The 2017 NNCI REU Convocation

August 6-8, 2017

Georgia Institute of Technology
Institute for Electronics and Nanotechnology

National
Nanotechnology
Coordinated
Infrastructure

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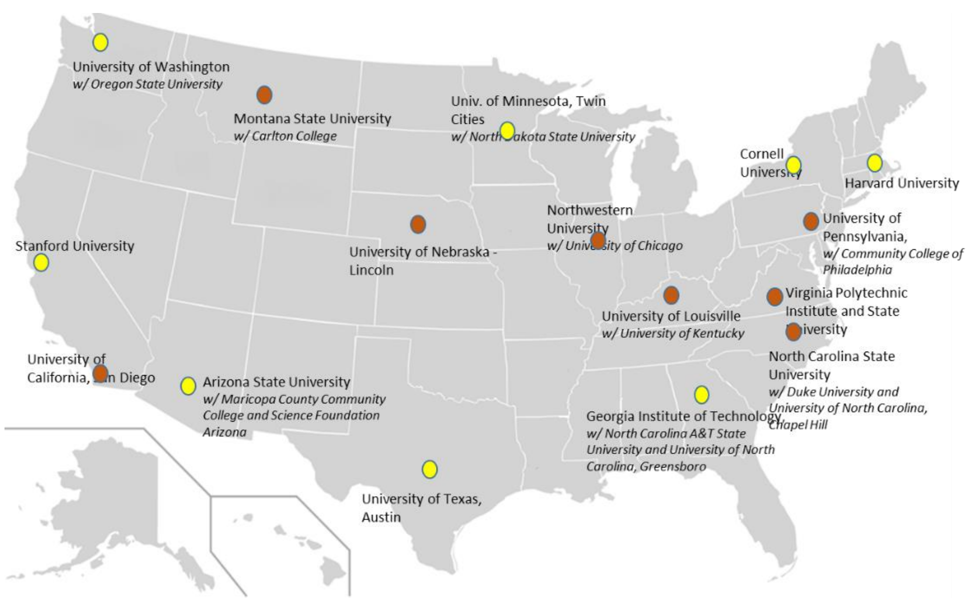
Acronyms and university affiliation(s) of the NNCI sites *participating* in the NNCI Convocation

CNF	The Cornell NanoScale Science & Technology Facility	Cornell University
CNS	Center for Nanoscale Systems	Harvard University
MANTH	Mid Atlantic Nanotechnology Hub	University of Pennsylvania
KY MMNIN	Kentucky Multi-Scale Manufacturing and Nano Integration Node	University of Louisville
MONT	Montana Nanotechnology Facility,	Montana State University
NCI-SW	Nanotechnology Collaborative Infrastructure Southwest	Arizona State University
RTNN	Research Triangle Nanotechnology Network,	North Carolina State University, Duke University, and University of Chapel Hill
SENIC	Southeastern Nanotechnology Infrastructure Corridor,	Georgia Institute of Technology
SHyNE	Soft and Hybrid Nanotechnology Experimental Resource,	Northwestern University
TNF	Texas Nanofabrication Facility,	University of Texas at Austin

Location of NNCI Sites



Thanks for attending the first NNCI REU Convocation!

2017 NNCI REU Convocation
Georgia Institute of Technology, Atlanta, GA

Sunday August 6, 2017

12:00 – 5:00 p.m. Intern Check-in at Regency Suites Hotel
6:00 – 8:30 p.m. Welcome and Pizza Party, Marcus Nanotechnology Building 345 Ferst Drive

Monday August 7, 2017 Global Learning Center

8:30 – 8:40 AM Welcome Room

Session A: Global Learning Center Room 222
Moderator: Marylene Palard, University of Texas at Austin

Session B: Global Learning Center Room 323
Moderator: David Dikensheets, Montana State University

