched facet silicon inverse tapers coupling loss as low as 0.7dB per facet. This taper can be fabricated on a wafer scale enabling mass production of silicon photonic devices with broadband, high efficiency couplers.

Summary of Research:
Many different approaches for coupling light from an optical fiber to a silicon waveguide have been proposed and demonstrated over the past decade. Most of these use either a taper structure for end-fire coupling or a grating for normal or quasi-normal incidence coupling \cite{1-6}. Inverse taper waveguides are inherently broadband, however the end facet is typically polished one chip at a time to achieve high coupling efficiency. On the other hand, grating couplers are inherently bandwidth-limited, more complex for high efficiency coupling (though not as efficient as inverse tapers), but can be fabricated on a wafer scale without the need for one chip at a time processing. Here we demonstrate an inverse taper waveguide fabricated with an etched facet that enables high efficiency coupling and can be fabricated on a wafer scale without the need for cleaving or polishing, therefore enabling high volume manufacturing of silicon photonic devices.

The challenge in fabricating deep etched facets for photonics is the high loss originating from the imperfect etching of the two different materials (Si and SiO\textsubscript{2} with different etch rates) \cite{6, 7}. Here instead the etched facet silicon inverse taper waveguide consists of a silicon nanotaper terminated with an oxide gap such that we only etch one material to form the facet. Etching only one material enables high efficiency coupling with a smooth and vertical sidewall since the silicon tip of the nanotaper is buried in the oxide and isn’t damaged during the etching processes. So long as the oxide gap is small, its presence has a very weak effect on the mode matching between the tapered fiber and the inverse taper waveguide.

To measure the coupling efficiency of the etched facet nanotapers with oxide gap, we fabricate 250 nm by 500 nm waveguides of 0.69 mm, 2.78 mm, and 4.37 mm with

Figure 1: Schematic of etched taper facet with oxide gap.

Figure 2: Top view SEM of etched facet inverse taper waveguide with a 2 µm oxide gap.
Optics & Opto-Electronics

160 nm tapers. The waveguides are patterned with e-beam lithography, etched in a chlorine chemistry and clad with 2.4 µm of PECVD silicon oxide. Then, openings are patterned using contact photolithography to define the inverse taper waveguide facets and the silicon dioxide (both the PECVD overcladding and the buried oxide) is etched in an ICP RIE using a CHF$_3$/O$_2$ chemistry. Finally the silicon substrate is deep etched using the Bosch process to give access to the chip facet (see SEM of taper on Figure 2). The gap between the end of the nanotaper and the edge of the facet is varied from 2 µm to 6 µm to account for the misalignment of the contact aligner used to define the inverse taper waveguide facets. We measure a coupling loss as low as 0.7 dB per facet for a taper tip width of 160 nm. The coupling loss is measured using the “cut-back” by first measuring the throughput of the waveguides of different lengths and then fitting the results to a straight line. We measured the throughput of the tapered fibers back-to-back (with no silicon die present) and determined that their loss was 1.6 dB. To calculate the coupling loss, we subtract the tapered fiber losses from the insertion loss measured via the “cut-back” method. For a 2 µm oxide gap, we measured an insertion loss of only 3.1 dB with a low coupling loss of 0.7 dB per facet. All devices with oxide gaps between 2 and 6 µm had a coupling efficiency within 0.5 dB.

Acknowledgements:
We thank Morton Photonics for supporting this work under DARPA SBIR contract # W31P4Q-09-C-0298, “Highly-Integrable Microresonators with Fast Tunable Group Delay for Broadband RF True Time Delay.” This work was performed 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-0335765).

References:
Integrated Graded-Index Luneburg Lens for Robust Fiber-to-Chip Coupling

CNF Project Number: 980-01
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Abstract:
We demonstrate a high contrast, low loss, graded-index lens for robust fiber-to-chip coupling of silicon waveguides. We experimentally show increased alignment tolerance in comparison to a conventional inverse taper.

Summary of Research:
Graded-index (GRIN) photonics has received a lot of attention in recent years [1, 2]. The ability to control the flow of light brought about by advances in transformation optics, metamaterials, and fabrication techniques is changing the way we design optical devices, enabling features once unimaginable. Nonetheless, although many such devices have already been demonstrated, most are limited by scattering losses resulting from the discrete structures that compose their effective graded material. In this work, we employ a low loss platform for high contrast GRIN devices to fabricate a compact Luneburg lens [3, 4] with a 30 µm diameter that is integrated with conventional silicon waveguides to improve their alignment tolerance in fiber-to-waveguide coupling.

Similarly to a conventional lens, the Luneburg focuses light at a position determined by the angle of incidence of the beam. On the other hand, this spherical GRIN lens does not suffer from aberration or coma, which on a conventional lens change the focusing spot depending on the distance of the light beam to the optical axis. Additionally, due to its index profile $n(\rho) = n_0 \left(2 - \rho^2\right)$, where $\rho$ is the normalized radius, the index at the edge of the lens $n(1) = n_0$ can be designed to match the surrounding environment, rendering this lens reflectionless over a wide wavelength range.

Combined, these properties make the Luneburg lens a great candidate for coupling large delocalized fiber modes into highly confined silicon waveguides with tolerance for the exact incoming position of the fiber beam, a fundamental aspect for future optical networks. As illustrated in Figure 1A, the integrated Luneburg lens, present in the same substrate of our waveguides and inverse tapers [5], effectively plays the role of gathering the signal coming from the fiber and delivering it to the taper even if the former is slightly misaligned to the latter. We thus expect increased coupling robustness along the chip edge direction, while, as our results show, introducing no changes in the out-of-plane direction.

To produce the required profile for the Luneburg lens (with index variation of 41% from edge to center) we used a tapered slab waveguide [6, 7] in a compact size (15 µm radius), suitable for integration. On an SOI wafer with 250 nm device layer the waveguides and tapers were defined by electron beam lithography and anisotropic plasma etching. Then the Luneburg lenses were patterned via focused ion beam milling. Finally, the samples were cladded with 2 µm of SiO₂. An optical microscope image of the cladded device is shown in Figure 1A, as well as an atomic force microscope (AFM) scan of the silicon slab height compared with the designed lens profile (Figure 1B), which matches quite well the fabricated device.

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Figure 1: Luneburg lens coupler. (A) Schematic of operation of the Luneburg lens coupler and optical microscope image of the fabricated device. (B) Scanned silicon slab height (dots) compared to the designed profile (solid line).
We measured the coupling misalignment loss for two Luneburg couplers and two conventional inverse tapers. The results, seen in Figure 2A, show the normalized collected power when the input fiber is scanned across the devices via a piezoelectric stage in both horizontal (along the edge) and vertical (out-of-plane) directions. We can observe that the Luneburg couplers improve the overall alignment robustness of the system in the horizontal direction as expected. In Figure 2B cross-sections of the maps are shown for the four devices along the horizontal direction. We can see, for example, 6 dBm less penalty for the Luneburg couplers than for the tapers at 4 µm misalignment. Additionally, a similar analysis shows that the presence of the lens does not hinder the sensitivity in the vertical direction. Simulations performed with COMSOL Multiphysics closely agree with the experimental results.

GRIN photonics holds the promise of new capabilities for future and existing optical systems. This work demonstrates the use of one of these devices, a compact aberration-free Luneburg lens, to improve the alignment sensitivity of fiber-to-waveguide coupling for future optical networks.

References:

Figure 2: Experimental demonstration of improved misalignment robustness by the Luneburg coupler. (A) Maps of normalized power collected by scanning the input fiber on each device. (B) Cross-sections of the power maps along the horizontal direction and simulation results for the horizontal sensitivity (solid and dashed lines for the Luneburg and inverse taper only couplers, respectively).
High Quality Factor and High Confinement Silicon Resonators using an Etchless Process

CNF Project Number: 980-01
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User: Austin G. Griffith

Affiliation: Department of Electrical and Computer Engineering, Cornell University
Primary Source of Research Funding: U.S. Air Force AFSOR program FA9550-09-1-0704 on “Robust and Complex on-chip Nanophotonics,” National Science Foundation through CIAN ERC under Grant EEC-0812072
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Abstract:
We demonstrate high-Q factor and high confinement silicon ring resonators fabricated by a local oxidation of silicon (LOCOS) process. We achieve an intrinsic quality factor of 525,000 in a 410 µm-circumference ring resonator.

Summary of Research:
Thermal oxidation has shown great promise in reducing silicon waveguide losses. Optical losses from both surface roughness and absorption sites at the waveguide boundaries have been decreased by smoothing the waveguide with thermal oxidation [1]. This has spurred interest in ‘etchless’ fabrication processes, where thermal oxidation has been used to define the structure of the waveguide [2-4]. High confinement etchless waveguides have been demonstrated successfully [3]. Etchless resonators have been demonstrated with high quality (Q) factors, but with delocalized optical modes [2, 5]. There is much interest in achieving high confinement resonators — both to limit cross talk in photonic systems, and to enhance nonlinear interactions. The challenge in demonstrating high confining resonators lies in the difficulty of controlling the exact dimensions of the structure and achieving small coupling gaps.

Here we demonstrate the first high confinement etchless silicon ring resonator with high intrinsic Q factor of 525,000 at 1549.6 nm, and a propagation loss of $\alpha = 1.3$ dB/cm by controlling the etch rate of the oxidation mask. The effective index of the TE mode at 1500 nm is approximately $n_{\text{eff}} = 3.1$ from computer simulation. The device required a 48 µm long coupling region to achieve coupling — experimentally confirming the high confinement of the optical mode.

We fabricated the resonator with long 48 µm coupling regions, with a gap of 400 nm. The patterned silicon nitride mask was 1 µm wide. The device was fabricated on silicon-on-insulator (SOI) wafer, with a top silicon layer of 500 nm, and a buried oxide of 3 µm. Coupling is achieved through nanotapers [6].

We used low-pressure chemical vapor deposition (LPCVD) to deposit 200 nm of silicon nitride, which acted as the mask layer. The silicon nitride was then coated with ma-N 2405 resist, and patterned with electron beam lithography. We etched the silicon nitride using reactive ion etching (RIE) with CF$_4$ gas. The resist was stripped, and then we used dry and wet thermal oxidation to oxidize the silicon. The silicon nitride was left in place. The device was clad with 2 µm of plasma enhanced chemical vapor deposition (PECVD).

The resonance peak measured at 1549.6 nm is shown in Figure 2. At the resonant wavelength, the ring is under-coupled, with transmission fraction $T_o = 0.16$. We calculated the unloaded Q to be 525,000. The propagation loss is extracted from the measured Q and the FSR of the structure:

$$\alpha = 2 \pi \lambda / (Q_u \times L \times \text{FSR})$$

where L is the length of the resonator, and FSR is the free spectral range of the resonator [7]. From this equation, we calculated $\alpha = 1.3$ dB/cm for this structure.

We have demonstrated that small coupling regions between etchless waveguides can be successfully fabricated, while keeping overall losses low. Because of this, we were able to demonstrate high confinement and high intrinsic Q factor etchless resonators.
References:


Fabrication of Nanofluidic Channels on Fused Silica Wafers

CNF Project Number: 1096-02
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Abstract:
Current project has been dedicated to fabricating 500 nm wide and 400 nm deep channels on fused silica wafers. The channels are fabricated on a bottom wafer using photolithography, and reservoirs connecting the ends of the channel are made on a top wafer. The two wafers are then bonded together by water-bonding.

Summary of Research:
The fabrication procedure has been developed for repeatable and reliable fabrication of the nanometer-sized channels, sealed between two fused silica wafers.

Each wafer assembly consists of two fused silica wafers bonded to each other and contains 82 individual flow-cells fabricated on it. Each flow-cell consists of two reservoirs 3 mm away from each other. A 1 mm wide and 400 nm deep channel connects these reservoirs. Halfway from the reservoirs, the channel is divided by a 15 µm wide and 400 nm high ridge, in which 500 nm wide channels are made.

In the past years, the nanochannels were used to develop a new method for nanoparticle recognition [1-3]. The sensitivity and discrimination capability of these interferometric detection methods are continually being improved [4, 5]. Additionally, new applications in biological [6] and medical [7] fields are being explored.

Fabrication Procedure:
A fused silica glass wafer (Mark Optics, USA) was pre-cleaned in a nanostrip bath for 30 minutes. The wafer was vapor primed in the YES oven. The wafer was spin-coated with i-line photoresist (OiR 620-7i) at 3000 RPM for 30 seconds with three seconds ramping speed. The nano-sized parts of the flow-cell were patterned using the GCA Autostep 200 DSW i-line wafer stepper, and the micro-sized features were patterned using EV620 contact aligner (Electronic Visions, Phoenix, AZ). The channels were etched using reactive ion etching technique in the Oxford 81 etcher. The remaining resist was then removed using acetone and in the hot resist stripper. Second glass wafer was used to seal the channels. Holes for liquid delivery were made in the second glass wafer using sand-blasting tool. Both wafers are cleaned in acetone, powder detergent solution, isopropanol, methanol, nanostrip, and also in the MOS clean area. They are then bonded together using wafer-bonding, and dried overnight and also in the 90° convection oven for two hours. Finally, the bonded wafers are annealed in the N₂ anneal furnace.

References:
Figure 1: Optical micrograph of the fabricated glass nanochannels.

Figure 2: AFM topography of a nanochannel.
Leveraging Opto-Mechanics to Design High Performance RF Oscillators

CNF Project Number: 1380-05
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Abstract:
Cavity opto-mechanics enabled radiation pressure (RP) driven oscillators offer an all optical radio frequency (RF) source without the need for external electrical feedback. We developed a process to fabricate opto-mechanical resonators, waveguides and grating couplers in silicon nitride, and demonstrated an RP driven opto-mechanical oscillator (RP-OMO) operating at 41 MHz. In addition to this amplification of mechanical motion, we also achieved simultaneous cooling of another mechanical resonance at 77 MHz by exploiting two closely spaced optical cavity resonances. We also developed a monolithic silicon acousto-optic modulator operating at 1.09 GHz and demonstrated frequency modulation of a 1564 nm wavelength pump laser.

Summary of Research:
Quartz oscillators in the tens of megahertz range offer superior far-from-carrier phase noise [1], but have high flicker noise. MEMS-based oscillators are smaller in size and span various transduction schemes [2-4]. Barring Steeneken, et al. [4], the other oscillators are not self-sustained and need a feedback amplifier. The amplifier’s flicker noise shows up as 1/f^3 noise for close-to-carrier offset frequencies thereby degrading long term stability. An alternative type of oscillator is the RP-OMO, based on strong coupling between high-Q optical and mechanical modes of a resonator [5]. However this silica microtoroid OMO requires a serial and unconventional post-fabrication CO2 laser reflow step.

Our goal is to utilize MEMS-based processes to fabricate chip-scale, integrated RP-OMO in a CMOS compatible process. A low phase noise RP-OMO demands high-Q mechanical and optical modes, large moving mass and power handling. Hence, we choose silicon nitride as the device layer, which has high mechanical [6] and optical [7] Qs.

We start with silicon wafers with 4 µm SiO2 thermally grown and deposit 300 nm low pressure chemical vapor deposition (LPCVD) Si3N4. The devices are fabricated using e-beam lithography followed by reactive ion etching into the nitride device layer. Next we deposit plasma enhanced chemical vapor deposition (PECVD) SiO2 cladding over the devices. A second mask is used to pattern release windows near the resonator. This is followed by a partial reactive ion etch (RIE) into the cladding to reduce release time. We then perform a timed release etch in buffered oxide etchant and dry the chip using a critical point dryer to prevent stiction. A scanning electron micrograph (SEM) of the resulting device is shown in Figure 1.

Due to the rich mode spectrum of a silicon nitride microring resonator, situations may arise where the resonator has multiple optical mode families. The modal refractive indices of these mode families may have different temperature dependence. As such, it is possible to fix the laser wavelength...
such that the pump laser light is red detuned with respect to one of the cavity modes and blue detuned with respect to the other in thermal equilibrium. This allows for amplification (self-oscillations) of a mechanical mode with simultaneous cooling (damping) of another mechanical mode, as shown in Figure 2. A complete analysis of the oscillations is presented in [8] and the simultaneous heating and cooling is explored in [9]. The oscillator does not have any 1/f or higher order slopes in the phase noise spectrum [8].

In addition to designing silicon nitride opto-mechanical oscillators, we also recently demonstrated an integrated monolithic silicon acousto-optic frequency modulator [10]. Figure 3 shows an SEM of the device. The frequency of mechanical motion (1.09 GHz) is comparable to the photon lifetime in the optical cavity in this device (1.27 GHz at 1564 nm). The motion of this opto-mechanical resonator thus results in generation of asymmetric higher and lower frequency sidebands around the input laser line in the optical frequency domain as shown in Figure 4. This asymmetry can be explained by coincident amplitude modulation and frequency modulation on the laser light. The mathematical treatment and experimental details are presented in Tallur, et al. [10]. This constitutes the first demonstration of electro-acoustically generating photons of different colors from a single pump laser in silicon.

References:

Porous Polymer Waveguides and Ring Resonators

CNF Project Number: 1472-06
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Abstract:
Optofluidic biosensors are promising for future label free diagnostics because of their ability to localize electromagnetic energy in optically resonant structures and concentrate bioanalytes in microfluidic channels. However, many traditional optical biosensors often store the majority of their optical energy away from the bioanalyte solution, leaving only the evanescent field to interact with biomolecules. Nanoporous polymer waveguides and ring resonators were developed that allowed increased interactions between core energy and biomolecules. Our results show a 40% increase in biosensor sensitivity due to these increased interactions.

Summary of Research:
Label-free biosensors represent a promising new technology because of their ability to avoid the use of fluorescent, radio, and enzymatic label molecules. Label-free devices have been developed based on optical, mechanical, and electrical methods of signal transduction [1-4]. Optical devices are of particular interest because of their ability to localize electromagnetic energy and bioanalytes to minute volumes. However, many current optical biosensors rely on fabrication techniques and materials developed in the semiconductor industry, and face difficulties associated with wide-spread fabrication and the cost of devices. Recently, others have investigated the feasibility of using non-traditional nanofabrication techniques and materials to reduce costs and make devices that are easier to mass produce [2]. However, many of these cheaper polymer devices aren’t capable of matching the sensitivity of their silicon counterparts.

In this work, we investigate how the sensitivity of polymer optical biosensors can be increased to match that of silicon devices and how polymers can provide other advantages in optical biosensing, including robustness and flexibility. Specifically, we use nanoimprint lithography as an alternative to traditional techniques and create porous polymer waveguides and ring resonators where biomolecules can interact directly with the core waveguide energy. Because the energy in the waveguide core is many times higher than in the evanescent field, porous devices are more sensitive to changes in the cladding solution than typical ring resonators (Figure 1).

Nanoimprint masters are fabricated on silicon wafers, by depositing a 2 µm film of silicon dioxide, spinning SPR 955-0.9 (MicroChem Corporation, Newton, MA, USA) positive photoresist on top, exposing on an i-line autostepper, and developing the resist. The resulting pattern is then etched into the silicon dioxide using fluorine gas based chemistry, and the remaining photoresist stripped.
Waveguides are then fabricated by depositing a 2-3 µm silicon dioxide cladding onto a silicon wafer, spinning a blend of polystyrene and polymethylmethacrylate on top, and imprinting using the master. Later, dimethylsulfoxide can be used to dissolve PMMA from the composite structure, leaving a porous polystyrene waveguide behind. Figure 2 shows characteristic scanning electron micrographs of an example porous ring resonator.

Waveguides are coupled into using a 1550 nm tunable laser and a lensed fiber and a set of translational stages, and are coupled out of using a lens to collect light leaving the waveguide. Microfluidic channels fabricated using traditional photolithography and PDMS-casting are used to deliver fluids directly to the resonators.

By varying the refractive index of the cladding solution and plotting the resonant wavelength of each ring, it’s possible to determine the sensitivity of each device (Figure 3). Further, by comparing sensitivity results between both pure and porous polystyrene devices it’s capable to determine an approximately 40% increase in the sensitivity of the device, as shown in Figure 4. Considering both polymer devices cost advantages and the sensitivity porous resonators can provide, future devices fabricated this way could be used in many sensing applications.

References:

Figure 2: SEMs of the final device are shown. (A) shows a complete racetrack resonator, (B) shows an end facet creating using the cleave and snap technique and (C) shows the porous ring structure. Note that only the ring is made porous, to reduce losses due to scattering throughout the waveguide.

Figure 3: By plotting the location of a resonant peak as a function of cladding refractive index it’s possible to determine the sensitivity of our ring resonators. A resonant peak is shown for different glucose solution concentrations in both a (A) nonporous resonator and a (B) porous resonator.

Figure 4: Resonant wavelength is plotted as a function of the change in cladding solution refractive index. A 40% increase in sensitivity (nm/RIU) is seen when resonators are made porous.
Fabrication of Phase Grating Based Angle Sensitive Pixels by CMOS Post-Processing

CNF Project Number: 1651-08
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Abstract:
Typical imaging systems generate a two dimensional intensity map of the light incident on them. Light field image sensors capture, in addition to intensity, the direction of the impinging light rays. In previous work [1], we built CMOS sensors called angle sensitive pixels (ASPs) that output both the local intensity and angle of the incident light. This prior work, however, was limited in its imaging capabilities due to drastically reduced quantum efficiency as compared to a conventional active pixel CMOS image sensor. In the present work, we develop device structures for angle sensitive pixels that offer greatly improved quantum efficiency without compromising function. We fabricate these structures by simple post-processing on a standard CMOS die.

Summary:
Angle sensitive pixels rely on a near field diffraction effect, known as the Talbot effect, to extract the local angle of incidence. ASPs use a periodic transmission grating to generate a diffraction pattern. They then detect the incoming angle by detecting lateral shifts in this diffraction pattern. In earlier ASP designs, the diffraction gratings were manufactured using the interconnect metallization layers offered by a commercial digital or mixed mode IC fabrication process. Since these gratings were made of metal they reflected a substantial portion of the incident light, thereby degrading the captured signal.

The periodic diffraction patterns essential for ASP function can be generated without any reflection loss by using binary phase gratings [2]. Phase gratings are periodic structures made of two transparent materials of different refractive indices (e.g., air and glass).

We incorporated phase gratings into our angle sensitive pixels by post-processing foundry fabricated dies containing metal grating designs. The metal gratings serve as templates for the fabrication of the phase gratings. The post-processing involves three simple steps: i) a photoresist mask to protect bond pads and support circuits, ii) a plasma etch to define the phase grating in the interlayer dielectric, and iii) an aluminum etch to remove the metal gratings.

The plasma etch is performed using a CHF$_3$/O$_2$ plasma in order to etch away both the SiN passivation and the SiO$_2$ inter-metal dielectric. The duration of this etch sets the depth of the grating. The other design parameters — the grating period and position — are determined by the metal grating template which adheres to foundry tolerances.

Measurements show that phase grating based angle sensitive pixels show a sinusoidal response to angle that is similar to metal gratings with a quantum efficiency that is nearly four times greater.

References:
Figure 1: Process flow for the fabrication of phase grating from metal amplitude gratings.

Figure 2: SEM image of the fabricated phase gratings.
Demonstration of Optical Nanocircuits at Infrared Wavelengths

CNF Project Number: 1724-08
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Abstract:
In this report, we fabricate arrays of Si$_3$N$_4$ nanorods with specific deep subwavelength cross-sections, quantitatively evaluate their performance as lumped circuit elements in mid-infrared frequencies, and show that the nanorods effectively form a two dimensional optical nanocircuit when they are illuminated by the infrared light. This is the first experimental demonstration of a metamaterials-inspired nanocircuit in mid-infrared frequencies.

Summary of Research:
Our previous theoretical work has paved the way for the realization of lumped circuit elements in optical frequencies. By replacing the conduction current of a standard radio frequency circuit with the optical displacement field current, we can extend the circuit paradigm to optical frequencies. For instance, a nanoparticle can function as a nanocapacitor, a nanoinductor, or a nanoresistor, provided that its permittivity satisfies Re($\varepsilon$) > 0, Re($\varepsilon$) < 0 or Im($\varepsilon$) $\neq$ 0. In this work, we report the first experimental demonstration of a two-dimensional nanocircuit at infrared frequencies.

For the demonstration, an array of deeply subwavelength dielectric nanorods is designed, as shown in Figure 1. In this design, the air gaps are considered as lossless nanocapacitors, the dielectric nanorods are considered as nanoinductors (when Re($\varepsilon$) < 0) in a certain wavelength regime, and nanocapacitors (when Re($\varepsilon$) > 0) at other wavelengths. This led to the low-stressed silicon nitride grown by low-pressure chemical vapor deposition (LPCVD) method. We have shown that this dielectric material has a negative real part of permittivity near 11 $\mu$m, perfect for the designed structure.
The fabrication starts with 4-inch double-side-polished bare silicon wafers. An initial 600 nm low-stressed LPCVD silicon nitride film is grown on the wafers. Different dielectric film thicknesses (225 nm, 300 nm and 375 nm) are achieved by reactive ion etch (RIE). For the e-beam job, about 70 nm ZEP520A e-beam resist is used. And a thin Cr layer is shadow evaporated on the resist patterns to help the etching of different thicknesses (175 nm, 250 nm and 325 nm) of nanorods. After that, both Cr and the e-beam resist are stripped off.

The wafers then go through an optical lithography process, depositing 50 nm gold on the wafer surface, leaving only small windows (600 µm × 600 µm) occupied by the nanorods. A second optical lithography is later done on the backside of wafers, exposing big windows (1.2 mm × 1.2 mm) aligned to the small windows on top. The nitride film in the big windows is removed by RIE, and the silicon wafers are etched through in hot KOH solution, with their top surface protected by a layer of ProTEK B3. After the wet etch, the wafers are dried in a CO₂ critical point dryer.

At this point, the nanorods are supported by a base nitride film of ~ 50 nm, which is carefully removed from the backside using RIE. Finally, all nanorods (thickness: 175 nm, 250 nm and 325 nm) are freely suspended in air, with only their two ends attached. Over all, nine nanorod arrays with different configurations are fabricated (see Figure 2).

The nine samples are measured using an FTIR spectrometer. Their transmittance data is then compared with the circuit theory calculation, and with the result obtained from full-wave simulations. The excellent agreement between experimental results, the full-wave simulations and the circuit theory, clearly demonstrates that the designed nanorod arrays function as optical lumped circuit elements.

Continuous Operation of a Hybrid Solid-Liquid State Reconfigurable Photonic System without Resupply of Liquids

CNF Project Number: 1764-09
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Primary Source of Research Funding: NBTC
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Abstract:
In this work, we demonstrate solutions to the two major technical roadblocks that prevent the widespread adoption of liquid state photonics. By developing an on-chip gravity based liquid core/cladding separation system and unique solid-to-liquid-to-solid state coupling techniques, we have demonstrated here the ability to address two major roadblocks in cooperating solid and liquid state optical components. Quantitatively, we demonstrate the ability to reduce liquid consumption over 1,000-fold compared to the state of the art, while enabling reconfiguration on the order of 100 times greater than what is achievable with other optomechanical approaches.

Summary of Research:

Introduction. Optofluidics offers a number of potentially transformative advantages for photonic systems [1]. At present, however, there are a number of technological roadblocks that prevent the practical integration of liquid-state elements into traditional high-speed solid-state photonic systems. Two of the most important of these are the need for continuous resupply of liquids and the difficulty in shuttling light between the liquid- and solid-states. In the paper [2] we present an integrated system that solves both these problems.

Discussion. Figure 1a-1c, shows the fabrication method used to integrate the liquid and solid waveguides onto a single substrate. Briefly, standard photolithography techniques were used to fabricate a single layer SU-8 structure that contains: the microfluidic channels to form the liquid-core waveguide, the input and output solid-core waveguides, and the separation reservoir. The patterned SU-8 layer was covered by a single PDMS sheet to seal the microfluidic channels and provide more capacity for the separation reservoir (Figure 1c). Working principle of the recirculation system and a microscopic view of the actual device are shown in Figure 1d and 1e. The recirculation was enabled by the use of immiscible liquids, DI water (RI = 1.336) as the core solution and Fluorinert electronic oil (FC-40, RI = 1.22) as the cladding. After forming the liquid waveguide, the core and cladding liquids were collected and separated in the reservoir by taking advantage of the differences in their densities and pumped back into microfluidic channels by external micropumps.

Figure 1: Schematic showing fabrication process used to create the hybrid chip along with a view of the physical chip during end-fire coupling.

Figure 2: (a) Microscopic view of the device without the liquid waveguide. (b-d) Switching between output waveguides. (e-g) Liquid waveguides with small and large curvatures. (h-j) Effect of improved mode matching between the liquid and solid waveguides.
Figure 2a shows an overview of the hybrid photonic system during operation with Figures 2b and 2c illustrating the two end-fire switching states for the liquid core waveguide. Figure 2d shows the profile of the normalized optical intensities for the coupled (red) and non-coupled waveguides (blue) projected onto the reflectors. To test the effects of liquid waveguide curvature on the coupling performance, the coupled output solid waveguides were offset between 5 µm (Figure 2e) and 95 µm (Figure 2f) from the center axis (equivalent to a curvature change from $10^2$ to $10^3$ m$^{-1}$), while the output solid waveguides were fixed 35 µm from the center axis. Figure 2g shows cross-talk values as a function of the offset of the output waveguide. Figure 2h-2i show adaptation of the width of the liquid core by changing the upstream flow conditions. As we decreased the applied pressure of the core flow from 70 to 17 kPa, the width of the liquid waveguide decreased from 50 to 20 µm (shown in Figure 2j) improving the cross-talk value from 5 dB to 12 dB.

We demonstrate evanescent coupling between liquid- and solid-core optical waveguides for the first time. By controlling the pressure of the cladding flow on the left side channel in Figure 3a, we could alter the width of the cladding flow between the liquid and the solid waveguide to change the cross-talk value. Figures 3d and 3e show the projected light on the reflector placed at the end of the output solid waveguide and cross-talk values as a function of the pressure of the cladding flow. The coupling ratio increased as the pressure of the flow decreased to narrow the gap between the liquid and the solid waveguide. At pressures lower than 7 kPa, stable physical contact between the liquid waveguide and the solid waveguide was obtained as shown in Figures 3b and 3c.

Fluidic recirculation is fundamentally important to the development of a practical hybrid system as it allows one to operate the device continuously without having to refill or remove liquids from the chip. To demonstrate the long-term operation of our device we performed the end-fire optical switching for 20 continuous hours, without requiring the resupply of liquids. As above, we measured the system performance using cross-talk values at different switching speeds (1 s, 3 s, and 5 s). The results are shown in Fig. 4.

Figure 4a shows images of the end fire coupling at the start and at the end of the 20 hour operational period at 1s switching speed and Figure 4b shows the cross talk values measured periodically during the operation. The liquid consumption compares with the 40 mL/20 hours of liquid consumed by the previous device for the same time period, representing 200-fold improvement.

**Conclusions:**

For the first time we demonstrate direct evanescent and end-fire coupling between liquid- and solid-state waveguides and an on-chip fluid core/cladding separation and recirculation system that reduces the consumption of liquids more than 200 fold over the state of the art. The device is operated continuously for over 20 hrs without performance degradation or requiring the replenishment of liquids. We believe that our system represents an important step towards the development of practical optofluiddically enabled photonic systems.

**References:**


Microfabricated X-Ray Optics for CHESS

CNF Project Number: 1855-09
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Abstract:

We report on the fabrication and testing of three x-ray optics: a silicon nitride Transmission Mirror (TM), a Spoked Channel Array (SCA), and a von Hamos x-ray energy analyzer. Transmission mirrors together with traditional x-ray reflection mirrors form a tunable broadband optic, which is useful in selecting appropriate x-ray energies for Laue diffraction experiments, and therefore functions as a bandwidth filter. The SCA optic is a fan-shaped arrangement of deep reactive ion etched channels in silicon made using the Oerlikon tool at the CNF. The channel width determines the linear size or collection resolution of x-ray fluorescence from the sample being probed. These measurements determine elemental composition vs. depth of the sample.

Summary of Research:

The Transmission Mirror (TM). The objective of this project is to develop TM optics that are robust and have very long lifetimes. The critical parameters that determine the optical properties must satisfy the conditions of being thin, flat, smooth, and uniformly thick. In previous attempts, the TM lifetime was not long enough in the x-ray beam; lasting from a couple of hours with soap-bubble films to a day or two with Mylar films [1-2]. Si3N4 serves as the appropriate material because of the low atomic number and being radiation-hard. Low stress silicon nitride (LS-SiN) is deposited on a double-side polished silicon wafer. The windows are formed from a backside 80°C KOH etch. A topside dry etch is also performed across the front end prior to the wet etch to make it much easier to free the TM after the through etch.

The von Hamos Analyzer. A von Hamos analyzer is used to collect, energy resolve, and spatially disperse x-ray fluorescence so a detailed line shape is recorded. This optic is realized by the cylindrical bending of an SOI wafer. The scheme as seen in Figure 2(b) [3] is achieved by processing a slotted bar pattern on the handle of the wafer by a deep reactive ion etch of silicon. The handle used to diffract x-rays is about 400 µm thick. It is etched down to the oxide stop layer using photoresist as an etch mask into 121, 10 mm × ½ mm diffracting elements spaced 32 µm apart (Figure 2(a)). The 40 µm device layer can permit a bend radius as small as 100 mm to focus more intensity with no sacrifice to the energy resolution.

Spoked Channel Arrays. The technique of using x-ray fluorescence in obtaining elemental vs. compositional information from heterogeneous samples is the focus of this project. Here, we have added some refinements based on our prior work in fabricating SCAs from silicon for confocal-XRF (CXRF) as an alternative to the glass polycapillary [4-7].

Figure 1: (a) Schematic of silicon nitride TM windows on a silicon wafer. (b) Etched silicon wafer with 300 nm thick silicon nitride windows.

Figure 2: (a) A von Hamos bar analyzer etched from an SOI wafer. (b) Schematic of the analyzer illustrating the geometrical principle of operation.
To improve the efficiency of collecting x-ray fluorescence from a point source, we modeled alternative channel configurations that enable us to achieve better etch depths. Our optic design consisted of three variations having channel widths of 1, 2 and 5 µm. We also included a 1 µm thick sacrificial etch-wall at the entrance and exit of the channel to protect the integrity of the channels. The principle of operation, as shown in the inset of Figure 3, shows the fan-shaped arrangement of the etched channels, which collects a slice of the hard x-ray fluorescence from a point source. The point source is formed by focusing the x-ray beam using a single-bounce monocapillary. By translating the sample through the 3D active volume, a composition vs. depth information can be extracted from the sample as also shown.

References:

Directed Nano Assembly of Materials Using Silicon Photonics and Plasmonics

CNF Project Number: 1857-10
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User: Romy Fain

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        National Science Foundation Graduate Research Fellowship
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Abstract:
The development of a nanoscale manipulation system to construct new material structures using photonic resonators is the focus of this research. Its promise is the ability to build, from the nanoscopic level, any material, naturally occurring or otherwise, in any configuration, revolutionizing every aspect of life: medicine, manufacturing, energy. For example, one possible application of this technology would be high efficiency photovoltaic material construction. This project attempts to make progress towards this goal specifically by using silicon photonic resonators to trap small particles and then fuse them into structures not easily fabricated by previously available methods.

Summary of Research:
Background. Photonic manipulation is one area of particular promise for nanoscopic manipulation. In this field, highly concentrated light inside photonic devices is used to transfer the momentum from incident photons to a particle. This transfer of momentum can be utilized to manipulate nanoparticles extremely precisely via radiation pressure and subsequent near-field optical trapping forces associated with light escaping from a source.

Using focused laser light to hold and move a particle, or optical tweezing, is a precise method of moving microscale particles. But there is a physical limit to the size of particle that can be manipulated due to the trapping force on the particle being proportional to the cube of the particle radius. Attempting to increase the intensity of light to handle smaller particles encounters other fundamental limits in diffraction and coupling between focal depth and light species interaction length. For these reasons, it is extremely difficult to manipulate dielectric particles smaller than 100 nm in this manner [1].

Near-field photonics picks up where optical tweezers leave off, in that a trap for particles as small as 10 nm with input powers of 10 mW is theoretically possible and 22 nm has been previously demonstrated in this lab. Usually with this method, optical resonators are evanescently coupled to a single mode waveguide. One example of such a resonator that we utilize is a ring shaped waveguide resonator that guides certain wavelengths and amplifies them to a terminal intensity as more light is evanescently coupled into it via a straight rectilinear waveguide.

Plasmonics offers other advantages, including 40 times the trapping forces of optical tweezers. With this technique, coherent oscillation of valence electrons in metals is used to enhance the intensity of the evanescent field [2]. And Liu, et al. [4], has demonstrated using plasmonics to induce and control the rotational speed and direction of a nano structure. The torque generated from their 100 nm rotor was sufficient to rotate a silica disk 4,000 times larger in volume than the rotor itself.

Methods:
We have demonstrated reliably repeatable particle trapping using silicon photonic ring resonators (see Figure 1).

We expect to build structures with these and other resonator designs from nanoscale particles, for example, by using...
biochemistry and the binding affinities of avidin and biotin to bind functionalized particles to each other. Other possibilities include fusing particles thermally on the resonator and incorporating plasmonic structures to increase trapping forces (see Figure 2).

**Goals:**

1. Capture, transport, and assemble sub-100 nm particles
2. Assemble a useful material that could not be assembled by other methods, for example: an ultra high-efficiency piezoelectric or photovoltaic material
3. Manipulate biological particles including viruses, DNA and various proteins including guided diffusion
4. Build a nanoscale robot from nanotubes of various chiralities with differentiated properties, for example: electrode, semiconductor, piezoelectric

**References:**


Optical Manipulation of Nanoscale Objects
and Biomolecules with Photonic Crystal Nanotweezers

CNF Project Number: 1857-10
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Abstract:
We characterized previously demonstrated photonic crystal resonators for optical tweezing. We introduced a formalism to characterize the transport properties in the vicinity of the photonic crystal traps. With the knowledge of the device limitations, we went on to demonstrate the novel trapping of biomolecules (Wilson Disease Molecule). Last, we added a new component to the possibilities offered by these devices: optical torque wrenching.

Summary of Research:
We previously developed and demonstrated optical tweezing with photonic crystal resonators [1]. Nanophotonic resonators are particularly convenient as they offer the possibility to confine and amplify light fields to sub-wavelength geometries. This is of great interest in optical trapping since the gradient force, the optical force arising from the interaction of light and particles, which allows for trapping, scales with the gradient of the field intensity. With this method, and the improvements we implemented, we theoretically predicted an excellent confinement of the particles down to 7 nm for only 10 mW of power input. We also predicted trapping stiffness as high as 69 pN nm⁻¹ W⁻¹ and a potential depth of 170,000 kBT.

We characterized the transport properties in the vicinity of the optical hotspot and evidenced that thermophoresis plays a great role in the transport of solutes. We introduced an energetic method to compare the physical phenomena. With the understanding of the flow and thermal properties of the resonators, we addressed its weaknesses. To that intent, we designed and built a photonic crystal resonator operating at 1064 nm and made of silicon nitride rather than silicon. This new device, as we showed, allows for similar trapping characteristics yet does not suffer from the thermal issues of our previous devices. With this new device, we trapped and manipulated nanoscale objects such as 22 nm polystyrene beads, quantum dots, and biological molecules such as the Wilson Disease Protein.

The photonic crystals were designed using a deterministic method recently published with the CNF cluster and MPB/MEEP. The devices were fabricated with electron-beam lithography using the JEOL 9300. The silicon was etched with a chlorine recipe in the Plasma Therm 770 (left chamber), while the silicon nitride was etched in the Oxford 100 (CHF₃/O₂). Last, up to 3 µm of a silicon oxide layer was sputtered with the CVC (lift-off process) on the non-active components.

Further investigating the behavior of an optofluidic photonic crystal resonator, we are interested in the thermal effects of the resonant silicon device on the particle transport. Water, in the microwave wavelength has a high absorption coefficient that causes it to warm up under the influence of a silicon resonator. In particular, when one is to increase the interaction between molecules and electromagnetic field, the heat generation is also increased. The power lost to heat can be estimated by electromagnetic simulations. The temperature profile is then used as a source term in the Navier-Stokes equations and diffusion equation to solve for the flow, including the temperature-induced buoyancy and thermophoresis.