9:40-9:55 a.m. Break

Session C. Global Learning Center Room 222

Moderator: Lynn Rathbun, Cornell University

Session D: Global Learning Center Room 323

Moderator: Nancy Healy, Georgia Institute of Technology

10:55 11:10 a.m. Break

Session E Global Learning Center Room 222

Moderator: Kathryn Hollar, Harvard University

12:00-1:00 p.m. Lunch

Session F Global Learning Center Room 222
Moderator: Ana Sanchez Gonzalez, University of Louisville

F.1 1:00-1:10 p.m. Mr. Jason Mulderrig, MANTH
Atomic Force Microscopy-based Mechanical Testing Reveals the Mechanisms of Plasticity in Disordered Nanoparticle Packings…………………………………………………………………………….Page 28
F.2 1:10-1:20 p.m. Mr. Michael Hoeft, CNS
Optimal Strength Nano-Cellular Materials…………………………………………………………………..Page 20
F.3 1:20-1:30 p.m. Mr. Roman Marcarelli, CNS
Low-loss Zero-index Metamaterials Induced by a Bound State in the Continuum………Page 25
F.4 1:30 -1:40 p.m. Ms. Katrina Raichle, MANTH
Decreasing the Defects in Free-Standing Nickel Inverse Opal Cellular Solids………………Page 33
F.5 1:40-1:50 p.m. Mr. Steven Ochoa, SHyNE
Transition Metal Sulfide Heterostructures for Hydrogen Evolution Reaction………………Page 30
F.6 1:50-2:00 p.m. Mr. Paul Cuillier, KY MMNIN
Photoconductivity in Rare-earth Doped Nanocrystalline Titanium Dioxide…………………Page 15

2:00-2:15 PM Break

2:15-2:55 p.m. Special Presentation: international REU program at the National Institute for Materials Science Tsukuba, Japan
Presenters: Robert Accolla, Robert Chrotowski, Maya Martirossyan, John Nance, Skye Tackett, Cooper Thome,

2:55-3:10 pm Break

Session G Global Learning Center Room 222
Moderator: Melanie-Claire Mallison, Cornell University
G.1 3:10-3:20 p.m. Mr. Richard Jiang KY MMNIN
Modification of Signal Propagation Velocity through Printed Circuit Boards by Using High Dielectric………Page 21
G.2 3:20-3:30 p.m. Mr. Daniel Teal CNF
*Piezoelectric RF SAW-Base Energy Detector*s………………………………………………………………Page 37
G.3 3:30-3:40 p.m. Mr. Daniel Goto KY MMNIN
Articulated Four Axes Microrobot……………………………………………………………………………….Page 19
G.4 3:40-3:50 p.m. Mr. Nicholas Theut SENIC
Stretchable Electronics: Processing and Analyzing P3HT-PDMS Devices………………………Page 38
G.5 3:50-4:00 p.m. Ms. Grason Gasser KY MMNIN
Dielectrophoresis for Particle Subpopulation Analysis………………………………………………Page 19

4:00-4:15 p.m. Break

4:15–4:45 p.m. Special Presentation: Fellowships and other Funding Opportunities
Presenter: Dr. Lynn Rathbun, Cornell University

4:45 p.m. Adjourn
Evening at Tech Rec 6:30 to 9:00 pm – Dinner and game room

Tuesday August 8, 2017 Global Learning Center

8:30 – 8:35 AM Welcome Room

Session H: Global Learning Center Room 222

Moderator Leslie O'Neill, Georgia Institute of Technology

H.1 8:35-8:45 a.m. Mr. Syed Nabeel Shah CNS

Laser-activated Thermoplasmonic Substrates for Intracellular Drug Delivery…………….Page 35

H.2 8:45-8:55 a.m. Mr. Gabriel Guisado CNF

Bacterial Mechanics on a Chip……………………………………………………………………….Page 20

H.3 8:55-9:05 a.m. Mr. Thomas Leonard TNF

HCG Optical Characterization………………………………………………………………………….Page 39

H.4 9:05-9:15 a.m. Ms. Lilia Escobedo MANTH

*Fabrication and Characterization of Ti3C2 MXene Electrodes for Studyi*ng *Neural Circuits*……………………………………………………………………………………………..Page 17

H.5 9:15-9:25 a.m. Ms. Minh-Chau Le CNS

Prevention of Occlusion and Cell Adhesion in 3D-printed, Liquid-infused Tympanostomy Tubes………………………………………………………………………………….Page 24

H.6 9:25-9:35 a.m. Mr. Michael D'Agati TNF

Channels for Optical Chemical- and Bio-sensors…………………………………………………..Page 15

9:35-9:50 a.m. Break

9:50-10:50 Special Presentation – Career Panel

Guests presenters: Dr. Miles Sakwa-Noval-Global Thermostat; Dr. Samantha Andrews-Associate Director of instruction at Project Lead the Way; Mr.Thomas Johnson-Averette-IEN Process Equipment Engineer III;

Session I: Global Learning Center Room 222

Moderator: Jamey Wetmore, Arizona State University

I.1 10:50-11:00 a.m. Mr. Yuji Okamoto NCI-SW & iREG Program

Vacuum Spray Deposition of ITO Nanoparticle Buffer Layer for Suppression of Sputtering Damage on Perovskite/Si Tandem Solar Cells……………………………………………………Page 30

I.2 11:00-11:10 a.m. Mr. Carl Felstiner KY MMNIN

Perovskite Solar Cells……………………………………………………………………………………..Page 20

I.3 11:10-11:20 a.m. Mr. Yuki Nakashima TNF & iREG Program

Printed CuInSe2 Nanocrystal Photovoltaic Devices (NIST)…………………………………….Page 28

I.4 11:20-11:30 a.m. Mr. David Lonstein TNF

Optical Properties of Buried High Contrast Gratings…………………………………………….Page 24

I.5 11:30-11:40 a.m. Mr. Brendan Noone KY MMNIN

Exploring Strategies to Effectively Fabricate Conductors within 3D Printed Plastic Components……………………………………………………………………………………………..Page 29

11:40-1:00 Box Lunch and Site photos

1:00-2:00 p.m. Special Presentation: Societal Issues of Nanoscale Science and Engineering

Presenter: Dr. Jameson Wetmore, Arizona State University, Associate Director for Social and Ethical Implications, NNCI

2:00-2:10 Break

Session J Global Learning Center Room 222
Moderator: *Nancy Healy, Georgia Institute of Technology*

J.1 2:10-2:20 p.m. Mr. Aaron Svidunovich MONT
Environmental Effects on SU-8 Flexures in MEMS Devices……………………………………………Page 37
J.2 2:20-2:30 p.m. Mr. Ryan Silva KY MMNIN
Gas Microfluidics using MEMS Micro-pumps…………………………………………………………….Page 36
J.3 2:30-2:40 p.m. Mr. Michael Klaczko CNF
Probing the Potential of Nanostructured Polymer Brushes………………………………………..Page 23
J.4 2:40-2:50 p.m. Ms. Robin Peter SHyNE
Design and Fabrication of Micro-Tip Arrays for Nano-Scale Atom-Probe Tomography……Page 32
J.5 2:50-3:00 p.m. Mr. Benjamin Wollant MONT
Characterization of SU-8 Spin Coating over Wafer Topography…………………………………Page 40