Thermophoresis is found to play a predominant role in the transport of solutes near the resonators. In order to compare the forces at play (thermophoresis, optical forces, and entropic forces), we chose an energetic approach expressing the thermophoretic free energy computed from the concentration landscape. We find that the thermophoretic free energy constitutes a barrier to the diffusion of particles. Although smaller in magnitude than the electromagnetic well (10 kT vs. 1000 kT), it spans a much wider region and prevents molecules from diffusing [2].
Changing the operating wavelength to 1064 nm lowers the absorption of water by two orders of magnitude. Because silicon is not transparent at 1064 nm, we had to change material to silicon nitride, therefore changing the fabrication method and design. Thermal and flow theoretical study of the photonic crystal reveals a temperature increase reduced by two orders of magnitude and a free energy barrier also reduced by two orders of magnitude.

With this new device, we demonstrated trapping of 22 nm polystyrene beads nanoparticles, and 10 nm quantum dots. We also demonstrated trapping of biomolecules (Figure 1), trapping Wilson Disease Protein (WD 10); an ~ 46 kDa molecule approximately 10 nm in size [3].

References:
Highly Sensitive Spectroscopic Interferometers Using Photonic Crystal Structures

CNF Project Number: 1862-10
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Abstract:
We propose using slow light to greatly enhance the spectral performance of on-chip interferometers. We propose various optimized photonic crystal structures to improve the performance of various types of integrated spectrometers. We fabricated the proposed waveguide structure on silicon-on-insulator platform using e-beam lithography and induction coupled plasma (ICP) etching.

Summary of Research:
Photonic crystals have become an important component for a wide variety of applications, such as light sources, filters, modulators, platforms for quantum electrodynamics study, etc. Meanwhile, miniaturized spectrometers are key components to realize various types of systems-on-a-chip for, e.g., integrated molecular and biological substance detection and recognition and wavelength-division-multiplexing-based telecommunications and optical signal processing. In this project, we study the use of photonic crystal structures for on-chip spectroscopic applications. In particular, we study three types of photonic crystal (PhC) devices, including the use of slow-light PhC line-defect waveguides to construct on-chip slow-light interferometric spectrometers, high-quality-factor PhC cavities as frequency selective filters, and an engineered PhC super-prism spectrometer.

It has been recently shown that slow light can be used to enhance the performance of various types of spectroscopic interferometers [1, 2]. Here we use calzone PhC line-defect waveguides as the slow-light medium [3] to construct on-chip interferometric spectrometers. Despite the difference in geometries of various types of interferometers, the potential spectral performance of an interferometric spectrometer is primarily determined by the properties of the constituting slow-light media. By controlling the geometric parameters, we can obtain optimized structure designs with a large and almost constant group index over different desired ranges of wavelength.

Under practical fabrication conditions, a group index of 30 over approximately 10 nm of wavelength range can be achieved with relatively low propagation loss and spectral resolution of a few to tens of gigahertz. Secondly, we have constructed high-quality-factor PhC cavities with optimized vertical coupling [4]. By cascading such structures through a bus waveguide, one can construct a spectrometer with resolution as fine as tens of megahertz. Different frequency components of the input spectrum from the bus waveguide are coupled vertically by cavities at different locations, and can be read out by a regular observation camera.

Lastly, photonic crystal can have very high frequency-dependent anisotropy, which can lead to the superprism effect. By engineering the hole shape and the lattice geometry, we show that one can obtain a PhC super-prism structure with linear angular dispersion over a large wavelength range, and therefore can lead to a miniaturized prism spectrometer with resolution on the order of 1 nm.

Figure 1: (left) SEM micrograph of a L-3 PhC cavity with optimized structure for vertical coupling; (right) measured resonance of such a L-3 PhC cavity with a Q factor of approximately 67k.
In summary, we have shown that by engineering different types of photonic crystal structures, one can achieve various types of on-chip integrated spectrometers with resolutions ranging from megahertz to hundreds of gigahertz to meet different spectroscopic demands.

**Acknowledgements:**

This project received support from the Defense Threat Reduction Agency-Joint Science and Technology Office for Chemical and Biological Defense (grant no. HDTRA1-10-1-0025). This work was performed in part at the Cornell NanoScale Science and Technology Facility, a member of the National Nanotechnology Infrastructure Network, which is supported by the National Science Foundation (Grant ECS-0335765).

**References:**

Gel Optical Waveguides with Live Encapsulation and Integrated Microfluidics

CNF Project Number: 1872-10
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Abstract:
We demonstrate optical waveguides fabricated out of agarose hydrogel that are capable of encapsulating live cells, and other biological molecules, within the core of the waveguide where it is possible for them to interact with the direct optical mode rather than the weak evanescent field. We also demonstrate integrated microfluidics for possible optofluidic and biosensing applications.

Summary of Research:
Our project looks at developing optical devices from non-traditional materials for biological applications. Typical optical devices use the weak evanescent field for biosensing [1] or optical manipulation [2]. Hydrogels provide an alternative to traditional silicon or silicon nitride devices in that they allow for direct encapsulation for cells and biological materials within the body of the gel itself. They have been explored earlier for fabricating optical devices, but the methods used have been incompatible with incorporating biological material within the gel [3, 4]. We try to alleviate these difficulties by using agarose hydrogel, which is known to be biocompatible and has been used earlier to encapsulate cells [5], and can be used to form microstructures using soft lithography techniques [5].

Figure 1 shows the hydrogel waveguides fabricated using a soft lithography approach. A 2% (w/v) agarose gel (refractive index n = 1.3357) served as the waveguide core, which was patterned on a previously prepared substrate of 1.5% (w/v) (n = 1.3343) agarose hydrogel using a patterned PDMS stamp. The PDMS stamp was, in turn, cast from photolithographically fabricated SU-8 masters. The final structure consisted of multimoded rib optical waveguides of size 130 × 130 µm and were found to guide light effectively.

We further quantified the scattering/absorption losses of our waveguides by measuring the intensity of the scattered light along the length of the waveguide. We found the losses to be of the order of 13 dB/cm with small deviations across several waveguides (Figure 2), which were comparable to other hydrogel optical systems [3].

We also integrated microfluidics over the soft optical waveguides, using an ‘open-end’ channel design as shown in the inset of Figure 3. The open end design consisted of microchannels cut open at the two ends and then aligned along the length of the waveguide to prevent any part of the microfluidic layer from pressing down upon the soft waveguides. The ends were later sealed with a solution of low melt agarose which was allowed to slowly gel in air.

In order to demonstrate the bio compatibility of our fabrication process, we encapsulated live metastatic cancer cells (MDA-MB231) within our gel waveguides. We confirmed the viability of the cells within the gel waveguides by using Calcein AM, a widely used live cell stain. As can be seen in Figure 4, the stain was excited by the 488 nm light coupled into the waveguide and live cells could be seen fluorescing brightly when observed under a fluorescence microscope.

References:

Cornell NanoScale Science & Technology Facility
Figure 1: Agarose gel waveguide with 2% (w/v) gel as core and 1.5% (w/v) gel as substrate (Inset: magnified view) [6].

Figure 2: Scattering/Absorption losses measured across different waveguides. Dotted line indicates the average measured loss [6].

Figure 3: An assembled device with both the optical and the microfluidic layer (Inset: A cross sectional view of the open end architecture).

Figure 4: Gel waveguide, with Calcein-stained cells embedded within the core, coupled to an argon laser (488 nm). The waveguide has been imaged under a fluorescence microscope and live cells have been circled for clarity [6].
Fabrication of Three-Dimensional Simple Cubic Silicon Woodpile Photonic Crystal Structures

CNF Project Number: 1880-10
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Abstract:
A four-layer three-dimensional (3D) photonic crystal of simple cubic woodpile structure is realized with hydrogenated amorphous silicon. The periodicity of the structure is 300 nm; the width and height of the silicon are both 150 nm. The absorption is increased from 60% to 90% in wavelength range 400-600 nm and the peak at around 650 nm is increased from below 20% to 70%, when compared to a 600 nm a-Si:H thin film.

Summary of Research:
3D photonic crystals possess photonic bandgaps that prohibit the propagation of light in certain wavelength range in all three directions in space. A layer-by-layer woodpile structure can exhibit a full 3D photonic bandgap. They also can show other potential applications such as enhanced and selective infrared emission near the band edge. It is also of great interest to utilize photonic crystals for photon management and visible light trapping. In order to control optical light in the visible wavelength range, the physical dimensions of the structures have to be in nanoscale. A recent theoretical study showed that 3D photonic structures could greatly enhance the light trapping and absorption in thin film photonic crystal structures due to strong resonances by parallel interface refraction (PIR).

Here, we report on a four-layer simple cubic woodpile photonic crystal structure successfully fabricated with hydrogenated amorphous silicon (a-Si:H) and its enhanced absorption over an unpatterned a-Si:H thin film. In Figure 1, a schematic of the 4-layer (4L) simple cubic woodpile structure is shown. The periodicity of each silicon-grating layer is 300 nm, and the width and height of the silicon are both 150 nm. Each layer is aligned with the grating direction rotated 90° against the adjacent layers.

Firstly, a 150 nm thick amorphous silicon thin film is deposited on a glass substrate by PECVD. Then the JEOL e-beam lithography system (JBX-9300FS) is used to create grating pattern of 150 nm width. Single step deep RIE etching is used to transfer the pattern to the underneath amorphous silicon. Hydrogen silsesquioxane (HSQ) is spin-coated onto the pattern to provide a filling dielectric material and for structural support. It is converted to low refractive index oxide by thermal curing. Excess HSQ is removed and the surface of the each layer is planarized by RIE etching. A 4L woodpile structure is fabricated by this process with the precise alignment of the e-beam lithography system for the adjacent layers. (See Figure 2.)
The total transmission and reflection of the sample are measured by an integrating sphere with a xenon lamp white light source. The absorption can be calculated with the equation: \( \text{Abs} = 1 - (T+R) \). It shows the absorption is increased from 60% to 90% in the shorter wavelength range (400-600 nm) and the peak at around 650 nm is increased from below 20% to 70%, when compared to a 600 nm a-Si:H thin film. The 4L sample is also 600 nm in total thickness. However, due to the 50% HSQ filling, the equivalent volume material is only that of a 300 nm a-Si:H thin film. Therefore, the structure has enhanced absorption while using half of the active material. The next step is to study the effect of different periodicity of the structure on the absorption spectra.

**References:**


Waveguide Investigation for Single Molecule Detection

CNF Project Number: 1920-10
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Abstract:
The integration of optical waveguides in devices has the potential to significantly reduce the input power requirements. A process flow utilizing LPCVD and PECVD methods to deposit nitride films was used to investigate the coupling efficiency and AF of waveguides. The zero-mode waveguides are such a structure and Pacific Biosciences has extended its work in studying it for its optical confinement and chemical properties. Investigation of enhancing the chemical properties of the zero-mode waveguide was done by over etching into the zero-mode waveguide to make it have a deeper hole.

Summary of Research:
Research involving waveguide structures requires a low AF to be practical in single molecule detection. The waveguide structures were patterned using CNF’s ASML 300C DUV stepper and Oxford 82 to etch away the excess nitride film. A layer of oxide was then deposited over the waveguides using the GSI PECVD tool.

Pacific Biosciences has extended its research on ZMW to include a deeper well. This eZMW was fabricating by using an oxide etch with the Oxford 81 to etch further into the fused silicon.

Figure 1: A nitride waveguide surrounded by oxide.

Figure 2: A zero-mode waveguide with an 89 nm over-etch into the FUSI.
A High-Frequency Silicon Optomechanical Oscillator

**CNF Project Number:** 1997-11  
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**Abstract:**  
We demonstrate a compact silicon optomechanical oscillator with a large intrinsic mechanical frequency-Q product of $4.32 \times 10^{12}$ Hz. It is able to operate at a high frequency of 1.294 GHz in the air.

**Summary of Research:**  
Recently, self-sustaining optomechanical oscillators — realized by taking advantage of strong dynamic back-actions in micro-/nano-optomechanical cavities — have attracted a great deal of research interest [1-3]. In this work, we successfully demonstrate a high-frequency optomechanical oscillator on silicon-on-insulator (SOI) platform by combining strong optomechanical coupling, tiny effective motional mass, high optical and mechanical qualities into a single compact device. The employed device structure is a compact silicon microdisk resonator with a radius of 2 µm (Figure 1), which is fabricated on a SOI wafer with a silicon layer of 260 nm and a buried oxide layer of 2 µm. The device pattern is defined using e-beam lithography with ZEP520A photoresist. Silicon layer is etched by fluorine-based plasma etching. And finally the silica pedestal is under-etched by hydrofluoric acid.

In general, the optomechanical coupling in a whispering-gallery cavity scales inversely with device radius, $R$, as $g_{om} = -\omega_0/R$. We are able to shrink the device radius down to only 2 µm while maintaining a high optical quality factor due to the high refractive index of silicon. Radiation pressure in the device couples strongly to the radial-stretching mechanical mode (RSMM) whose dominant motion is along the radial direction of the microdisk. Simulation by the finite-element method (FEM) shows the mechanical frequency is 1.261 GHz for the fundamental RSMM in the microdisk of 2 µm radius with an effective motional mass of 5.7 picogram. A large undercut-to-radius ratio of 95% is achieved to dramatically suppress the clamping loss [4] and thus enable a high mechanical quality factor even at a high mechanical frequency.

The device is tested in the air environment. The employed optical TE mode at 1502.03 nm exhibits an intrinsic optical $Q$ factor of $3.5 \times 10^5$ (Figure 2). By locking laser frequency half-way into the cavity resonance at the blue-detuned side, we measure the RF spectrum of the cavity transmission, and clearly observe the fundamental RSMM at 1.294 GHz with a mechanical $Q$ factor of $3.34 \times 10^3$. Consequently, the mechanical mode exhibits a frequency-quality product of $4.32 \times 10^{12}$ Hz. To enhance the dynamic back-action for exciting optomechanical oscillation, we increase the laser-cavity detuning at the blue-detuned side to about 1.6 times the linewidth of the loaded cavity, so that the created Stokes sideband falls into the cavity resonance.

Figure 3 clearly shows that the mechanical mode is excited into coherent oscillation and the peak value of the mechanical spectral intensity is dramatically enhanced by more than 50 dB when the dropped optical power is increased to 4 µW. Detailed analysis of the mechanical spectrum (inset of Figure 3) shows that the mechanical linewidth is drastically suppressed down to 854 Hz, corresponding to an effective mechanical $Q$ factor as high as $1.52 \times 10^6$. 

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**Figure 1:** Scanning electron microscopic (SEM) image of the device. The silicon microdisk sits on a 2 µm high silica pedestal with an undercut width of ~1.9 µm measured from the image.
Figure 2: Experimentally recorded (blue) cavity transmission for a TE optical mode with an intrinsic $Q$ factor of $3.5 \times 10^5$, together with a theoretical fitting (red). The cavity resonance is located at 1502.03 nm. The inset shows the optical field profile. (See full color version on inside front cover.)

Figure 3: RF spectrum of the optomechanical oscillator. The red and blue traces show the cases with dropped optical power of 0.63 and 4.0 $\mu$W, respectively. The inset shows the detailed spectrum of the oscillator, where the red curve shows a Lorentzian fitting. (See full color version on inside front cover.)

References:


Effects of Interfacial Electronic Structure on In-Plane Spin-Transfer Torque in MgO Magnetic Tunneling Junctions

CNF Project Number: 111-80
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User: Yun Li

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Abstract:
Here, we report the study of magnesium oxide (MgO)-based magnetic tunneling junctions (MTJs) with symmetric electrodes FeCoB in both the as-grown and annealed (350°C) state. Analysis of the adjusted torque indicates that the annealing reduces the inelastic tunneling component of the junction conductance, and creates or enhances a peak at about 0.15V above the Fermi level in the density of states of the minority band of one or both of the electrodes. This peak location is essentially the same as the peak of the minority band states that has previously observed on the surface of bcc (100) Fe [1-2]. Thus we conclude that the most likely source of the asymmetry of the bias dependent torque is due to Fe minority band states that are localized at the electrode-tunnel barrier interface.

Summary of Research:
In this report, we study MgO-based MTJs of the composition (thickness in nanometers) bottom contact/IrMn 10/CoFeB 2/ MgO 1/CoFeB 1.5/ top contact. The top CoFeB was patterned by e-beam lithography and an ion beam etching technique to produce an elliptical or circular cross-section with a nominal size of 200 × 120 nm2 or 150 × 150 nm2 for current induced switching and spin torque ferromagnetic resonance (ST-FMR) measurement (Figure 1). The resistance-area (RA) product for the parallel configuration in our as-grown MTJs (annealed) is about 16Ω*µm2 (20Ω*µm2).

The etching was stopped at the MgO barrier and thus the bottom layer is largely unpatterned to minimize the stray field from the bottom ferromagnetic electrode. After the patterning process, some of the MTJs were measured directly and others cut from the same wafer were annealed at 350°C for 1h under an easy-axis magnetic field of 2T in vacuum to enhance the tunneling magnetoresistance (TMR).

Our results indicate that the tunneling mechanisms for two cases. For the as-grown sample (Figure 2), inelastic tunneling determines the bias dependence of in plane torque. Specifically, the distribution of inelastic tunneling centers is asymmetric, with more scattering on the left side.

This asymmetry is probably due to the film growing process: the left interface (between the bottom electrode and the tunneling barrier) is further oxidized or damaged by oxygen plasma during the following MgO sputtering, which does not happen for the right interface.

After annealing (Figure 3), the dominated tunneling mechanism is elastic tunneling with DOS dependent on energy. Especially there is a DOS peak (probably interface states of electrodes) in minority band located just above the Fermi level.

Considering the fact that the main difference between those two samples is the crystalline structure of the FeCoB, our results indicate that: First, the inelastic tunneling is favored in the MTJs with amorphous electrodes, while annealed samples prefer elastic tunneling. Regarding B diffusion and its reaction with O at the interface during the annealing, the interfaces have been cleaned. With improved interfaces and the barrier, inelastic tunneling could be suppressed, leading to a much higher TMR. Second, samples with crystalline electrodes have much sharper feature in minority DOS close to Fermi level than those with amorphous FeCoB layers, which is reasonable because the disorder in crystalline structure usually acts to broaden the DOS peaks.

References:
Figure 1: AFM image of nanopillar array.

Figure 2: Schematics to illustrate the tunneling mechanisms for the as-grown sample.

Figure 3: Schematics to illustrate the tunneling mechanisms for the annealed device.
A Three-Terminal Spin Transfer Torque Device Utilizing the Giant Spin Hall Effect of Tantalum

CNF Project Number: 111-80
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Primary Source of Research Funding: Cornell Center for Nanoscale Systems
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Abstract:
We demonstrate a new three-terminal spintronics device that utilizes the spin Hall effect (SHE) induced spin transfer torque (STT) from tantalum (Ta) to efficiently and reversibly switch the magnetic orientation of a thin free layer electrode of an MgO magnetic tunnel junction having in-plane magnetization. The low write currents (≤ 1 mA), large output impedance and good thermal stability (45 kBT) that has been achieved with this SHE three-terminal device approach, which separates the write and read operations in a manner that is relatively straightforward to fabricate, demonstrate an attractive candidate for application in next generation STT MRAM and non-volatile spin logic circuits.

Summary:
Spin-polarized currents can be used to apply torques to magnetic moments by direct transfer of spin angular momentum, thereby enabling manipulation of nanoscale magnetic devices using currents that are orders of magnitude lower than required for magnetic-field-based control [1-3]. So far the only way to generate spin currents strong enough for spin-torque manipulation of magnets in practical applications has been to send an electron current through a magnetic polarizing layer, with the result that the most promising device geometry for applications has been the two-terminal magnetic tunnel junction (MTJ), possessing a ferromagnetic layer (FM)/tunnel barrier/FM structure. MTJs can have excellent spin torque efficiency, but are proving challenging to operate reliably: it is difficult to manufacture large scale memories in which enough spin current can pass through the tunnel barriers to drive reliable magnetic switching without occasionally damaging a barrier.

On the other hand, it has been known for some time that a spin current can, alternatively, be generated in non-magnetic materials by the spin Hall effect (SHE) [4-6], in which spin-orbit coupling causes electrons with different spin orientation to deflect in different directions yielding a pure spin current transverse to an applied charge current. Here, we study the SHE in the high resistivity form of tantalum (β-Ta). We employ this giant SHE to implement a novel three-terminal device geometry in which the SHE-STT from Ta produces current-induced switching of in-plane polarized CoFeB layer, with read-out using a magnetic tunnel junction with a large magnetoresistance. This geometry is straightforward to fabricate and can have comparable efficiency to conventional two-terminal MTJs while providing greatly improved reliability and output signal levels, and therefore offers a superior approach for magnetic memory and nonvolatile spin logic applications.