3:00-3:20 p.m. Move to Marcus Nanotechnology Building for Poster Session

3:20-3:25 p.m. Set up Poster Session 1

3:25-4:10 p.m. Poster Session 1

4:10-4:15 p.m. Set up Poster Session 2

4:15-5:00 p.m. Poster Session 2

5:00 p.m. Wrap up and adjourn

Evening on your own

2017 NNCI REU Convocation
at the
Georgia Institute of Technology

Presentation Abstracts
in Alphabetical Order

Piezoelectric RF SAW -Based Energy Detectors

Laser Assisted Patterning of Free Standing Polystyrene Thin Films

Abe, Hiroya
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NNCI Site: KYMMNIN University of Louisville, Louisville, KY
International Research Experience for Graduate Students
REU Principal Investigator: Dr. Kevin Walsh, Electrical and Computer Engineering, University of Louisville
REU Mentor: Dr. Robert Cohn, ElectroOptics Research Institute & Nanotechnology Center, University of Louisville
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Suspended polymer fibers (bridges) can have wide potential applications in micro/nano electronics and optics. Polymer fiber bridges can serve as optical guides, high surface area reactive chemical sensors and mechanical sensors. As previously reported [J. Rathfon et al.] fabrication of suspended polymer fibers utilized self-assembly method, where polymer fiber bridges were formed from suspended polymer thin films on microfabricated pillar structures via holes nucleation during thermal annealing. However, since the holes formation occurred randomly when the thin film is melting, the random array of the branched and torn fibers were observed. In this work, we report regarding laser-assisted process that allows controlled formation of the well-ordered patterned array of the polymer fibers. Updated technique includes additional step where the holes in the suspended film are formed by laser ablation. The thin films (no more than 100 nm) made from polystyrene (PS) and dye (sudan red 7b) solution were suspended onto the micro-scale pillars, and then the film was patterned with 532 nm pulse laser. After the patterned films were annealed at approximately 60 ˚C for 1 min, an ordered arrays of PS fiber bridges were obtained at high yield compared with previous methods.

Small Pyramids for Light trapping in Silicon-Based Heterojunction Solar Cells

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Huxel, Justin: Chemistry, Central Arizona College

NNCI REU Site: NCI-SW Arizona State University, Tempe, AZ
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NNCI REU Mentor: Som Dahal, School of Electrical Computer & Energy Engineering, Arizona State University
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A polished silicon surface reflects ~40% of incident light; through surface texturing the amount of light reflected is decreased to ~10%. Currently, conventional texturing processes using dilute KOH result in pyramids of about 3-5 µm base dimension. The present work was focused on developing a process for creating small, uniform pyramids of ~1 µm across the surface of silicon wafers. The goal of this project was to determine if small pyramids were both effective in light trapping and reducing the surface recombination after surface passivation. By decreasing the size of the pyramids, we can significantly reduce the surface area compared to conventional texturing which should significantly lower the surface recombination and improve overall minority carrier lifetime. The texturing chemistry and texturing time were varied to achieve the small pyramids and the reflectance was compared with the baseline process

of ASU's Solar Power Lab. We compared the pyramid size, the minority carrier lifetime after surface passivation and reflectivity of our samples. Our results indicate that samples with the pyramid size of 1-1.75 µm have the highest minority carrier lifetime without compromising reflectance.

Fluorescent DNA-Binding Proteins for Single-Molecule Visualization

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REU Mentor: James E. Baker, Chuang Tan
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Single molecule manipulation techniques are powerful tools to examine the physical properties of nucleic acid biopolymers. Although the width of DNA is well below the diffraction limit, many remarkable studies have been achieved by monitoring microsphere handles attached to the nucleic acids, including investigations of the mechanical properties of DNA and RNA, interactions between DNA and vital molecular motors, and the ability of topoisomerase enzymes to maintain DNA topology. Combining single-molecule manipulation techniques with fluorescence visualization methods may open new pathways for future nucleic acid studies. Novel Fluorescent DNA-binding proteins have recently been developed and could serve as a reversible method of visualizing DNA directly. This study aims at complimenting single molecule manipulation techniques with fluorescent DNA-binding proteins. *E. coli* cells were transformed in order to purify fluorescent DNA-binding proteins of interest. These proteins were then used in simple single molecule experiments to verify viability. Fully characterized proteins will be incorporated into future single-molecule manipulation studies to enhance measurement capabilities.

Improvement of Hydrothermal Synthesis of Lithium Ion Battery Cathode Nanoparticles

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NNCI REU Site: SHyNE Resource, Northwestern University, Evanston, IL
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Lithium transition-metal phosphates ($LiMPO_4$, where M = Mn, Co, Fe, Ni) have been identified as potentially viable cathode materials in lithium ion batteries due to their low cost, energy density and limited environmental impact, but these materials do have multiple drawbacks. Utilizing manganese and iron in the form of $LiFe_xMn_{1-x}PO_4$, however, has proven to combine both the optimal redox potential of manganese and the high electron and ion conductivity of iron. Developing this material as regularly-shaped nanoparticles provides structural stability, a greater capacity and a more efficient discharge of

energy for the battery. In this project, $LiFe_xMn_{1-x}PO_4$ nanoparticles were synthesized hydrothermally with varying ratios of manganese to iron and at varying temperatures to observe the impacts of these parameters on the size and shape of the resulting nanoparticles. The powders were tested for their phase composition, morphology and crystal structures in order to determine which set of parameters produced single-crystalline nanoparticles.

Improving Electrical and Optical Properties of Thin Silicon Solar Cells Utilizing Nanopillars by Silica Nanosphere Lithography and Metal Assisted Chemical Etching

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Graham, Alyssa: Applied Science, Central Arizona College

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Thin silicon wafers (< 100 µm thick) are less expensive to manufacture than regular silicon wafers (200-300 µm thick), and thus have generated substantial interest in the solar power industry. Using thin wafers for solar cells decreases the cost of manufacturing, but also decreases the absorption of the longer wavelengths of light. Much research has been conducted using nanopillars to alleviate this problem, but current fabrication methods are time-consuming and expensive. We studied the use of a combination of silica nanosphere lithography and metal-assisted chemical etching as a faster and less costly method for fabricating nanopillars. The effects of varying processing parameters on nanopillar fabrication will be reported, together with reflectance data from finished samples.

Thermal conductivity measurement of polymer fibers using the four-probe method

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Electronic components rely on the passage of electric current to perform their duties. Since current flow through a resistance is accompanied by large amounts of heat generation, they become potential sites for excessive heating. The heating of these components can cause them to malfunction or even stop working.

It is very desirable to develop devices using materials that efficiently control heat flow to protect heat sensitive components. Previous studies show that thermal rectifiers can control the amount and direction of heat flux. In most applications, thermal rectifiers are used as efficient coolers, since they can conduct heat efficiently in one direction carrying heat away from heat generating components and insulate in the other direction insulating heat sensitive components. The design of efficient thermal rectifiers requires knowledge of the thermal conductivity of many materials. In this work, a detailed procedure for manufacturing a thermal conductivity measurement device is presented. This device has four suspended heater lines composed of platinum and silicon nitride which allows us to calculate the thermal conductivity of the desired material. The procedure includes the use of metal depositions, spin coating photoresist, and etching. We used an optical microscope (OM) to observe the final geometry of the device. The OM images demonstrate that the procedure presented here is reliable to obtain a device that can be used to measure the thermal conductivity of various materials such as polymer fibers. In the future, this device will be used to investigate if the polymer fiber synthesized by Prof. Wudl's group behaves as a thermal rectifier.

Hydrogel Probes for Atomic Force Microscopy

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Atomic force microscopy (AFM) is used to measure forces and image surfaces with atomic resolution. The probe used in AFM consists of a cantilever, tip, cantilever holder. Traditionally, cantilevers have been fabricated using silicon because of established protocol that achieves high resonance frequencies and is less susceptible to vibrational noise. While silicon probes are useful for AFM, there are many disadvantages such as (1) inability to image at fast scan rates, (2) artifacts and distortions damaging the sample, (3) short tip life, (4) limited tip geometry and aspect ratios that can be achieved, (5) and limited mechanical tunability. Thus, an ideal probe would be able to image at a fast rate, minimize damage to the sample, have a longer tip life, achieve different tip geometries and aspect ratios, and have tunable mechanical properties. To address these limitations, we will generate hydrogel probes based on polyethylene glycol diacrylate (PEGDA). Specifically, we will improve upon previous work on to batch-fabrication of PEGDA probes. We plan to vary different parameters such as the polymer to photoinitiator weight percent ratio and time the tips undergo oxygen plasma ashing to determine their effects on mechanical properties and tip radii. In doing this, we hope to observe and accomplish a wider achievable range for mechanical properties and sharper tip radii for more accurate measurements. We also plan to expand tip geometries and radii not currently achievable in previous literature to include larger spherical radii for biological studies by ink-jet printing precise droplet volumes. Altogether we aim to complement silicon probes with hydrogel probes to address the increasing demand to study biological samples using AFM.