The device we demonstrate here consists of a micron-sized Ta strip and, on top of it, a CoFeB/MgO/CoFeB nano-pillar magnetic tunnel junction (MTJ) as shown in Figure 1.

Figure 1: Schematic illustration of the Ta three-terminal SHE-STT device and the circuit layout for differential resistance (dV/dI) measurements.
The multilayer Ta(6.2)/Co_{40}Fe_{40}B_{20}(1.6)/MgO(1.6)/Co_{40}Fe_{40}B_{20}(3.8)/Ta(5)/
Ru(5) (thickness in nanometers) was first sputter deposited onto a thermally-
oxidized Si substrate then patterned into designed structure by e-beam
lithography and subsequent ion-milling processes. The device showing
here has a MTJ pillar lateral size ~ 100 nm × 350 nm with its long axis
perpendicular to the Ta micron-channel direction. An SEM image of the
device is shown in Figure 2. The resistance of the bottom Ta channel is
about 3 kΩ, which corresponds to a resistivity around 170 μΩ·cm. It has
been known that β-Ta is widely seen in sputter deposited Ta thin films and
the resistivity can be as high as 200 μΩ·cm. Therefore, the high resistivity of
Ta channel in our device indicates the existence of β-Ta.

The field-induced switching behavior of the device is shown in Figure 3. By
applying an in-plane external magnetic field $B_{\text{ext}}$ parallel to the long axis of
the MTJ, the differential resistance ($dV/dI$) was recorded by a typical lock-in
method with a small amplitude sensing current. The TMR minor loop of the
device showing here indicates a coercive field $B_C \approx 4$ mT and a TMR ≈ 50%.
The DC current-induced magnetization switching behavior of the device is
shown in Figure 4. We applied an in-plane external magnetic field of -3.5
mT to cancel the dipole field from the top fixed layer. While applying a DC
current along the Ta bottom channel, the device can also be switched in
between antiparallel (AP) state and parallel (P) state at $I_{\text{DC}} \approx \pm 1$ mA.
The switching polarity corresponds to a negative spin Hall angle, as
predicted by previous theoretical studies. We also measured the slight
variance in critical switching currents with respect to current ramp rate. By
fitting the data of critical switching currents into a thermally-assisted spin
torque switching model [7], we found the zero-thermal-fluctuation critical
switching currents for AP to P and P to AP are almost the same $I_{c0} = 2.0 \pm 0.1$
mA, with the energy barrier between two states to be $U = 45.7 \pm 0.5$ kBT.
From a typical STT switching formula, we further estimated the spin Hall
angle of β-Ta to be 0.15 [8].

References:
(2000).
Spin-Torque Effect in Asymmetric FeCoB/MgO/FeB Magnetic Tunnel Junctions

CNF Project Number: 111-80
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Abstract:
Spin-torque effect in asymmetric FeCoB/MgO/FeB magnetic tunnel junctions exhibits asymmetric electronic transports, tunneling magnetoresistance (TMR) bias dependence and spin-torque effects. As-grown amorphous FeCoB and FeB alloy electrodes exhibit asymmetric transport and TMR bias dependence (TMR~20%), regardless of voltage bias polarities. Annealed FeCoB and FeB electrodes show high TMR (~90%) and reverse the asymmetry in the TMR bias dependence, conductance dI/dV and spin-torque ferromagnetic resonance measurements. These results suggest the spin-dependent electronic transports in the annealed asymmetric junctions dominate the transport behaviors and could possibly related to the strong peak in the minority density of state in bcc Fe electrodes.

Summary of Research:
Spin-torque effect enables electrical manipulation of a single nanomagnet. Because of its potential application to non-volatile random access memory, spin-torque effect has been studied extensively. Recent studies have found that there are two different spin torques. One is called in-plane torque acting like anti-damping effect. Another torque affects nanomagnets like magnetic fields and is called the field-like torque. Experiments [1, 2] have confirmed the existence of both types of spin torques.

Furthermore, several studies has found that the field-like torque could induce unreliable switching and could possibly affect switching reliabilities. Therefore, theses effects may significantly hinder the commercial application.

Oh, et al., [3] shows asymmetric electrode compositions in magnetic tunnel junction could lead to back-hopping switching. Here, we found that field-like torque is highly sensitive to chemical compositions of electrodes. The field-like torque can further be altered and manipulated by chemical compositions in the electrodes.

To understand how electrodes affecting the spin-torque effect, we studied the switching phase diagram (SPD) by measuring the P-to-AP and AP-to-P switching fields with various dc bias voltages. By the variation of switching fields, we could quickly and roughly estimate the field-like torque effect. Figure 1 shows the switching phase diagram of nanopillars with FeCoB/MgO/FeB and FeB/MgO/FeCoB electrode compositions.
compositions. Figure 1 ([a], [b]) are the switching phase diagram from as-grown nanopillar devices and ([c], [d]) are from annealed nanopillar devices. As-grown devices are able to perform bi-polar switching within certain field ranges in both electrodes composition.

Figure 2 is the differentiate conductance $dI/dV$ in asymmetric MTJs. Figure 2 ([a], [b]) are as-grown nanopillar devices exhibiting similar voltage bias dependence in $dI/dV$. Interestingly, annealed nanopillar devices show opposite $dI/dV$ while exchanging the electrode compositions. Other than $dI/dV$ reversal in the asymmetric MTJs, tunneling magnetoresistance (TMR) also reversed. Regardless of electrode chemical compositions, $dI/dV$ with positive voltage bias increases faster than negative voltage in all as-grown devices. The high $dI/dV$ with positive bias suggests there are more accessible states or lower barriers in the electrode-insulator interface from MgO RF sputtering. These as-grown results indicate sputtering process dominates the electronic transport properties in the asymmetric MTJs, not the electrode compositions. On the contrary, annealed devices show opposite $dI/dV$ and TMR bias dependence while we exchange electrode compositions. These annealed transport behaviors point that the electronic structures in the asymmetric MTJs manifest after annealing and dominates both the spin-dependent and electronic transport in asymmetric MTJs.

We suspect that the strong peak in the minority density of states in bcc Fe [4] is responsible for the phenomenon. The strong peak in the minority DOS would induce the increasing in the $dI/dV$ in the positive bias and also lower the spin-polarization with high bias causing the asymmetric TMR bias dependence. In addition, we utilize spin-torque ferromagnetic resonance (ST-FMR) to examine the spin-torque effect in the asymmetric MTJs. ST-FMR results exhibit opposite anti-symmetric Lorentzian while we reverse the electrode compositions in MTJs. Anti-symmetric Lorentzian component in the FMR curve is proportional to field-like torque (derivative of torque with respect to voltage). Opposite anti-symmetric Lorentzian component points to sign difference in the field-like torque. This result further suggests highly asymmetric field-like torque under high voltages.

In conclusion, we have measured electronic transports, spin-dependent transports and spin-torque effects in asymmetric MTJs. We have found the asymmetry in the transport behavior is directly related to electrode chemical composition, instead of defects induced by sputtering processes in high TMR devices. Through ST-FMR measurement and switching phase diagrams, we found the field-like torque is highly sensitive to electronic structures in the magnetic electrode materials. Our results prove the concept that the field-like torque can be altered and controlled by the electrode compositions and possibly can be used to reduce the unreliable switching in the in-plane MTJs.

References:
Photocurrent Measurements of Supercollision Cooling in Graphene P-N Junction Devices

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Abstract:
We measure the hot electron cooling rate in graphene by using the photothermal effect to extract the temperature \( T(t) \) of a dynamically cooling electron gas near the Fermi level. We find the photothermal current \( 3 \) generated in a graphene p-n junction is well described by the energy dissipation rate \( C dT/dt = -A(T^3 - T_b^3) \), where the heat capacity is \( C = \alpha T \) and \( T_b \) is the base lattice temperature. These results are in disagreement with predictions of electron-phonon emission in a disorder-free graphene system, but in excellent quantitative agreement with recent predictions of a disorder-enhanced supercollision (SC) cooling mechanism. With this new electron cooling mechanism established, we can accurately determine the hot electron temperature in graphene and graphene devices.

Summary of Research:
With uniform broad spectral coverage, fast response and high carrier mobility, graphene-based p-n junctions have the potential to be a defining optoelectronic material for next-generation photodetectors, bolometers and plasmonic devices [1]. Graphene is a gapless material, inherently making efficient photocurrent (PC) production challenging. The optimal PC production conditions require that (1) the electron escape time from the p-n junction is fast, (2) the hot electron cooling rate is slow, and (3) the electrons at the junction are hot relative to device temperature [2]. In this work we measure aspects of all three requirements, and establish a fundamental mechanism for hot electron cooling in graphene and graphene devices.

Graphene hot electron cooling by acoustic phonon emission has been predicted to be very slow, with timescales exceeding >300 ps [3]. In Figure 1 (left) we illustrate how the energy-momentum relation of the acoustic phonons, restricts phonon emission energies to small energies < 4 meV. The slow cascade of acoustic phonons for energy dissipation creates a major cooling bottleneck. Alternatively, Song, et al., predicts that impurities and lattice recoil effectively relax the momentum conservation constraint, resulting in a more rapid energy relaxation; they call this process supercollision (SC) cooling (see Figure 1 (right)) [4]. Compared to the disorder-free acoustic model, the energy dissipated in a SC acoustic emission is many orders of magnitude larger, resulting in enhanced cooling efficiency with rates orders of magnitude faster (~ 1 – 10 ps) [4]. To date, however, this theory has not been tested.

To begin, we fabricated p-n junctions from large-grain graphene grown by the CVD method and obtained a device carrier mobility of ~ 8000 cm^2/V·s [5]. A tuneable back gate \((BG)\) and top gate \((TG)\) couple electrostatically to graphene, and define two p-n junctions where the PC production is maximal. We optically excite the graphene p-n junction region with 180 fs pulses produced by two independently-tuneable oscillators plus near-IR optical parametric oscillator (OPO).
The collected PC amplitude is plotted as the laser is raster-scanned over the p-n junction (see Figure 2). We collect the electrical current generated ($\Delta Q_{12}(t)$) from the graphene p-n junction as a function of laser-pulse delay time ($t_d$, see Figures 2 and 3). We then combine this time-resolved photocurrent with the photothermal effect to directly calculate the hot electron temperature at the graphene p-n junction.

Figure 3 plots time-resolved PC curves for different base lattice temperatures at constant incident laser photon flux of $1.1 \times 10^{14}$ photons/cm$^2$. Upon warming the lattice to room temperature, the amplitude of PC signal shrinks by a factor of ~ 3, and the kinetic decay exhibits a dramatic shift toward a rapidly decaying exponential function.

To compare with theory, we solve the SC model using our extracted cooling rate. With no adjustable parameters, the SC model accurately predicts in Figure 3 both the (i) amplitudes, and (ii) strongly varying functional decay observed for lattice temperatures ranging from 10 to 295 K. We conclude that the SC electron gas heat loss rate, $H_{SC} = A(T^3 - T_l^3)$, describes electron cooling in graphene over a wide range of electron (10-3000 K) and lattice (10-295 K) temperatures.

At low lattice temperatures, the associated hot electron cooling time in the SC model is given simply by $\tau_e = 1.8 \text{ ns} / T_l$. These cooling times are much faster than those predicted by acoustic phonon emission but are in excellent agreement with disorder-assisted supercollision cooling. This work has broad implications for the use of graphene in terahertz plasmonic devices, photodetectors and bolometers.

References:

Manipulating Surface States in Topological Insulators

CNF Project Number: 598-96
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Abstract:

We have developed a nanofabrication procedure for integrating topological insulators into micro and nano-electronic devices. Starting with films of bismuth selenide grown by the Samarth group at The Pennsylvania State University, we can fabricate top-gated devices without ever exposing the fragile bismuth selenide layer to air. We have begun to characterize the electron transport in such samples using resistance and Hall measurements at temperatures down to 2 K. We are working to fabricate devices from topological insulator samples having carrier densities sufficiently low that the chemical potential can be tuned to the Dirac point using electrical gating.

Summary of Research:

Topological insulators are a recently discovered class of materials which are electrically insulating in the bulk, but have metallic surface states that traverse the band gap, shown schematically in Figure 1. In these states the spin of the electron is locked perpendicular to its momentum due to strong spin-orbit coupling. There is a growing number of proposed applications that make use of the unique properties of these surface states [1]. We are currently characterizing thin films of bismuth selenide as a first step to investigating possible device applications.

We have recently developed a nanofabrication process for creating top-gated Hall bar structures that allow us to investigate the properties of the surface states without ever exposing them to air. Starting with thin films of bismuth selenide grown on sapphire substrates with molecular beam epitaxy by the Samarth group at Penn State, we first remove a protective layer of amorphous selenium and replace it with alumina in situ. We then pattern and etch the bismuth selenide into micron-scale Hall bars with current and voltage leads. After etching, we etch through the alumina coating on the leads and deposit large metallic contact pads. We use atomic layer deposition to deposit hafnia over the surface of the entire sample which, along with the alumina, form the insulating layer for the top gate. Two final steps deposit a metallic top gate and remove hafnia from the contact pads. A finished device is shown in Figure 2.

We have characterized several devices using weak antilocalization and Hall effect measurements at temperatures down to 2 K. We are currently in the process of processing and characterizing a new set of lower-carrier density samples grown on indium phosphide.

References:

Figure 1: A schematic diagram of the band structure of a topological insulator.

Figure 2: A finished gated Hall effect device made from bismuth selenide grown on sapphire.

Figure 3: Channel conductance in one of our devices as a function of gate voltage and magnetic field at 2.2 K, with peaks at zero field associated with weak antilocalization.
Spin Injection into Graphene by the Spin Hall Effect

CNF Project Number: 598-96
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Abstract:
Graphene is a promising material for micron-scale spin transport because of its long spin-flip length and spin relaxation time. Our goal is to explore whether it is possible to use the spin Hall effect to inject spin current into graphene. We have fabricated devices from patterned graphene grown by chemical vapor deposition with thin (100 nm) Pt wires for spin current injection and ferromagnetic electrodes for spin detection.

Summary of Research:
One potential use of graphene is in the field of spintronics—the study of the control and manipulation of the spin degrees of freedom—where it might be employed as a spin conserving medium. Graphene has very weak intrinsic spin-orbit coupling and weak hyperfine interaction, so that its spin relaxation time $\tau_{sf}$ and spin-flip length $\lambda_{sf}$ are predicted to be long, up to 10 µs and 100 µm, respectively [1]. Spin injection and spin precession have been observed in graphene on silicon oxide with the use of ferromagnetic electrodes, experimentally obtaining $\tau_{sf} \sim 500$ ps and $\lambda_{sf} \sim 1$ µm [2, 3].

In this work, we propose to use the spin Hall effect in Pt [4] to inject spin current into graphene. As shown in Figure 1, in a material with strong spin-orbit coupling, the scattering of electrons is spin-dependent, such that a charge current generates a pure spin current in a perpendicular direction. Recent work has shown that in Pt the spin Hall angle $\Theta_{SH}$, the dimensionless ratio of the spin current density to the charge current density, is at least 0.06, and the spin current generated can be used to switch an adjacent magnetic layer [5].

In our device geometry, shown in Figure 2, a Pt wire is in direct contact with the graphene, while a ferromagnetic detector electrode is coupled to the graphene through a thin oxide layer. A charge current driven through the Pt wire in the longitudinal direction generates spin current in perpendicular directions. Because of the smaller density of states in graphene, most of

Figure 1: Spin Hall Effect – Charge current generates spin current in a perpendicular direction.
Figure 2: Spin injection/detection scheme.
the spin current generated by the SHE will actually not be transmitted into the graphene but instead builds up a spin-dependent chemical potential at the interface, associated with a backflow of spin current. The spin-dependent chemical potentials must be continuous across an Ohmic contact between the Pt and the graphene, which means that there should also be spin imbalance and a spin current in the graphene, decreasing away from the interface with a decay length of $\lambda_{sf}$. At the ferromagnetic Py detector placed 0.5-1.0 $\mu$m away from the Pt wire, a spin voltage should be detected that is proportional to the spin imbalance in the graphene.

Device Fabrication (Figure 3): We grow single layer graphene on copper foil using the First Nano furnace. After a one-hour anneal in hydrogen gas at 1000°C, we flow methane gas for 20 minutes in the growth step. We spin PMMA on the graphene-covered foil as a support layer and etch away the copper in ferric chloride. The PMMA membrane in water is then ready for transfer to a substrate. We use optical microscopy, Raman spectroscopy, and atomic force microscopy to verify the single layer nature of the graphene. To fabricate the device, we first define 100 nm wide Pt wires on an oxidized silicon substrate with electron-beam lithography (JEOL6300), metal evaporation, and lift-off. We transfer graphene onto the substrate and pattern it with photolithography (ABM) and oxygen plasma etching. A second aligned electron-beam lithography step is performed to define ~ 100 nm wide spin detector electrodes, with a 2 nm thick $\text{Al}_2\text{O}_3$ tunnel barrier separating the graphene from the ferromagnetic Ni$_{0.8}$Fe$_{0.2}$ layer (60 nm). Finally, contact pads are defined by photolithography and evaporation/liftoff of 100 nm of Au.

On the final device (Figure 4), we have taken preliminary measurements of nonlocal resistance arising from spin injection through ferromagnetic electrodes. Spin Hall effect measurements are in progress.

References:
Probing Spin Transfer Torque in Magnetic Nanoparticles

CNF Project Number: 598-96
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Users: Chen Wang, Hsin-Wei Tseng, Alex R. Mellnik, Jennifer L. Grab

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Primary Source of Research Funding: Army Research Office
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Abstract:
We study spin dependent tunneling and spin transfer torque in CoFeB magnetic nanoparticles 1-3 nm in diameter embedded in MgO-based magnetic tunnel junctions. By making nano-pillar devices containing less than 100 nanoparticles and taking advantage of the Coulomb blockade effect, we are able to resolve the magnetoresistance associated with magnetic switching of single nanoparticles. We detect the first experimental evidence of spin transfer torque in magnetic nanoparticles by observing an asymmetry in the switching phase diagram of a single ferromagnetic nanoparticle.

Summary of Research:
Spin transfer torque, a consequence of spin angular momentum exchange between a spin-polarized current and a nanoscale magnet, has been intensively studied over the past 15 years for both fundamental interest in spin transport and potential applications in magnetic storage industry. Most previous experiments have studied the effects of spin torque on lithographically-defined ferromagnets with sizes on the order of 100 nm × 100 nm × 3 nm, large enough to be treated as macroscopic objects in the context of electronic band structure and magnetization dynamics. There have been predictions that in very small electrically-isolated magnetic nanoparticles, only a couple of nanometers in size, spin transfer torque might display resonance enhancement due to quantized electronic states, and/or drive magnetic switching with very low current due to reduced magnetic damping. Here, we produce magnetic nanoparticle tunnel junction devices, aiming to study spin-polarized tunneling and the effects of spin transfer torque in magnetic nanoparticles.

To begin device fabrication, we deposit a thin film layer stack of (from bottom to top) Ta / Ru / Ta / IrMn / CoFeB / MgO / CoFeB nanoparticles / MgO / Ru / Ta / Ru onto an oxidized Si wafer by DC and RF magnetron sputtering under an in-plane magnetic field of about 200 Oe. The nanoparticles are formed due to a self-assembly mechanism when we sputter a very thin layer of CoFeB film (0.4-0.6 nm) onto MgO. The thin layer of CoFeB particles is sandwiched by MgO on both sides to act as tunnel barriers. We use electron-beam lithography and ion beam etching to pattern the whole layer stack into nano-pillar devices smaller than 50 nm × 50 nm in lateral dimension (Figure 1). We can apply tunneling current between the top electrode and bottom electrode through the double MgO barriers via nanoparticles.
Figure 2 shows hysteresis loops of resistance vs. external magnetic field applied along the direction of the exchange bias (acting on the bottom CoFeB layer from IrMn and set by the growth field) at selected temperatures for a typical device. The resistance change is due to the tunneling magnetoresistance (TMR) between the bottom CoFeB electrode and the CoFeB nanoparticles embedded between the two MgO layers. For all temperatures, there are two sharp switching edges for the resistance, corresponding to switching of bottom CoFeB electrode. One switching happens at close to zero field, and the other at substantially negative field, reflecting the added coercivity and the exchange bias from the IrMn layer. At higher temperatures such as 150 K, with increasing external magnetic field the nanoparticles become increasingly aligned with the direction of the field, resulting in smooth tails on the $R$ vs. $H$ data.