Design, Fabrication and Testing of Microfluidic Devices for Biochemical Analysis

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Mathematics & Physics, Vassar College

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Microfluidic devices have been commonly constructed using poly-di-methyl siloxane (PDMS), which is a soft elastomer, optically transparent, and bio-compatible. Prior fabrication techniques have demonstrated that a major limitation of PDMS when used for bioanalysis is the affinity of PDMS to small hydrophobic molecules, which have led to biomolecule absorption from the medium, biasing the experimental condition. In this experiment, a microfluidic system was constructed using a combination of semiconductor fabrication techniques and soft lithography procedures. PDMS was utilized to protect the silicon oxide microchannels formed on silicon wafers. Fluorescence detection was applied to test the microfluidic devices for biochemical analysis

Investigation of Atomic Layer Deposition for Distributed Bragg Reflector Mirror Stack

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Light extraction efficiency of Deep Ultraviolet (DUV) Light-Emitting Diodes (LEDs) is currently limited by a high number of photons being emitted within a range of angles such that they experience total internal reflection within the device. High reflectance Distributed Bragg Reflectors (DBRs) have been proposed as a vehicle for increased light extraction efficiency of DUV LEDs. These DBRs consist of a mirror stack of alternating materials with high and low indexes of refraction. The materials used also require a wide bandgap to avoid photon absorption. The current standard method of fabrication involves using molecular beam epitaxy to grow different varieties of AlGaN for the mirror stack. Here, we investigate the use of atomic layer deposition (ALD) as an alternative method for building DBR mirror stacks for increased light extraction of DUV LEDs.

Metasurfaces for Optical Power Limiting

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NNCI Site: TNF The University of Texas at Austin
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REU Mentor: Nishant Nookala, Electrical and Computer Engineering, The University of Texas at Austin
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In recent years, plasmonic metasurfaces have been introduced and demonstrated to provide a great degree of control over the amplitude and phase of incident optical fields. Such metasurfaces are comprised of arrays of nano-scale metallic inclusions that act to confine light to sub-wavelength volumes, and whose spectral response is largely dependent on the geometry of the individual nano-resonators. Introducing an optical dipole transition, such as an intersubband transition (IST) formed via confined states in a semiconductor heterostructure, tuned to the cavity resonance of the resonator results in strong coupling between the competing cavity and dipole resonances. A corresponding polaritonic splitting occurs where the overall spectral response of the metasurface is both dampened and split. Changing the IST position by Stark tuning via an applied voltage bias allows one to transition between weakly coupled and critically coupled metasurface resonances when the IST is tuned towards and away from the cavity resonance, respectively. Such metasurfaces can be employed as flat electro-optic modulators or tunable filters. To demonstrate this effect, in this work, we designed and fabricated a metasurface consisting of a 400-nm thick multi-quantum-well semiconductor layer sandwiched between a metallic ground plane and surface. Individual nano-resonators were designed, patterned, and etched to form three-dimensional dipole cavities. We observe that the unbiased spectral response of dipoles with geometric resonance tuned to the IST at 38 THz exhibits two polaritonic branches separated by ~11 THz, as expected. In the future, we hope to extend this work by observing our device performance under applied bias.

Optimization of Low-loss Lithium Niobate Nanowaveguide Fabrication

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Lithium niobate's wide bandgap, high bandwidth, and large second order electro-optic coefficient make it a favorable material for electro-optic devices despite its scalability limitations. Our group has succeeded in creating nanoscale LN waveguides which show significant improvement from previous fabrication efforts. However, the current dry etching process gives way to significant surface roughness and a large loss of efficiency. Here, we explore different parameters and factors to improve the quality of the dry etching step in our fabrication process. We evaluate the smoothness and etch depth using SEM and profilometer measurements. The results of this project will lead to the creation of more efficient devices with powerful optical communication applications.