However, at low temperature (below 10 K), discrete hysteretic jumps of the resistance (in addition to the electrode switchings) start to appear, which correspond to switchings of single magnetic nanoparticles. Our tunnel junction device has about 75 nanoparticles connected in parallel by our estimate. However, the Coulomb blockade effect blocks the majority of the particles from contributing to the low-voltage charge transport, reducing the effective number of particles in the device and allowing us to resolve the switching of single particles.

Measurements such as shown in Figure 2 allow us to determine the magnetic stability of the nanoparticles at low temperature with essentially no effect from electrical transport. (Only a very small current is needed for the resistance measurement.) Then we apply electrical voltage pulses of various amplitudes, $V$, under various magnetic fields $H$, and test whether a particular magnetic nanoparticle switches its magnetization. Such a switching phase boundary of $H$ vs. $V$ for 4 ns pulses is shown in Figure 3.

We see that the magnetic field range where the nanoparticle has bi-stable states (the difference between the upper switching field and lower switching field) is dramatically reduced at high bias, due to heating from the voltage pulse. At the same time, we note that the phase boundary is not symmetric for positive and negative bias, with the center field of the bi-stable range higher for negative bias than for positive bias. This asymmetry of switching phase diagram is an indication of the spin transfer torque effect.

We can use a thermally-assisted spin-torque switching model to make a rough estimate that the magnitude of the spin transfer torque is about 20% of $(\hbar/2)\sin\theta$ per electron, assuming the magnetic damping of the nanoparticle is 0.012 (similar to bulk CoFeB). This torque is the order of magnitude one would expect from the simplest spin torque model.

Therefore our first experimental evidence of spin torque in nanoparticles does not reveal any result substantially different from in a bulk ferromagnet. However, we note that the strong heating inherent to our high power pulse measurement most likely destroys any quantum effects that might be present at low temperature. Our next step is to perform spin-transfer-driven ferromagnetic resonance (ST-FMR) measurements on these magnetic nanoparticle devices to probe their response at lower voltages.
X-Ray Imaging of Magnetic Normal Modes Driven by Spin Transfer Torque in Magnetic Nanopillar Devices

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Abstract:
We have fabricated Py/Cu/CoFe magnetic nanopillars with 100 × 250 nm² elliptical cross sections on top of suspended silicon nitride membranes, designed for performing x-ray transmission microscopy of magnetization dynamics. We are now studying these devices to image the magnetic normal modes that are excited by the spin transfer torque from a microwave-frequency current passing through the nanopillar.

Summary of Research:
A spin-polarized current passing through a ferromagnet can exert a torque on the ferromagnet that is strong enough to either reverse its magnetization or excite steady-state high-frequency (1-20 GHz) magnetic precession, depending on the device structure and applied magnetic field [1]. This spin-torque effect is under intensive study both because of its fundamental importance and because of potential applications in magnetic random access memory (MRAM) and high speed signal processing. One of the critical outstanding issues in this field is the nature of the magnetization dynamics excited by the spin torque. Imaging [2, 3] of the spatial and temporal dependence of the magnetic dynamic modes with high resolution x-ray transmission microscopy can offer a direct view of any spatial inhomogeneities. X-ray transmission microscopy can selectively image a ferromagnetic element via x-ray magnetic circular dichroism (XMCD), by which the transmission of x-ray photons through a ferromagnetic material depends on the angle between the polarization of the photons and the magnetization.

We start device fabrication with a Si substrate coated with 150 nm of low stress (film tension < 200 MPa) silicon nitride films on both sides, one of which will be made into suspended membranes to allow for x-ray transmission microscopy (Figure 1). We deposit the metallic multilayers of the structure (in nm): {Ta 3 / CuN 20 }₂ / Ta 3 / Cu 2 / Py 15 / Cu 4 / Co₉₀Fe₁₀ / Cu 2 / Pt 20 (where Py is the magnetic alloy permalloy, Ni₈₁Fe₁₉) in a sputtering system. E-beam lithography and ion milling are then used to define pillars with elliptical cross section of 100 × 250 nm² (Figure 1). Multiple steps of photolithography and ion milling are used to make the contact leads, and silicon oxide is deposited by electron-beam evaporation to provide electrical isolation between top and bottom leads. To form the suspended nitride membrane, the Si substrate is etched anisotropically in heated KOH solution, using photolithographically-patterned silicon nitride on the back side of the substrate as the etch mask.

The XMCD experiments are carried out at the scanning transmission x-ray microscope (STXM) at the Advanced Light Source of Lawrence Berkeley National Laboratory, which has a spatial resolution of 15 nm and temporal resolution of 70 ps. The x-ray is incident at 30° from the normal direction of the device plane (Figure 1), and its energy is tuned to the characteristic Co L₃ resonance absorption edge. These experiment conditions allow detection of the in-plane component of the magnetization of the 5 nm thin CoFe layer in the nanopillar. A magnetic field is applied in the device plane along the short axis of the ellipse.

Figure 1: Device schematic: nanopillar device on top of a suspended silicon nitride membrane.
Figure 2 shows the electrical characterization of the spin-transfer-driven ferromagnetic resonance [4] (ST-FMR) spectrum of one sample as a function of microwave current frequency, \( f \), and magnetic field, \( H \). For x-ray studies, we excite the sample into a particular resonance mode using appropriate values of \( f \) and \( H \) (for example, point A at about 3 GHz and -20 Oe in Figure 2), and use the x-ray microscope to image the spatial configuration of the magnetization at eight different phases evenly spaced throughout one oscillation cycle. Magnetic contrast images are shown in Figure 3 relative to the average of all phases. The contrast images suggest the presence of both an approximately uniform center mode and a nonuniform edge mode — the edge precesses at a different phase and amplitude than the center of the device. We are currently working on accumulating sufficient data from different devices, analyzing and modeling the magnetic normal modes, and comparing with micromagnetic simulations to understand the mode structures.

References:
Graphene in the Third Dimension

CNF Project Number: 900-00
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Abstract:
We have developed a new technique for interacting with single-layer graphene in the third dimension. Using mechanical and optical probes in a liquid environment, we can pick up a graphene sheet and fold it at high angles. We are able to precisely image the shape of the sheet in three dimensions, and can look at graphene on-edge using optical techniques for the first time. These techniques let us extract the bending stiffness by observing the graphene’s thermal motion at room temperature. Finally, we demonstrate graphene’s potential as a monolayer sheet hinge for applications such as drug delivery boxes.

Summary of Research:
Our understanding of graphene’s mechanical properties is limited by the fact that experiments so far have been carried out on flat sheets or at small deviations from flat \cite{1, 2}. To address this question of how an atomically-thin membrane will behave under significant distortions, we need a number of tools: control over the graphene’s adhesion to its substrate, a means of controlling its folding, and a way of imaging the sheet.

We begin with monolayer graphene grown by chemical vapor deposition (CVD), which is lithographically patterned and transferred to fused silica covered with an aluminum release layer. We add 40 nm-thick gold pads to the end of the graphene strips, creating what we will refer to as a dumbbell. The aluminum is etched using a mild hydrochloric acid solution, and—without drying out the chip—we cover the completed devices with a droplet of deionized water and surfactant, which will become our working medium. Our experimental setup (Figure 1) consists of an inverted optical microscope and a number of manipulators: laser light (1064 nm, 200 mW CW), a micromanipulator-controlled probe arm, and even the possibility of future optical or magnetic tweezers.

We have a number of options for controlling the graphene’s motion. The first is our IR laser; when it strikes a gold pad, a convective volcano of higher-temperature water is created above its surface, and the flow alone is often enough to kick the pad and its attached graphene up into the solution. As shown in Figure 2, we strike a short dumbbell with our IR laser, using the convective flow to flip up one gold panel. This simple result has exciting implications. Resilient, easy-to-fabricate nanoscale hinges are needed for applications such as microcapsules for drug delivery boxes.
delivery, but existing hinges are difficult to fabricate and face size limitations [3, 4]. A simple sheet hinge would solve many of these problems, but polymers tear before they can be made sufficiently thin. Graphene’s impressive in-plane strength and low bending energy solves these problems, and the existence of carbon nanotubes suggest that graphene hinges could be scaled down to the truly nanoscale regime. Our results demonstrate graphene’s use as an ideal sheet hinge, in which a single layer of atoms is strong enough to hold panels together yet could be easily scaled down by orders of magnitude.

More control over the graphene motion comes from a probe attached to a computer-controlled micromanipulator. The probe can be pushed against one gold pad to move the graphene as it rests on the surface. Figure 3 shows the repeatable creation of wrinkles in the graphene by pushing one gold pad towards the other and then pulling it back. We can also use the probe to pick up the graphene/gold dumbbell, and the addition of a stepper motor allows us to rotate the probe and image the graphene optically from the side for the first time (Figure 4(a)). This orientation also highlights the thermal motion of the graphene sheet, which lets us extract the bending stiffness of the material—a value that, to our knowledge, has never been directly measured for graphene. Our preliminary measurements suggest that wrinkles significantly affect the sheet’s stiffness, leading to significant variation between visibly wrinkled and flat graphene dumbbells.

The apparent importance of the graphene sheet’s shape in its mechanical behavior underlines the importance of developing a rigorous method of imaging the three-dimensional sheet shape. We achieved this using a regular array of polymer dots (SU-8), as in Figure 4(b). The short focal plane of the lens offers information about motion in the out-of-plane direction, meaning that we can extract quantitative tracking information for the complete graphene sheet shape as it folds. Furthermore, SU-8 is a highly optically-trappable material, and our laser is designed to support optical tweezer studies, so in the near future we may be able to exert force directly on the sheet through SU-8 “handles.”

In the quest for greater control over graphene motion, we hope to expand these studies using more controlled probes, including optical traps and—by replacing the gold pads with magnetic elements—magnetic tweezers. Already our approach offers a unique set of tools for exploring the mechanical properties of graphene in the third dimension, and has given us an unusual ability to interact intuitively with an atom-thick material. And finally, these techniques demonstrate the feasibility of graphene for use as a hinge, contributing its unparalleled mechanical properties and atomic size scales to create the world’s only nanoscale sheet hinge.

References:
Vortex Dynamics in Nanofabricated Superconducting Devices

CNF Project Number: 1314-05
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Abstract:
We are fabricating superconducting devices for controlling the dynamics of vortices with experiments ranging from DC measurements up to microwave frequencies. Vortices are quantized bundles of magnetic flux that thread many different superconductors over a particular range of applied magnetic field. These measurements are useful for probing fundamental physical properties of vortices and for developing devices based on the controlled motion of magnetic flux in superconductors, including vortex ratchets. In addition, trapped vortices are an important loss mechanism that can limit the performance of superconducting microwave detectors and quantum coherent circuits.

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, it is possible to control the vortex dynamics to probe such phenomena as commensurability and ratchet effects [1]. Nanofabricated devices are also useful for probing the microwave response of vortices in superconductors. Vortices trapped in superconducting traces can result in substantial reductions in the quality factor of microwave resonators. Thus, understanding and controlling this dissipation mechanism can be important in the design of superconducting systems that use microwave resonators, including sensitive photon detectors and quantum computing applications.

We are fabricating a system of superconducting, thin-film microwave resonators for studying the loss contributed by trapped flux over the frequency range from 2-11 GHz [2]. By cooling the resonators indifferent magnetic fields, we are able to probe the loss from vortices as a function of field at the resonance frequencies contained in our design. We are currently developing resonator layouts optimized for trapping and detecting only a few vortices [3] for probing the interactions between a single vortex and a single microwave photon.

We have also employed a technique developed at Leiden University for controlling vortex confinement [4], where we fabricate devices with narrow thin-film channels with weak vortex 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 have fabricated similar weak-pinning channels but with various constrictions of the channel walls for controlling the potential energy landscape experienced by vortices in the channels. We have studied the behavior of vortex ratchets formed from such structures arranged in a circular Corbino geometry on a superconducting disk [5].

We fabricate our microwave resonators from various superconducting films, including aluminum deposited onto sapphire or silicon wafers in our electron-beam evaporator at Syracuse University. We define the patterns on the Autostep 200 and transfer them into the films with reactive ion etching. We measure these circuits at temperatures down to 50 mK in our lab at Syracuse University. We fabricate our weak-pinning 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 CF4. We measure these channel devices in cryogenic systems that we have constructed in our lab at Syracuse University.
References:


Melting Dynamics of Colloidal Crystals on Patterned Surfaces

CNF Project Number: 1361-05
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Abstract:
The melting dynamics of colloidal crystals are studied in the presence of patterned substrates created using electron-beam lithography. Patterned substrates allow for the control of surface symmetry and lattice constant in an otherwise “hard-sphere” system. Crystalline islands are self-assembled on the patterned substrates and then melted. The underlying substrate symmetry and strain are found to alter both growth and melting dynamics, including the diffusion of adatoms on growing islands, sub-diffusion in lattice interstitials, and the melting rate of individual islands.

Summary of Research:
Microfabricated templates have been successfully used to direct the growth of colloidal particles into self-assembled structures [1]. 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]. Recently we have used this technique to report the diffusive nature of step-edge barriers and island growth in epitaxially grown colloidal crystals [4].

In our current research project, we construct a 10,000 sq. micron grid consisting of 1.0 μm holes placed 500 nm deep into glass. A mixture of 1.0 μm charge-stabilized polystyrene colloidal particles and a nonionic surfactant is sedimented onto the patterned surface. The holes trap a monolayer of particles, forcing them to assume the underlying symmetry, while the surfactant serves to introduce an attractive force between the colloids. A single crystalline layer directly above the trapped layer is studied using an inverted microscope. The dynamics of crystallization of this layer of colloidal particles is directly observed with single particle resolution in real time.

Substrate fabrication utilizes electron-beam lithography to pattern periodic arrays of 1.0 μm holes in a 500 nm thick layer of polymethylmethacrylate (PMMA) with a center-to-center distance of 1.050 μm (Figure 1). This pattern is transferred into the glass via a chromium mask and reactive ion etch. A square lattice of micron-size holes is used to grow a face-centered cubic crystal along the <100> face, while a triangular lattice can be used to explore the <111> face. In combination with a temperature dependent attractive interaction [5], we are able to observe the dynamics of both island growth and melting. An initial layer of colloidal particles self-assemble into the holes due to the surfactant-induced attraction between particles and between particles and the substrate (Figure 2). Island growth occurs atop the initial layer of colloidal particles with the same symmetry as the underlying lattice. A small change in temperature allows us to weaken the attractive forces in the system, causing the islands to melt. Because of the geometric constraints that the lattice imposes on the diffusion of particles, the rate of melting is found to vary dramatically depending on the underlying lattice symmetry (Figure 4).

References:
Figure 1: SEM of 1.0 µm holes written into PMMA on a glass cover-slip patterning using electron-beam lithography. The features between the holes are unexposed PMMA with a width of 50 nm. The upper-left corner of the image displays the FCC [111] (triangular) configuration of holes, while the lower-right corner features the FCC[100] (square) array.

Figure 2: Optical image of the self-assembly of two layers of 1.0 µm polystyrene particles on a triangularly patterned substrate. The white (in-focus) particles are particles that reside on the second layer of this crystal, which are diffusing atop the lattice of black (out-of-focus) particles. The black particles reside in the holes in the patterned substrate shown in Figure 1. The patterned holes are visible on the top-left and bottom corners of the figure, and appear as a faint “third layer.”

Figure 3: Plots of island size versus time for colloidal particles crystallized on a patterned substrate. Comparisons between lattice symmetries show that the melting rate on a square face is about three times slower than that on a triangular face.
Electrical Transport Across Individual Grain Boundaries in Polycrystalline Graphene

CNF Project Number: 1486-06
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Abstract:
Graphene’s exceptional physical properties stem directly from the symmetries of its crystalline structure. However, large-area graphene produced by chemical vapor deposition (CVD) is polycrystalline, and so the presence of grain boundaries will manifest in the film’s electronic properties. We have devised a method to electrically address individual grain boundaries in CVD graphene, exploiting the large-scale structural imaging of transmission electron microscopy (TEM). We find electrical transport across grain boundaries to be directly correlated with the quality of their connectivity.

Summary of Research:
Recently, it has been shown using dark-field TEM imaging that graphene films grown by CVD consist of many interconnected crystal grains [1, 2]. Grain boundaries are defect structures that may impede the flow of electric current, but their precise electronic properties were not well understood. We have fabricated special TEM chips that are compatible with electrical measurements (Figure 1A) to study the electronic transport properties of grain boundaries in CVD graphene [3]. After transferring graphene and imaging the grain morphology with dark-field TEM, we used electron-beam lithography to pattern devices consisting of individual grain boundaries. In Figure 1B, we show a scanning electron micrograph of one such device overlaid with a false-color dark-field TEM image of the underlying graphene. The areas with faded colors represent graphene that was subsequently etched away. From such a device geometry, we were able to determine the absolute resistivity of grain boundary. We found that grain boundary resistivity was not a constant value, but strongly dependent on carrier density. In Figure 1C, we plotted this resistivity at both the Dirac point and at p-type doping across eleven devices from two growths. Overall, the grain boundaries from the “fast” growth were an order of magnitude more conductive than those from the “slow” growth. This result can be understood from closer examination of the structure of grain boundaries produced from these two growth methods. While fast growth grain boundaries form seamless connections within the instrument resolution of dark-field TEM (Figure 1C), grain boundaries from the slow growth exhibit a stark crystalline discontinuity, which could suggest the incorporation of amorphous material that additionally impedes electrical transport. The results of our findings demonstrated that the quality of connection between graphene grains are just as important as the size of grains in determining the overall electronic performance of CVD graphene films.

References:
Figure 1: A. Schematic of TEM chip compatible with electrical measurements. B. Overlaid scanning electron micrograph and dark-field TEM images showing device crossing a single grain boundary. Scale bar, 1 µm. C. Grain boundary resistivities (at Dirac point and p-type doping) across eleven devices from fast and slow growths. D. Dark-field TEM images of grain boundaries from fast and slow growths show better connectivity from the fast growth. Scale bar, 100 nm.
Quantum-Limited Measurement and Entanglement in Superconducting Circuits

CNF Project Number: 1577-07
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Abstract:
An outstanding goal in condensed-matter physics is the generation, manipulation, and high-fidelity readout of coherent quantum states in micro- and nanofabricated circuits such as superconducting circuits based on Josephson junctions. These systems can be viewed as “artificial atoms,” with energy levels that are tunable over a broad range on nanosecond timescales. The circuits can be made to couple strongly both to one another and to microwave photons. Thus, they form an ideal test bed for the exploration of fundamental quantum concepts. We are investigating the physics that governs decoherence in these circuits and developing techniques for measurement approaching the quantum limit.

Summary of Research:
Superconducting quantum circuits incorporating Josephson junctions are a leading candidate for scalable quantum information processing in the solid state. Despite recent advances, the microscopic physics that governs decoherence of the quantum state is incompletely understood. In the case of the Josephson phase and flux qubits, dephasing is due to a low frequency magnetic flux noise with 1/f spectrum and magnitude at 1 Hz around 1 µΦ₀/Hz⁰. Our recent experiments suggest that this noise is due to a high density of unpaired electron spins on the surfaces of the superconducting films that are used to realize the qubit. We are conducting additional experiments to understand the physics that drives spin fluctuations, and we are exploring novel surface treatments to realize superconducting detectors and qubits with reduced noise.

At the same time, accurate characterization of entanglement in solid-state quantum circuits demands measurement capabilities approaching quantum-limited performance in the microwave regime. We are investigating novel nanofabrication and device techniques to realize superconducting amplifiers with noise performance approaching the quantum limit. These devices will be used in the full tomographic characterization of solid-state qubit circuits. In addition, we are developing a Josephson photon counter, a microwave frequency analog of the avalanche photodiode: absorption of a single microwave photon causes the junction to switch to the voltage state, producing a large, readily measured classical signal.

We are using the CNF to fabricate reticles that are needed for the preparation of thin-film superconducting devices at the Wisconsin Center for Applied Microelectronics. Superconducting aluminum and niobium thin films are grown by sputter deposition, while dielectric films are grown by plasma-enhanced chemical vapor deposition. The films are patterned photolithographically and etched with chlorine- and fluorine-based reactive ion etching. Device characterization is performed at millikelvin temperatures in our laboratories at the University of Wisconsin. In addition, graduate student Guilhem Ribeill spent one week at the CNF working with collaborators in the Plourde group (Syracuse University) to develop superconducting qubits and amplifiers based on submicron Josephson junctions.
Figure 1: Multiplexed Josephson junction photon counter.

Figure 2: Ultrasound noise microwave amplifier based on the superconducting low-inductance undulatory galvanometer (SLUG).

Figure 3: High fidelity Josephson phase qubit incorporating a crystalline silicon shunt capacitor.
Investigation of Charge Transport in Quantum Dot Solids

CNF Project Number: 1645-08
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Primary Source of Research Funding: US Department of Energy
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Abstract:
Lead-selenide nanoparticle (NP) films are being investigated as a low-cost, potentially highly efficient material for photovoltaics. Although very efficient photon absorbers, devices made with these films suffer from charge transport limitations. Identification of the charge transport limitation is a hurdle in the advancement of NP based solar cells. The notion of a NP as an artificial atom has driven an effort to demonstrate electronic band formation due to stronger NP interaction. We present temperature dependent mobility measurements of PbSe NP films and discuss the origin of the negative temperature coefficient.

Summary of Research:
Semiconducting quantum dots are a novel material for optoelectronic applications such as light emitting diodes, photodetectors and solar cells. By reducing the size of semiconductor crystals into the nanometer scale, basic material properties can be tuned due to quantum effects. For solar cell applications this could allow for materials tuned to absorb the solar spectrum more efficiently. Quantum dots are also attractive due to the ability to fabricate devices using inexpensive techniques such as spin casting or inkjet printing [1].

The electrical properties of quantum dot solids vary widely due to the exponential nature of the wavefunction overlap between particles. This is strongly influenced by the particle surface chemistry, as illustrated by Figure 1. A small change in surface molecule length can result in orders of magnitude change in conductivity [2]. Because of the large variability and potentially high resistance of quantum dot films, it is necessary to limit the sample scale in order to generate measurable current at reasonable voltages. To this end we have fabricated field effect transistor (FET) devices with various configurations to use as test substrates.

Figure 2 shows a common device geometry. After fabrication of the device in the cleanroom, quantum dots are deposited by spin casting, dip coating, drag coating, or drop casting. The FET geometry allows measurement of conductivity, contact resistance, and field effect mobility. These quantities serve as the metric by which new quantum dot solids are evaluated. The FET devices we fabricate allow us to rapidly investigate the effect of new chemical or physical treatments on quantum dot solids and drives the effort to create better performing materials for solar energy applications.

References:
Figure 1: The influence of nanoparticle surface chemistry on conductivity. Conductivity and mobility decrease with ligand length.

Figure 2: Device structure used to measure data shown in Figure 1.
Perpendicular Magnetic Anisotropy in Ultrathin Co|Ni Multilayer Films Studied with Ferromagnetic Resonance and Magnetic XRM

CNF Project Number: 1673-08
Principal Investigator: Andrew D. Kent
User: Ferran Macià

(in collaboration with P. Warnicke, M.-Y. Im, P. Fischer, and D. A. Arena)

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Primary Source of Research Funding: ARO-MURI Grant No. W911NF-08-1-0317
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Abstract:
We have combined macroscopic and microscopic analytical techniques to understand magnetic properties of ultrathin Co and Ni multilayered magnetic films. Our approach combines ferromagnetic resonance (FMR), element specific hysteresis loops (ESHL) and high resolution x-ray microscopy (XMCD). FMR provides a quantitative measurement of the anisotropy fields, ESHL determines the coercive fields and the evolution of macroscopic magnetization and XMCD imaging provides a microscopic measurement of the magnetic domain structures as a function of field. For the XMCD studies we have fabricated silicon nitride windows at CNF and deposited our magnetic films on these windows. We have systematically studied three samples with different numbers of bilayer repeats.

Summary of Research:
We have deposited Co-Ni multilayers, capped with Pt, on a permalloy (Py)-Cu base layer by evaporation in an ultra high vacuum chamber. The samples, with a complete layer stack |3Ta|50Cu|3Ta|10Py|10Cu|0.2 Co|0.6Ni|n|0.2Co|5Pt| (thicknesses in nanometers), were deposited on silicon nitride (Si3N4) membranes supported by Si substrate frames to allow for x-ray transmission. To fabricate the silicon nitride membranes we began with double side polished silicon wafers and grew 100 nm of amorphous low-stress silicon nitride on both sides. The wafer were lithographically patterned and a number of small areas were etched away until obtaining silicon nitride membranes; first the silicon nitride was etched with reactive ion etch (RIE) and later on the silicon was etched with chemical etching with potassium hydroxide (KOH). The thickness of the Cu spacer layer (10 nm) separating the two magnetic layers (Py and CoNi), was chosen to magnetically decouple the in-plane magnetized Py layer from the out-of-plane magnetized CoNi multilayer. A CoPt capping layer was used to further enhance the interface-induced perpendicular magnetic anisotropy of the CoNi multilayer. To address the influence of the number of bilayer repeats, three different samples with n = 4, 6, and 8, repeats were studied.

In order to determine the magnetic anisotropy of the films, we conducted FMR spectroscopy with frequencies up to 40 GHz as a function of the applied field at room temperature. To record the weak signals of the ultrathin magnetic layers we used a coplanar waveguide (CPW) with a smooth signal transmission within this frequency range. The absorption signal was recorded as a function of both field and frequency with the sample mounted flip-chip on the CPW.

Figure 1 shows the frequency dependence \( f \) of the resonant field \( H_r \) for a configuration of an applied field perpendicular to the film plane. Inset shows a schematic of the sample layer stack and the magnetic moments orientations of Co|Ni and Py layers. (See full color version on inside front cover.)
to the film plane, as shown in the inset of the same figure. We have measured the perpendicular anisotropies of the three films: $K \equiv 3.89 \pm 0.05 \times 10^5 \text{J/m}^2$, $4.14 \pm 0.03 \times 10^5 \text{J/m}^2$, and $3.42 \pm 0.02 \times 10^5 \text{J/m}^2$ for $n = 4, 6, \text{and 8}$ respectively.

To understand better the microscopic origin of the perpendicular magnetic anisotropy in these systems, we measured XMCD spectroscopy and element-specific hysteresis loops at beamline U4B at the National Synchrotron Light Source (NSLS). Element-specific hysteresis loops shown in Figure 2 (b) and (c) were measured by tuning the photon energy to the $L_3$ edges of Co, Ni, and Fe and sweeping the out-of-plane magnetic field from an initial value of +700 mT (substantially larger than the saturation field of $\sim$ 100 mT). The loops for Co and Fe isolate the magnetic contributions from the Co|Ni and Py layers, respectively, while loops for the Ni (not shown) display a composite picture with contributions from both the Py and Co|Ni layers. As expected, the Fe hysteresis loops show a hard axis behavior consistent with the in-plane anisotropy of the Py layer. The Co hysteresis loops and the coercive fields, $H_c$, correlate well with the anisotropies determined from our FMR measurements.

We performed magnetic transmission soft x-ray microscopy measurements at beamline 6.1.2 (XM-1) at the Advanced Light Source in Berkeley. X-ray images with a spatial resolution of better than 25 nm were obtained at the Co $L_3$ absorption edge. Figure 3 shows the evolution of the magnetic domain structures at various applied magnetic fields for the multilayered samples. It can be seen that the dominant magnetization reversal mechanism is governed by domain nucleation, growth, and annihilation. It is worth noting that the clear observation of the magnetic domain pattern down to an effective Co thickness of 1 nm demonstrates a high sensitivity of the XMCD contrast in full field soft x-ray microscopy.

To summarize, this study highlights the power of combining macroscopic and microscopic analytical techniques to understand the magnetic properties of ultrathin magnetic films. FMR provided a quantitative measurement of the anisotropy fields, whereas element specific hysteresis loops determined the coercive fields and the evolution of macroscopic magnetization. A link to the microscopic origin was obtained with high resolution XMCD microscopy allowing observation of the evolution of the domain structure as a function of the applied field and a measurement of the change in characteristic domain widths. We have shown sensitivity of x-ray microscopy for layers down to 1 nm effective thickness.

References:
Fabrication of Nanoscale Josephson Junctions for Quantum Coherent Superconducting Circuits

CNF Project Number: 1735-08
Principal Investigator: Britton L.T. Plourde
Users: Michael DeFeo, Haozhi Wang

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Abstract:
We fabricate nanoscale superconductor tunnel junctions for experiments involving quantum coherent 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 microwave radiation at low temperatures. To probe such circuits, we are developing low-noise superconducting amplifiers and novel readout techniques for measuring the qubit state.

Summary of Research:
The unique properties of nanoscale Josephson junctions enable a wide range of novel superconducting circuits for investigations in many diverse areas. In recent years, circuits composed of such junctions have emerged as promising candidates 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]. The quantum coherent properties of the circuits are measured at temperatures below 50 mK with manipulation of the qubit state through microwave excitation.

We are currently working on a variety of experiments involving these nanoscale superconducting junctions that will allow us to probe the nature of quantum entanglement between qubits and microwave photons. By incorporating these junctions into superconducting quantum interference devices (SQUIDs), we are working to develop low-noise linear amplifiers in the microwave range [2-4]. We are also fabricating lumped element superconducting oscillators for implementing novel techniques for reading out the state of a superconducting qubit [5].

Measurements of these circuits are performed in cryogenic systems at Syracuse University, including a custom dilution refrigerator for achieving temperatures of 30 mK.

References:
Figure 1: Optical micrograph of microstrip SQUID amplifier with Al washer and input coil.

Figure 2: Closeup optical micrograph of tunnel junction and resistive shunt region.

Figure 3: Scanning electron micrograph of submicron superconducting tunnel junctions.

Figure 4: Measurement of amplifier gain as a function of frequency with SQUID amplifier cooled to a temperature of 0.35 K. Inset shows maximum gain as a function of input power.
Superfluid Quantum Interference in Multiple-Turn Reciprocal Geometry

CNF Project Number: 1823-09  
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Abstract:
We have constructed a large-area superfluid matter wave interferometer consisting of a multiple-turn interfering path in reciprocal geometry. Here, we summarize our recent observation of superfluid quantum interference from such a device utilized as a sensitive inertial sensor.

Summary of Research:
In conventional interferometry devices, beams of light or atoms are split and recombined while enclosing an area A. Certain physical interactions give rise to a phase shift between the two beams, with the size of the shift proportional to A and the “mass” of the constituent particles (m for atoms and $\hbar \omega / c^2$ for photons). Because of the larger mass, a phase shift for atoms with the same interferometer area is typically $10^{10-11}$ times larger than it is for visible photons. This makes the matter-wave based interferometer a promising candidate for providing unprecedented sensitivities to external phase shifting influences. For example, when configured as a Sagnac-interferometry-based gyroscope, such a device could be used for rotation sensing with possible applications in geodesy, seismology and inertial navigation.

For precision measurements identified above, interferometer parameters become a compromise between the desire for the best resolution achievable and the reality of the noisy environment. Although increasing the area would improve their sensitivity, interferometers need to be as compact as possible to suppress phase shifts due to environmental noise. A larger apparatus would also limit the applications of the device. A natural solution to these rather contradictory requirements is to let the interfering path circulate around a small physical area multiple times. One can even envision counter-winding the path in reciprocal geometry. Such a system would allow common rejection of phase shifts due to static perturbations, essentially making the device a 1st order gradiometer.

Our apparatus is schematically represented in Figure 1. A small volume is bounded on one side by two arrays of nanoscale apertures (Figure 2) and the other side by a flexible diaphragm. The inside is filled with superfluid 4He, and we configure the apparatus to form a loop of superfluid (multiple turns in reciprocal geometry) interrupted by two weak link junctions. When a constant chemical potential difference is applied across the arrays, fluid within each array exhibits mass current oscillation at the so-called Josephson frequency. The combined oscillation amplitude from two arrays exhibits interference depending on the external phase shift introduced in the system. Figure 3 shows the experimentally observed interference pattern versus Sagnac phase shift induced by the Earth’s rotation. Further details can be found in Ref. [1].

References:
Figure 1: Experimental apparatus.

Figure 2: SEM image of aperture array. The array consists of $75 \times 75$ nominally 60 nm apertures spaced on two micron square lattice in a 60 nm thick silicon nitride membrane.

Figure 3: Observed interference pattern when the device is used as a gyroscope.
Electronic Structure Calculations for Cr$_{1-x}$Al$_x$

CNF Project Number: 1845-09
Principal Investigator: Frances Hellman$^{1,2}$
Users: Zoe Boekelheide$^{1,2,3}$, Derek Stewart$^4$

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4. Cornell NanoScale Science and Technology Facility, Cornell University

Primary Source of Research Funding: This work was supported by the US Department of Energy under Contract No. DE-AC02-05CH11231

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Abstract:
Cr$_3$Al shows semiconductor-like behavior, attributed to a combination of antiferromagnetism and chemical ordering. We present a detailed study of chemical ordering in Cr$_3$Al. Using density functional theory within the Korringa Kohn Rostoker (KKR) formalism, we consider five possible Cr$_3$Al structures. The chemically ordered, rhombohedrally distorted X-phase has the lowest energy and should therefore be the ground state found in nature. The D0$_3$ structure has the highest energy and should not occur. While KKR indicates a pseudogap in the X-phase density of states, calculations using full potential linear muffin tin orbital and plane wave techniques show a narrow band gap.

Summary of Research:
The nature of a gap in Cr$_3$Al is currently of interest. Experimental results in the literature are suggestive of either a semimetal or degenerate semiconductor [1]. A full gap might suggest further study of Cr$_3$Al as a potential thermoelectric material [2]. In addition, recent theoretical work has predicted that Cr$_3$Al with binary Heusler (D0$_3$) ordering is almost completely spin polarized and that Mn-doped D0$_3$ Cr$_3$Al would behave as a true half metal with applications in spintronics [3]. While no evidence of D0$_3$ ordering has been shown in Cr$_3$Al, the Cr-Al phase diagram is not well established [4]. Thus, a study of the nature of a gap in Cr$_3$Al and how it is affected by chemical ordering is warranted.

We will explore here the three structures shown on the proposed phase diagram for Cr$_3$Al: bcc solid solution, C11$_b$ Cr$_2$Al + bcc Cr two-phase system, and X-phase Cr$_3$Al structure [4]. Also included are an off-stoichiometric C11$_b$ Cr$_3$Al structure, seen experimentally for films grown at 500-600°C [5] and the D0$_3$ (Heusler) structure. These structures are shown in Figure 1.

DFT calculations were done on the Intel cluster at the Cornell NanoScale Facility. All structures were evaluated using the AkaiKKR code, so the results for the different structures could be compared directly. This technique was chosen for its ability to treat site disorder, which was necessary for the bcc solid solution and off-stoichiometric C11$_b$ Cr$_3$Al structures. Disorder is treated using the coherent potential approximation (CPA) [6].

The total energy per atom is shown in Table 1. The X-phase structure has the lowest energy, suggesting that it is the low temperature stable phase for the Cr$_3$Al stoichiometry, confirming the proposed phase diagram [4].

Figure 1: Structures considered.
The bcc solid solution, C11_Cr2Al + bcc Cr two phase system, and C11_Cr3Al structure have the next lowest energies, consistent with their being stable phases at higher temperatures. Finally, the D0_3 structure has a significantly higher energy.

The table also shows the density of states (DOS) at $E_F$. The lowest DOS at $E_F$, as calculated by KKR, occurs for X-phase Cr3Al due to barely overlapping bands at $E_F$ [5]. DFT calculations often underestimate the band gap in materials, sometimes showing overlapping bands in materials known to have a full band gap. Therefore, to complement the KKR calculations, we performed calculations using a plane wave approach (Quantum Espresso) and a full potential linear muffin tin orbital approach (FP-LMTO) [7, 8].

Figure 2 compares the calculated DOS for the three approaches. It is clear that, overall, the approaches produce similar features in the DOS near $E_F$. Quantum Espresso predicts a small but complete gap of 200 meV and FP-LMTO predicts a smaller gap of 40 meV.

Thus, the semiconductor-like behavior in Cr3Al can be explained by X-phase ordering leading to a gap in the DOS. It is likely that a perfect crystal of X-phase Cr3Al would be a true narrow-gap semiconductor with a band gap greater than 40 meV. However, real samples thus far show the X-phase occurring in very small domains presumably separated by antiphase boundaries [9]. Disorder could smear the band edges or introduce defect states. The experimental observations of Cr3Al are consistent with a small but finite DOS at $E_F$ [1].

A band gap can only occur in a material with an even number of valence electrons per unit cell in order to completely fill the valence band. So how can Cr3Al, with 21 electrons per formula unit, have a band gap? The gap is possible because the primitive unit cell of the X-phase, shown in Figure 1, is actually Cr6Al2, having 42 valence electrons per unit cell. This aspect of the X-phase structure sets it apart from the other structures, which, although they may have a low DOS, have an odd number of valence electrons per unit cell and therefore must have partially filled bands crossing $E_F$.

This study of the chemical ordering of Cr3Al has important implications for materials design of transition metal compounds. The energy calculations indicate the D0_3 structure should not occur under equilibrium conditions. This may represent a lower limit on the number of valence electrons in the unit cell for the Heusler structure. Heusler alloys are usually studied with 22-31 valence electrons in the formula unit, while Cr3Al has 21 [10]. Similar compounds containing transition metals from the left side of the periodic table should be studied for X-phase type ordering.

**Table 1: Total energy (relative to minimum energy structure) and DOS($E_F$) for the five Cr3Al structures.**

<table>
<thead>
<tr>
<th>Structure</th>
<th>Total energy/atom (eV)</th>
<th>DOS($E_F$) (1/eV)</th>
</tr>
</thead>
<tbody>
<tr>
<td>bcc solid solution (Cr0.75Al0.25)</td>
<td>0.042</td>
<td>0.240</td>
</tr>
<tr>
<td>C11_b Cr2Al + bcc Cr</td>
<td>0.039</td>
<td>0.192</td>
</tr>
<tr>
<td>C11_b (off-stoich.) Cr3Al</td>
<td>0.048</td>
<td>0.224</td>
</tr>
<tr>
<td>D0_3 Cr3Al</td>
<td>0.075</td>
<td>0.207</td>
</tr>
<tr>
<td>X-phase Cr3Al</td>
<td>0</td>
<td>0.076</td>
</tr>
</tbody>
</table>

**Figure 2: The density of states for X-phase Cr3Al calculated using the three approaches.**

**References:**

Development of Superconductor Circuits for Readout of Quantum Nanomechanical Resonators

CNF Project Number: 1851-09
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Users: Francisco Paulo Rouxinol, Seung-Bo Shim

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Abstract:

Nano- and micromechanical structures integrated with superconductor circuits are promising candidates for fundamental studies of quantum mechanics and as elements for a range of applications including force sensing and quantum communication. In our work, we are fabricating a novel quantum electromechanical system that consists of three integrated nano- and micro-scale systems: a superconducting qubit, a superconducting resonant circuit, and a nanomechanical flexural resonator. This system will enable measurements of the discrete energy spectrum of nanomechanical resonators and will be a platform from which to study dissipation and quantum mechanical topics like entanglement and decoherence in nanomechanical systems.

Summary of Research:

In recent years, there has been a growing effort to integrate quantum technologies with mechanical structures in order to manipulate and measure quantum states of mechanics for applications ranging from quantum computing to sensing of weak forces to fundamental explorations of quantum mechanics at massive scales [1-9].

A central focus of this effort is the integration of superconducting electronics as control and measurement elements in nano and microelectromechanical systems (NEMS and MEMS). In just the last several years, important advancements have been made with such quantum electromechanical systems, providing researchers with a suite of tools for preparing, manipulating and measuring NEMS and MEMS near and even in the quantum domain [5-8]. In fact, it is now possible to utilize a superconducting resonant circuit (SRC) to cool a nanomechanical mode to its quantum ground state [8] or to engineer a superconducting two-level system (also known as a quantum bit or qubit) to prepare a micromechanical resonator in a quantum superposition of energy states [7]. The devices we are developing at Cornell NanoScale Science and Technology Facility (CNF) and at Syracuse University (SU) incorporate all three of these technologies (mechanics, SRC, and superconducting qubits) and will make possible an array of new experiments to probe and characterize quantum phenomena in hitherto unexplored limits.

At the heart of our devices is a capacitively-coupled superconducting charge qubit (also known as a Cooper-Pair Box, or CPB) and doubly-clamped nanomechanical resonator [5]. This qubit-coupled mechanical system is formally analogous to systems in cavity quantum electrodynamics that have been used for studying the quantum nature of light and thus is at the center of many theoretical proposals to elicit and control quantum behavior in nano- and micromechanical structures (see Ref. 5 and the references therein).

In our experiments, the role of the CPB will be two-fold: (1) In one series of experiments, it will serve as a probe by which we perform measurements of the discrete energy levels of a single nanomechanical resonator [10, 11]; and (2) In a second series of experiments, it will serve to mediate and probe an effective interaction between multiple nanoresonators [12]. The ability to read-out the energy states of a nanomechanical resonator will enable new studies of dissipation in nanomechanical systems as well as fundamental investigations of quantum fluctuation relations [13]. Moreover, the ability to couple multiple nanoresonators through a single CPB will provide a means for engineering highly non-classical entangled states of the nanoresonators and thus could serve as an important new tool for quantum computing architectures [12].

In both sets of experiments, the CPB will be read-out by an SRC composed of a lumped-element inductor and capacitor (LC) circuit (Figure 1 and Figure 2).
Our devices are patterned on 4-inch, <100>-oriented silicon wafers (~ 10 Ω⋅cm resistivity), which have been coated with low-stress silicon nitride using the GSI PECVD at CNF. We pattern large-scale features including the SRC and transmission lines (Figures 1 and 2) using photolithography. To do this we use CNF’s ASML 300C Stepper, followed by electron-beam evaporation of the aluminum in the laboratory of Prof. Britton Plourde at Syracuse University (SU), and then a standard lift-off procedure.

We then pattern the nanoresonator circuit and the Josephson junctions of the CPB using electron-beam lithography (Figure 3). This is accomplished using CNF’s JEOL 6300 followed by a double-angle evaporation of aluminum at SU. Finally the nanoresonators are released (Figure 4) by a two-step reactive ion etch process using CNF’s Oxford 82.

The first generation of devices is now being completed and will be measured this summer at milli-Kelvin temperatures on a dilution refrigerator in the LaHaye laboratory at SU.

This work is funded by a National Science Foundation CAREER grant, grant number 1056423.

References:

Figure 1: An optical image of three of our samples displaying the superconducting transmission lines and lumped element SRC for probing the CPB qubit.

Figure 2: An optical image of one of our samples displaying a close-up view of the SRC, which is composed of a planar coil and an interdigitated capacitor.

Figure 3: SEM of the CPB qubit in one of our samples.

Figure 4: SEM of one of our nanomechanical resonators similar to the ones that are currently being integrated with the CPB and SRC in our first generation devices.
Fabrication of Superconducting Devices for Quantum Information Science

CNF Project Number: 1873-10
Principal Investigator: Britton L. T. Plourde
Users: Joel Strand, Matthew Ware, Bo Xiao

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Abstract:

We are fabricating nanoscale superconductor tunnel junctions and microwave resonators for investigations in quantum information science. Such circuits have shown great promise in recent years for forming qubits, the elements of a quantum computer. We are developing architectures involving multiple superconducting qubits and microwave resonators. This involves a combination of photolithographic processing of large-scale features and electron-beam lithography for the tunnel junctions.

Summary of Research:

In recent years, circuits composed of nanoscale Josephson junctions have emerged as promising candidates 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]. The quantum coherent properties of the circuits are measured at temperatures below 50 mK with manipulation of the qubit state through microwave excitation.

We are working to develop architectures involving multiple superconducting qubits coupled to multiple low-loss microwave resonators [2]. We probe the coupling between each qubit and resonator either by measuring a vacuum Rabi splitting in the microwave transmission through the resonator when the qubit energy level difference is tuned to the characteristic frequency of the resonator, or by measuring the dispersive shift of the resonator frequency with the qubit detuned from the resonator [3].

We pattern these circuits at the CNF with nanoscale structures defined with electron-beam lithography integrated with photolithographically defined large-scale features on Si and sapphire substrates. The junctions are fabricated using the standard double-angle shadow evaporation technique, in which a resist bilayer of copolymer and PMMA is used to produce a narrow PMMA airbridge suspended above the substrate. Evaporation of aluminum from two different angles with an oxidation step in between forms a small Al-AlOx-Al tunnel junction from the deposition shadow of the airbridge. We have developed a process for defining these junctions on the JEOL9300 and we perform the aluminum evaporations in a dedicated chamber at Syracuse. We pattern large-scale features using the Autostep 200, with sputter deposition of Nb and NbN films in dedicated systems at Syracuse University. Measurements of these circuits are performed in cryogenic systems at Syracuse University, including a custom dilution refrigerator for achieving temperatures of 30 mK.

References:

Figure 1: Optical micrograph of superconducting qubit incorporating two Josephson junctions with interdigitated shunting capacitors coupled to Nb microwave resonator.

Figure 2: Qubit energy levels measured with low-temperature microwave spectroscopy as a function of magnetic flux through superconducting qubit loop.

Figure 3: Optical micrograph of superconducting qubit with Al paddle capacitor plates and Al-AlOx-Al tunnel junction in center.

Figure 4: Scanning electron micrograph of Al-AlOx-Al tunnel junction at center of superconducting qubit.
Photonic Crystal Nanocavities for Solid State Quantum Optics

CNF Project Number: 1889-10
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Abstract:
The radiative properties of an atom can be dramatically modified when it is coupled to an optical cavity [1]. Implementing such quantum optical effects in solid-state is essential for the realization of integrated quantum optical devices, including those relevant to quantum information science [2, 3]. The success relies on the fabrication of state-of-the-art artificial atoms and nanocavities. Our artificial atoms are InAs/GaAs self-assembled quantum dots (QDs) grown by molecular beam epitaxy. Our nanocavities are formed by defects in two-dimensional photonic crystal slabs. Here we report on the fabrication of a key building block for future photonic quantum technologies: the integrated coupling of photonic crystal cavities and waveguides.

Summary of Research:
Optical cavities are characterized by two main quantities: the mode volume, that is, the spatial extent of the electromagnetic confinement, and the quality factor (Q), which is proportional to the photon cavity lifetime. When trapped for long time (high-Q) in small volumes, photons strongly interact with the host material and create significant nonlinear effects. Photonic crystal nanocavities (PCNs) with ultra-high-Q and mode volume close to the diffraction limit (\(\lambda/n\))^3 have been achieved by several research groups [2, 4].

High-Qs are made possible by suppression of the electric field Fourier components within the light zone. But this comes at a price: High-Q PCNs are poorly coupled to the vertical direction (i.e. orthogonal to the slab). In this report we show the fabrication of L3 PCNs side-coupled to waveguides and optimized for highest Q and for highest far-field vertical collection [5].

The fabrication consists of the following steps: (i) we start with a molecular beam epitaxy (MBE) grown GaAs substrate. (ii) after defining the PCN pattern on the resist (ZEP 520A) by electron beam lithography, (iii) we transfer the pattern into the semiconductor by chlorine-based inductively coupled plasma reactive ion etching (ICP-RIE). Finally, (iv) the Al_{x}Ga_{1-x}As (x \approx 0.7) sacrificial layer is removed by selective HF wet etching.

Figure 1 shows the scanning electron microscope images of the fabricated PCNs. Figure 1(a) shows the high-Q L3 PCN formed by three missing air holes in the hexagonal lattice of holes patterned in a \(\lambda/n\) thick slab. The marked holes were shifted and shrunk to maximize the Q. Figure 1(b) shows the L3 PCN optimized for far-field vertical collection. Starting form the former design, Figure 1(a), the marked holes were enlarged. Figure 2 shows the photoluminescence spectra of our PCNs. The photo-luminescence spectrum was measured by the cross-polarization resonant scattering. Emission peaks are well fitted by Lorentzian curves.

The typical Qs for high-Q L3 PCN were resolution limited, \(Q = 7 \times 10^4\) (Figure 2a). As expected, a lower \(Q = 3 \times 10^4\) (Figure 2b) was measured in L3 PCNs optimized for far-field, but a remarkable enhancement of light intensity was detected in the vertical direction.

When integrating the L3 PCNs optimized for far-field together with side coupled waveguides (Figure 3), we were able to observe the light in the waveguides and extracted from the center PCN even by naked eyes (Figure 3 inset). We note that the PCNs optimized for far-field can also act like a grating coupler that can gather the light from vertical direction. This allows us to scale up the device more easily compared to the edge-coupling and the fiber taper techniques.

Light confinement in nanocavities relying only on index guiding is well understood, while current PCN understanding is much less mature because of the hybrid character of the confinement. (Photons are confined vertically by total internal reflection and laterally by Bragg reflection.) Our PCNs demonstrate that we can model and fabricate PCNs in GaAs with specific functionalities. Together with the QDs, we can implement designs [2, 5] that are suitable for applications where light extraction is key such as single photon emitters [6].
References:


First-Principles Study of the Surface Chemistry of Metal-Oxide Nanostructures

CNF Project Number: 1998-11
Principal Investigator: Yongqiang Xue
Users: Abraham Hmiel, Yongqiang Xue

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Abstract:
In this project, we utilize recent developments in density functional theory (DFT) and molecular dynamics (MD), including the self-consistent van der Waals (vdW) functional and the effective screening medium (ESM) theory, to study the surface chemistry and wetting of H₂O on nanostructured TiO₂ surfaces and metal-decorated and biased TiO₂ surfaces. The energetics, kinetics and dynamics of the water monomer, dimer, small cluster, monolayer and multilayer on these nanostructured TiO₂ surfaces, in the presence of surface defects and metal decoration, will allow a more complete understanding of the key surface processes that will govern the utility of these devices.

Summary of Research:
The rutile TiO₂ <110> surface, apart from being a prototypical model for study in metal-oxide interfaces, is technologically relevant in energy applications like fuel cells and photocatalytic water splitting [1]. Current carbonless support materials in development for fuel cell applications make use of TiO₂ nanorods > 10 nm in diameter that can be modeled as surface slabs in DFT. The current state of knowledge about this surface’s electronic structure is significant, but falls short in some areas, namely, the chemical reactions that take place at a water interface in the presence of surface defects or adsorbed metals and at electrical bias. Developments in density functional theory (DFT) allow for an accurate self-consistent treatment of van der Waals (vdW) dispersion effects between water molecules and other bound systems whose character is neither covalent, ionic, nor metallic [2]. Furthermore, the ability to simulate DFT with a screened electric field is useful for the accurate treatment of real devices, and a modification to the electrostatic boundary conditions to achieve this end has been grafted onto mainstream DFT code packages [3].

We use a modified version of the SIESTA [4] code on the CNF cluster with Perdew-Burke-Ernzerhof (PBE) and vdW [2] exchange-correlation (XC) functionals to relax rutile and anatase surface slabs of varying thickness and test for convergence, with primary focus on the rutile <110> surface slab. We use Troullier-Martins-style nonlocal norm-conserving pseudopotentials to screen the ionic nuclei with core electrons.

Our present results have corroborated H₂O+TiO₂ adsorption geometries and energies for the PBE functional [5], and preliminary results for the opt-b86 [6] functional indicate similar physics, although the interesting part of the study — performing molecular dynamics with self-consistent vdw-DFT with adsorbed metals and under electrical bias and observing the competing effects that change the structure of the hydrogen-bond network — is forthcoming.

In our paper, which has been accepted into Physical Review B, we have analyzed the atomic and electronic properties of the rectangular rutile TiO₂ nanowires oriented along the [001] direction and enclosed by {110} facets, extending the result of Bredow [7] to two dimensions of confinement. We find that the dependence of the nanowire properties on the number of TiO₂ layers are largely connected to the presence or absence of a mirror Ti-O plane along each confinement direction. Although this dependence on the even-odd parity of the number of TiO₂ layers has been discussed in previous studies on rutile TiO₂ <110> thin films and surfaces, we find that the ability to tune separately the number of layers in each confinement direction leads to novel size-and shape-dependent electronic properties in rutile TiO₂ nanowires, which arise from the interplay between surface relaxation and quantum confinement effects. We have focused exclusively on stoichiometric nanowires that can be constructed directly from the bulk rutile TiO₂ crystals.
References:

Electron Beam Lithography of Superconducting Qubits and Meander Wires

CNF Project Number: 2003-11
Principal Investigators: William D. Oliver, Andrew J. Kerman
Users: Ted Gudmundsen, William D. Oliver, Steve Spector

Affiliation: MIT Lincoln Laboratory
Primary Source of Research Funding: IARPA, Laboratory for Physical Sciences, and US Government
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Abstract:
Superconducting qubits [1, 2] and superconducting nanowire single-photon detectors [3] each contain circuit elements (Josephson junctions, meander wires, ...) with critical dimensions around 100 nm. In this project, we use the CNF electron-beam lithography systems and SEMs to pattern and image these small features on 50 mm and 150 mm wafers.

Summary of Research:
Superconducting qubits are solid-state artificial atoms, comprising lithographically-defined Josephson tunnel junctions and superconducting interconnects. Their potential for lithographic scalability, compatibility with microwave control, and nanosecond-scale operation times make superconducting qubits a leading candidate for quantum information science and technology applications.

In this work, we use electron-beam lithography and ZEP resist to pattern a bilayer shadow mask comprising Ge on MMA-MAA copolymer. This bilayer is then etched to undercut the copolymer, leaving suspended Ge bridges, which serve as the shadow mask used to form Josephson junctions via a double-angle evaporation process. In Figure 1, we show a tilted view of the bilayer after undercutting. The junctions formed in this manner have feature sizes around 150-250 nm. While this does not push the limits of electron-beam lithography, the flexibility of this approach (e.g., design changes in software rather than new optical masks) is important to us.

We also pattern NbN meander wires in this program for use in both single-photon detector arrays and superconducting qubits. These meander wires (thickness: 5-10 nm; width: 50-150 nm) have extremely high kinetic inductivity, while featuring a rather low parasitic capacitance. In this work, we are patterning the NbN wires using electron beam lithography and ZEP resist. We are working to improve width uniformity, which is important for the high-efficiency operation of the single-photon detectors. In addition, we are fabricating and incorporating these wires into metastable superconducting qubit designs, which leverage the large inductance to achieve a lifetime exceeding one second.

In Figure 2, we show a NbN meander wire after etching.

References:
Figure 1: SEM image of the Ge/MMA-MAA bilayer after etching. The resulting shadow mask is used with double-angle evaporation to fabricate Josephson junctions for superconducting qubits.

Figure 2: SEM image of a NbN meander wire. A similar geometry is used for both high-efficiency photodetection and for realizing the high-inductance element in the metastable RF SQUID qubit.
Determining Charge Mobility by Electric Force Microscopy Using a Transistor with Interdigitated Gate Electrodes

CNF Project Number: 863-00
Principal Investigator: Professor John Marohn¹
Users: Nikolas Hoepker², Louisa Brown¹, Joseph Singh¹, James O’Dea¹

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Abstract:
We have fabricated organic field effect transistors (OFETs) with interdigitated gate electrodes. These devices will be used in an electric force microscope in order to measure the charge mobility in the organic material.

Summary:
Our proposed experiment builds on prior work on organic electronic ratchets by Roeling and coworkers [1, 2] who demonstrated that charge in an OFET can be funneled from the source to the drain electrode without applying a source-drain bias. The charge is displaced laterally by applying a cyclic series of voltages to underlying interdigitated gate electrodes.

While Roeling and coworkers only measured the net current from the source to the drain electrodes, we here propose observing the induced lateral charge motion locally using an electric force microscope. A sketch of the proposed experimental setup is shown in Figure 1.

Initially, a non-uniform charge density is placed in the channel by applying a set of distinct gate voltages, $V_{q1}$ and $V_{q2}$. At $t = 0$, we set $V_{q1} = V_{q2}$. As a result, charge moves laterally away from the initially highly charged regions until a new steady state is reached.

We propose to measure the transient shift in the electrostatic potential by recording the frequency shift of an electric force microscope cantilever. With accurate modeling, we hope to extract the charge mobility in the organic layer from the time scale of this transient response, which we estimate to be 12 ms in one of our devices.

To fabricate the transistor substrates shown in Figure 2, we first used photolithography to pattern Pt electrodes onto a fused silica wafer. Then we deposited 315 nm of silicon oxide by plasma-enhanced chemical vapor deposition (PECVD), followed by a second photolithography step in order to pattern top Au electrodes. Electrical contact to the underlying Pt electrodes was made by etching windows through the PECVD oxide using a reactive ion etch process.

The main challenge was to create the underlying Pt electrodes. It is crucial that these electrodes, which were 2.25 meters (meters) long and spaced by only 1 µm, were not electrically shorted to one another.

References:
Figure 1: Here we show a sketch of the proposed experiment. At time $t < 0$, the applied gate voltages, $V_{G10}$ and $V_{G20}$, are distinct. At $t = 0$, we set $V_{G1} = V_{G2}$ and measure the resulting lateral transient motion of charge by electric force microscopy.

Figure 2: Images of the fabricated transistor substrate with top Au source and drain electrodes and underlying Pt gate electrodes.
Novel Fabrication Process of Silicon Nano-Field Effect Transistor for Detection Analysis

CNF Project Number: 1469-06
Principal Investigator and User: Dr. Nirankar N. Mishra

Affiliation: Center for Advanced Microelectronics and Bio-molecular Research, University of Idaho
Primary Source of Research Funding: United State Department of Agriculture
Contact: nmishra@uidaho.edu
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Abstract:
This project has achieved using e-beam and optical lithography: 1) A novel device design to make excellent contact stability between metal and semiconductor; 2) On-chip gold and platinum field effect electronic device with silicon nanowire for capturing Litsterolysin O (LLO) molecules; 3) A 10 nm gate dielectric layer of HfO₂, thermal silicon oxide and tantalum oxide and device characterization with yield 80% per wafer; 4) Single frequency real time detection of LLO on anti LLO polyclonal antibody modify device; and 5) Capacitive electrochemical impedance spectra (EIS) of Litsterolysin O.

Project Summary:
The silicon nanowire [1] field effect transistors (nano-FET) were fabricated [2] using minimum cost effective steps (Figure 1). The nano-FET devices were very stable and responsive due to our first-time-reported unique design approach in the contact area, which has several hole and wires to make excellent metal-semiconductor contact. Several p- and n-type polysilicon and monocrystalline silicon wafers were used in the process development of these devices. The medium Boron dopant levels on the devices layer (mono silicon) were shown the highest sensitivity. We also assume that there was at least 5-10% loss of boron dopant during specific chlorine plasma etch. The current-voltage (C-V) test of the polysilicon nanowire (20-150 nm size) based devices shown as variable field effect resistor whereas mono-crystal silicon devices have shown excellent NMOS type nano-FET characteristics (Figure 2).

Figure 1: Uniquely designed images of nanowires and contact pads, the better design improved the metal contact with semiconductor and also provide mechanical stability to the nanowire. These wires are ~ 20-100 nm wide and ~ 50-150 nm thin boron doped mono-crystalline silicon separated with 20 µm between two Au or Pt/Ti or Cr metal contacts.

Figure 2: Current-voltage plot of one of the device at -2V to 2V from back gate shows transistor curve of the nanowire with excellent on-off ratio.
Various insulator layers were also deposited on the nanowire surface, but only thin hafnium oxide (~8 nm) and thermal SiO$_2$ (~15 nm) showed outstanding insulating characteristics with negligible current leakage; whereas, devices with alumina and silicon nitride failed.

Current detection methods for LLO are time-consuming and expensive. The general goal of this study is to develop a capacitive on-chip real-time transducer for LLO detection. The devices were also tested with EIS, using different pH solutions, which show changes in impedance with each different pH solution. Finally, functional device surfaces were modified with an anti-LLO polyclonal antibody. Modified devices were then used for real time single frequency EIS at 1.1 Hz with 10 mV of AC. The 10 fmole LLO solution was injected on the system, and when the LLO interacted with the antibody, a stable drop in capacitance (~200 nF at 1.1 Hz) was observed over time (Figure 3).

We have also seen detachment of the LLO from the device surface by injected a bleaching solution that sent devices back to a baseline capacitance zone.

Our analysis shows these devices can be used for real time monitoring of LLO in any food product after preconcentration steps.

**Acknowledgements:**

This work was performed 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-0335765). This work was supported in part by the USDA grants CSREES 3447919833 and 3447919150.

**References:**


### Index of Reports by Project Number

<table>
<thead>
<tr>
<th>Project Number</th>
<th>Page Numbers</th>
</tr>
</thead>
<tbody>
<tr>
<td>111-80</td>
<td>198, 200, 202</td>
</tr>
<tr>
<td>386-90</td>
<td>96, 98, 100, 102</td>
</tr>
<tr>
<td>398-91</td>
<td>2</td>
</tr>
<tr>
<td>522-94</td>
<td>104</td>
</tr>
<tr>
<td>598-96</td>
<td>204, 206, 208, 210, 212</td>
</tr>
<tr>
<td>640-97</td>
<td>106</td>
</tr>
<tr>
<td>657-97</td>
<td>4</td>
</tr>
<tr>
<td>692-98</td>
<td>6</td>
</tr>
<tr>
<td>715-98</td>
<td>70</td>
</tr>
<tr>
<td>731-98</td>
<td>8</td>
</tr>
<tr>
<td>752-98</td>
<td>72</td>
</tr>
<tr>
<td>762-99</td>
<td>136, 138, 140</td>
</tr>
<tr>
<td>780-99</td>
<td>74</td>
</tr>
<tr>
<td>804-99</td>
<td>76, 108, 142, 144</td>
</tr>
<tr>
<td>848-00</td>
<td>10</td>
</tr>
<tr>
<td>863-00</td>
<td>146, 244</td>
</tr>
<tr>
<td>900-00</td>
<td>110, 214</td>
</tr>
<tr>
<td>980-01</td>
<td>164, 166, 168</td>
</tr>
<tr>
<td>1088-02</td>
<td>78</td>
</tr>
<tr>
<td>1096-02</td>
<td>170</td>
</tr>
<tr>
<td>1225-04</td>
<td>12</td>
</tr>
<tr>
<td>1230-04</td>
<td>80</td>
</tr>
<tr>
<td>1236-04</td>
<td>82, 112</td>
</tr>
<tr>
<td>1255-04</td>
<td>14</td>
</tr>
<tr>
<td>1283-04</td>
<td>58</td>
</tr>
<tr>
<td>1314-05</td>
<td>216</td>
</tr>
<tr>
<td>1356-05</td>
<td>114</td>
</tr>
<tr>
<td>1361-05</td>
<td>218</td>
</tr>
<tr>
<td>1371-05</td>
<td>60</td>
</tr>
<tr>
<td>1380-05</td>
<td>172</td>
</tr>
<tr>
<td>1393-05</td>
<td>16</td>
</tr>
<tr>
<td>1400-05</td>
<td>116</td>
</tr>
<tr>
<td>1465-06</td>
<td>18</td>
</tr>
<tr>
<td>1469-06</td>
<td>246</td>
</tr>
<tr>
<td>1472-06</td>
<td>174</td>
</tr>
<tr>
<td>1486-06</td>
<td>220</td>
</tr>
<tr>
<td>1540-07</td>
<td>20</td>
</tr>
<tr>
<td>1542-07</td>
<td>148</td>
</tr>
<tr>
<td>1577-07</td>
<td>222</td>
</tr>
<tr>
<td>1592-07</td>
<td>62</td>
</tr>
<tr>
<td>1636-08</td>
<td>26</td>
</tr>
<tr>
<td>1645-08</td>
<td>118, 224</td>
</tr>
<tr>
<td>1646-08</td>
<td>150</td>
</tr>
<tr>
<td>1651-08</td>
<td>176</td>
</tr>
<tr>
<td>1673-08</td>
<td>226</td>
</tr>
<tr>
<td>1681-08</td>
<td>152</td>
</tr>
<tr>
<td>1724-08</td>
<td>178</td>
</tr>
<tr>
<td>1726-08</td>
<td>22</td>
</tr>
<tr>
<td>1735-08</td>
<td>228</td>
</tr>
<tr>
<td>1743-09</td>
<td>24</td>
</tr>
<tr>
<td>1746-09</td>
<td>120</td>
</tr>
<tr>
<td>1764-09</td>
<td>180</td>
</tr>
<tr>
<td>1782-09</td>
<td>122</td>
</tr>
<tr>
<td>1816-09</td>
<td>26</td>
</tr>
<tr>
<td>1818-09</td>
<td>28</td>
</tr>
<tr>
<td>1823-09</td>
<td>230</td>
</tr>
<tr>
<td>1838-09</td>
<td>154</td>
</tr>
<tr>
<td>1840-09</td>
<td>156</td>
</tr>
<tr>
<td>1844-09</td>
<td>64</td>
</tr>
<tr>
<td>1845-09</td>
<td>232</td>
</tr>
<tr>
<td>1851-09</td>
<td>234</td>
</tr>
<tr>
<td>1854-09</td>
<td>84</td>
</tr>
<tr>
<td>1855-09</td>
<td>182</td>
</tr>
<tr>
<td>1857-10</td>
<td>184, 186</td>
</tr>
<tr>
<td>1858-10</td>
<td>30</td>
</tr>
<tr>
<td>1862-10</td>
<td>188</td>
</tr>
<tr>
<td>1863-10</td>
<td>86</td>
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<tr>
<td>1872-10</td>
<td>190</td>
</tr>
<tr>
<td>1873-10</td>
<td>236</td>
</tr>
<tr>
<td>1880-10</td>
<td>192</td>
</tr>
<tr>
<td>1888-10</td>
<td>32</td>
</tr>
<tr>
<td>1889-10</td>
<td>238</td>
</tr>
<tr>
<td>1920-10</td>
<td>194</td>
</tr>
<tr>
<td>1936-10</td>
<td>66</td>
</tr>
<tr>
<td>1940-10</td>
<td>34</td>
</tr>
<tr>
<td>1959-10</td>
<td>124</td>
</tr>
<tr>
<td>1969-10</td>
<td>88</td>
</tr>
<tr>
<td>1982-11</td>
<td>126</td>
</tr>
<tr>
<td>1986-11</td>
<td>36</td>
</tr>
<tr>
<td>1990-11</td>
<td>90</td>
</tr>
<tr>
<td>1997-11</td>
<td>196</td>
</tr>
<tr>
<td>1998-11</td>
<td>240</td>
</tr>
<tr>
<td>2003-11</td>
<td>242</td>
</tr>
<tr>
<td>2016-11</td>
<td>38</td>
</tr>
<tr>
<td>2017-11</td>
<td>40</td>
</tr>
<tr>
<td>2034-11</td>
<td>42</td>
</tr>
<tr>
<td>2036-11</td>
<td>128</td>
</tr>
<tr>
<td>2038-11</td>
<td>44</td>
</tr>
<tr>
<td>2040-11</td>
<td>46</td>
</tr>
<tr>
<td>2044-11</td>
<td>48</td>
</tr>
<tr>
<td>2046-11</td>
<td>158</td>
</tr>
<tr>
<td>2049-11</td>
<td>50</td>
</tr>
<tr>
<td>2053-11</td>
<td>130</td>
</tr>
<tr>
<td>2054-11</td>
<td>52</td>
</tr>
<tr>
<td>2058-11</td>
<td>160</td>
</tr>
<tr>
<td>2065-11</td>
<td>54</td>
</tr>
<tr>
<td>2067-11</td>
<td>68</td>
</tr>
<tr>
<td>2068-11</td>
<td>56</td>
</tr>
<tr>
<td>2074-11</td>
<td>162</td>
</tr>
<tr>
<td>2077-11</td>
<td>92</td>
</tr>
<tr>
<td>2079-11</td>
<td>132</td>
</tr>
<tr>
<td>2089-11</td>
<td>134</td>
</tr>
<tr>
<td>2099-12</td>
<td>94</td>
</tr>
<tr>
<td>2121-12</td>
<td>78</td>
</tr>
</tbody>
</table>
# Index of Reports: Principal Investigators & Users

## A

Abruña, Héctor D. ........................................ 58
Adiga, Vivekananda P. .................. 136, 138, 140
Agyeman-Budu, David .................. 182
Aksit, Mahmut ................................. 120
Alden, Jonathan S. .............................. 110
Alkayyali, Amani ................................... 34
Allen, Leslie H. ................................. 104
Andosca, Robert ............................... 154
Apsel, Alyssa ..................................... 4
Arias, Tomas A. ................................. 68
Auluck, Kshitij .................................... 70
Austin, Robert H. ......................... 2, 66

## B

Badolato, Antonio .............................. 238
Bai, Ying (Richard) ......................... 12
 Baird, Barbara A. ............................... 22, 98
Bajpai, Saumendra K. ....................... 46
Barnard, Arthur ................................. 214
Barrett, Aliyah ................................. 66
Barton, Robert A. ......................... 136, 138
Bau, Haim H. ..................................... 148
Beccera, Natalie ............................... 116
Bhave, Sunil A. ................................. 172
Bian, Kaifu ....................................... 118
Bilderback, Donald ......................... 182
Biser, Jeffrey ..................................... 134
Blees, Melina ................................. 214
Blocher, David B. .............................. 152
Boekelheide, Zoe ........................... 232
Borca-Tasciuc, Diana-Andra .............. 162
Boyd, Robert W. .............................. 188
Brenna, J. Thomas ............................ 62
Brock, Joel D. .................................. 128
Brown, Lola ..................................... 220
Brown, Louisia ................................. 244
Bryce, Brian A. ................................. 142
Buhrman, Robert A. .......................... 198, 200, 202, 210, 212

## C

Cao, Yunhao .................................... 84
Capetillo, Estibaliz .......................... 22
Cardenas, Jaime ............................... 164
Carr, Russell T. ............................... 44
Chan, Wei Min ................................. 108
Che, Sara ........................................ 18
Chen, Huangye ............................... 216
Chen, Peng ...................................... 64
Chen, Yih-Fan .................................. 186
Choi, Jinwoo .................................... 54
Choi, Joshua Jong ......................... 118
Choudhary, Eric .............................. 64
Cipriany, Benjamin R. ..................... 138
Cohen, Itai ...................................... 218
Corso, Thomas N. ............................ 50
Costello, Deirdre ............................ 26
Craighead, Harold G. ...................... 136, 138, 140
Cruz, Dana ..................................... 22
Cui, Yontao ..................................... 212

## D

Daniel, Susan ................................... 26, 28
Dasari, Prasad L.V.K. ..................... 60
Davidson, Patricia .......................... 54
De Alba, Roberto ............................ 140
de la Rama, Lito ............................... 104
DeFeo, Michael ............................... 228
Deffner, Bernd ................................. 106
DelNero, Peter ................................. 20
Dorta-Quinones, Carlos ................... 78
Duan, Huinan ................................... 82, 112, 116

## E

Engheta, Nader ............................... 178
Erickson, David .............................. 8
Esch, Mandy ..................................... 8

## F

Fain, Romy ...................................... 184
Finkelstein, Kenneth ...................... 182
Fischbach-Teschl, Claudia ............... 20
Foquet, Mathieu .............................. 194

## G

Gabrielli, Lucas Heitzmann .............. 166
Gao, Boshen ................................. 188
Gao, Guoying .................................. 60
Giannelis, Emmanuel P. .................. 100
Gillilan, Richard E. ....................... 34
Gingerich, Marcus ........................... 4
Grab, Jennifer L. ......................... 206, 210
Graham, Matt W. ............................. 204
Greene, Andrew .............................. 74
Gregoire, John M. ............................ 116
Griffith, Austin G. ........................... 168
Grogan, Joseph M. ......................... 148
Gutzik, Scott ................................. 126
Gudmundsen, Ted ........................... 242
Gupta, Arnol ................................. 82
# INDEX

## O

<table>
<thead>
<tr>
<th>Name</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>O’Dea, James</td>
<td>244</td>
</tr>
<tr>
<td>Ober, Christopher K.</td>
<td></td>
</tr>
<tr>
<td></td>
<td>96, 98, 100, 102, 106</td>
</tr>
<tr>
<td>Oenick, Marsha</td>
<td>38</td>
</tr>
<tr>
<td>Ohm, Christian</td>
<td>106</td>
</tr>
<tr>
<td>Oktabayrsky, Serge</td>
<td>74</td>
</tr>
<tr>
<td>Oliver, William D.</td>
<td>242</td>
</tr>
<tr>
<td>Oncescu, Vlad</td>
<td>86</td>
</tr>
<tr>
<td>Ong, Peijie</td>
<td>200</td>
</tr>
<tr>
<td>Onnata, Tiberiut</td>
<td>156</td>
</tr>
<tr>
<td>Otelaja, Obafemi</td>
<td>120</td>
</tr>
<tr>
<td>Ouyang, Christine</td>
<td>100</td>
</tr>
<tr>
<td>Oxaal, John</td>
<td>162</td>
</tr>
</tbody>
</table>

## P

<table>
<thead>
<tr>
<th>Name</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pai, Chi-Feng</td>
<td>200</td>
</tr>
<tr>
<td>Park, Jiwoong</td>
<td>204</td>
</tr>
<tr>
<td>Parpia, Jeevak M.</td>
<td>138, 214</td>
</tr>
<tr>
<td>Pasupuleti, Tushara</td>
<td>38</td>
</tr>
<tr>
<td>Person, Steven</td>
<td>170</td>
</tr>
<tr>
<td>Petersen, Poul</td>
<td>66</td>
</tr>
<tr>
<td>Pham, An</td>
<td>38</td>
</tr>
<tr>
<td>Phelps, Joshua B.</td>
<td>40, 72</td>
</tr>
<tr>
<td>Pilgrim, Gregory</td>
<td>122</td>
</tr>
<tr>
<td>Plaza, Manuel</td>
<td>128</td>
</tr>
<tr>
<td>Plourde, Briton L.T.</td>
<td>216, 228, 236</td>
</tr>
<tr>
<td>Poitrus, Carl B.</td>
<td>164</td>
</tr>
<tr>
<td>Pollack, Lois</td>
<td>6</td>
</tr>
<tr>
<td>Prot, Jean-Matthieu</td>
<td>8</td>
</tr>
</tbody>
</table>

## R

<table>
<thead>
<tr>
<th>Name</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rajwade, Shantanu</td>
<td>70</td>
</tr>
<tr>
<td>Ralph, Daniel C.</td>
<td></td>
</tr>
<tr>
<td></td>
<td>204, 206, 208, 210, 212</td>
</tr>
<tr>
<td>Reinhart-King, Cynthia A.</td>
<td>24, 46</td>
</tr>
<tr>
<td>Reuter, Mark C.</td>
<td>142</td>
</tr>
<tr>
<td>Ribeill, Guilhem</td>
<td>222</td>
</tr>
<tr>
<td>Richards, Mark</td>
<td>28</td>
</tr>
<tr>
<td>Roberts, Samantha</td>
<td>214</td>
</tr>
<tr>
<td>Robinson, Richard D.</td>
<td>120</td>
</tr>
<tr>
<td>Rodriguez-Lopez, Joaquin</td>
<td>58</td>
</tr>
<tr>
<td>Rogachev, Andrey</td>
<td>60</td>
</tr>
<tr>
<td>Rosenberg, Steven</td>
<td>154</td>
</tr>
<tr>
<td>Rouxinol, Francisco Paulo</td>
<td>234</td>
</tr>
<tr>
<td>Rubin, Joshua</td>
<td>144</td>
</tr>
<tr>
<td>Ruiz-Vargas, Carlos S.</td>
<td>220</td>
</tr>
</tbody>
</table>

## S

<table>
<thead>
<tr>
<th>Name</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sakakibara, Reyu</td>
<td>138</td>
</tr>
<tr>
<td>Sato, Yuki</td>
<td>230</td>
</tr>
<tr>
<td>Savage, John</td>
<td>218</td>
</tr>
<tr>
<td>Schwarz, Kathleen</td>
<td>68</td>
</tr>
<tr>
<td>Senko, Alexander W.</td>
<td>146</td>
</tr>
<tr>
<td>Serey, Xavier</td>
<td>186</td>
</tr>
<tr>
<td>Sethna, James P.</td>
<td>124</td>
</tr>
<tr>
<td>Sharf, Tal</td>
<td>32</td>
</tr>
<tr>
<td>Shaw, Jonathan</td>
<td>70</td>
</tr>
<tr>
<td>Shealy, James Richard</td>
<td>80</td>
</tr>
<tr>
<td>Shi, Su-Fei</td>
<td>204</td>
</tr>
<tr>
<td>Shi, Zhimin</td>
<td>188</td>
</tr>
<tr>
<td>Shim, Seung-Bo</td>
<td>234</td>
</tr>
<tr>
<td>Shire, Douglas</td>
<td>4</td>
</tr>
<tr>
<td>Shuler, Michael L.</td>
<td>8, 18</td>
</tr>
<tr>
<td>Singhai, Amit</td>
<td>22</td>
</tr>
<tr>
<td>Singh, Joseph</td>
<td>244</td>
</tr>
<tr>
<td>Sivarakrishnan, Sriam</td>
<td>176</td>
</tr>
<tr>
<td>Sliz, Josiah</td>
<td>54</td>
</tr>
<tr>
<td>Song, Young Hye</td>
<td>20</td>
</tr>
<tr>
<td>Spector, Steve</td>
<td>242</td>
</tr>
<tr>
<td>Strand, Joel</td>
<td>236</td>
</tr>
<tr>
<td>Stewart, Derek</td>
<td>108, 120, 232</td>
</tr>
<tr>
<td>Stokol, Tracy</td>
<td>18</td>
</tr>
<tr>
<td>Storch, Isaac R.</td>
<td>136, 138</td>
</tr>
<tr>
<td>Strock, Abraham D.</td>
<td>20</td>
</tr>
<tr>
<td>Sun, Yong</td>
<td>178</td>
</tr>
<tr>
<td>Sundararaman, Ravishankar</td>
<td>144</td>
</tr>
<tr>
<td>Sureshkumar, Radhakrishna</td>
<td>94</td>
</tr>
</tbody>
</table>

## V

<table>
<thead>
<tr>
<th>Name</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>van Dover, R. Bruce</td>
<td>82, 112, 116</td>
</tr>
<tr>
<td>VanDelden, Jay</td>
<td>230</td>
</tr>
<tr>
<td>Verbridge, Scott</td>
<td>20</td>
</tr>
<tr>
<td>Vinci, Richard P.</td>
<td>134</td>
</tr>
</tbody>
</table>

## W

<table>
<thead>
<tr>
<th>Name</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>Wakefield, Devin</td>
<td>22</td>
</tr>
<tr>
<td>Wallin, Christopher</td>
<td>136</td>
</tr>
<tr>
<td>Wang, Chen</td>
<td>210</td>
</tr>
<tr>
<td>Wang, Haozhi</td>
<td>228</td>
</tr>
<tr>
<td>Wang, Shu-yi</td>
<td>162</td>
</tr>
<tr>
<td>Wang, Si Ping</td>
<td>138, 214</td>
</tr>
<tr>
<td>Wang, Xiao</td>
<td>.78</td>
</tr>
<tr>
<td>Wardini, Jenna</td>
<td>32</td>
</tr>
<tr>
<td>Ware, Matthew</td>
<td>236</td>
</tr>
<tr>
<td>Welch, M. Elizabeth</td>
<td>106</td>
</tr>
<tr>
<td>Whitham, Kevin</td>
<td>118, 224</td>
</tr>
<tr>
<td>Wiesner, Ulrich</td>
<td>114</td>
</tr>
<tr>
<td>Woll, Arthur</td>
<td>182</td>
</tr>
<tr>
<td>Wu, Mingming</td>
<td>56</td>
</tr>
<tr>
<td>Wyatt, John</td>
<td>4</td>
</tr>
</tbody>
</table>

## X

<table>
<thead>
<tr>
<th>Name</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>Xiao, Bo</td>
<td>236</td>
</tr>
<tr>
<td>Xu, Yaqiong</td>
<td>84</td>
</tr>
<tr>
<td>Xue, Lin</td>
<td>212</td>
</tr>
<tr>
<td>Xue, Yongqiang</td>
<td>240</td>
</tr>
</tbody>
</table>

## Y

<table>
<thead>
<tr>
<th>Name</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>Yang, Yang</td>
<td>84</td>
</tr>
<tr>
<td>Yilmaz, Mehmet</td>
<td>150</td>
</tr>
<tr>
<td>Yuan, Chao-Chen</td>
<td>116</td>
</tr>
</tbody>
</table>

## Z

<table>
<thead>
<tr>
<th>Name</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>Zach, Michael</td>
<td>132</td>
</tr>
<tr>
<td>Zappone, William</td>
<td>88</td>
</tr>
<tr>
<td>Zaretski, Aliaksandr</td>
<td>214</td>
</tr>
<tr>
<td>Zehnder, Alan T.</td>
<td>126, 152</td>
</tr>
<tr>
<td>Zhang, Yefe</td>
<td>.98</td>
</tr>
<tr>
<td>Zhao, Ying</td>
<td>10</td>
</tr>
<tr>
<td>Zhelev, Nikolay</td>
<td>140</td>
</tr>
</tbody>
</table>