Photoconductivity in Rare-earth Doped Nanocrystalline Titanium Dioxide

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REU Mentors: Dr. Bruce Alphenaar, Electrical and Computer Engineering, University of Louisville & Dr. Francis Zamborini, Analytical Chemistry, University of Louisville
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Nanocrystalline titanium dioxide (TiO2) is commonly used in renewable energy applications like dye-sensitized and perovskite solar cells, so developing methods to improve its optical, electrical, and structural properties has been a focus of studies aiming to improve the performance of these devices. Doping the TiO2 layer of dye-sensitized solar cells with rare-earth oxide microparticles has previously been shown to increase its conductivity by 40-50 times, contributing to a 20-30 percent improvement in solar power conversion efficiency. To understand the mechanism behind this increase in conductivity and optimize the doping process to achieve greater solar cell efficiencies, this study investigates the density of states of erbium and neodymium oxide doped TiO2 electrodes through cyclic voltammetry. Cyclic voltammagrams of doped samples at varied voltage scan rates show a shift in the location of the peak associated with grain boundary defects, greater variation in the peak location with changing scan rate, and a current plateau at slower scan rates. These features suggest doping results in slower trapping rates and a change in the nature of available trap states, which both influence the conductivity and could be explained by the rare-earth oxides filling trap states deep in the TiO2 band gap.

Microfluidic Channels for Optical Chemical- and Bio-Sensors

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Department of Electrical and Computer Engineering, Stony Brook University

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With recent advances in optoelectronics and photonics, optical biosensors have flourished in the area of healthcare. Optical biosensors are typically built of three parts: a light source and detector, a photonic crystal substrate, and an analyte flow mechanism. Currently, a benchtop system can be used to do biosensor testing; however, this is not useful for an end user such as a nurse who would be handling the system on a day-to-day basis. A portable system for biosensor testing would be much more practical and would therefore need to be automated to allow for repeatable and reliable results. This project demonstrates bonding between polydimethylsiloxane (PDMS) layers to create microfluidic channels for analyte flow. The 250µm PDMS layers were obtained from Grace Bio-labs and had laser cut designs completed by the Harvard Center for Nanoscale Systems. A Nordson March PX-250 Asher was used to bond the layers together by oxygen plasma with low power (<20W), low oxygen flow (<15sccm), and short time (30sec). A custom-built apparatus was used to align the layers and provide force during the bonding

procedure. For testing, dyed water was flowed through the resulting microfluidic channels to ensure there were no leaks. Also, the microfluidic channels were used in combination with a photonic crystal waveguide and the benchtop system. This test demonstrates how the PDMS channels would be used to make a portable system more reliable and repeatable.

Synthesis and Characterization of Crosslinked Lysozyme-Dextran Nanogels for Drug Delivery

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Core-shell nanogels are promising vehicles for drug delivery due to their stability, versatility, and drug-loading capabilities. In this study, lysozyme-dextran core-shell nanogels were synthesized through a green, two-step process involving a Maillard reaction and a heat-induced gelation. To tune the mechanical properties of these particles, 1, 12 – diaminododecane, 4,9 – dioxa – 1, 12 – dodecanediamine, and 2, 2' - (ethylenedioxy)bis(ethylamine) were used to promote intra-nanogel crosslinking. Crosslinking was shown to influence the size of nanogels while maintaining their size uniformity. Crosslinkers were also found to be internalized by the hydrophobic core of the nanogel, much like a drug. The Young's modulus of nanogels was determined through nanoindentation AFM, and produced moduli in the single megapascal range. The observed deformability and spreading of the nanogels indicate a more complex, two-component system may be present.

Examining Polymer Crystallization using Raman Spectroscopy and Polar White Light

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Geomatics and Advanced Mathematics, Nicholls State University

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Kevlar (para-aramid) polymer fibers are strong and tough due to their high degree of crystallinity and distribution of crystalline and amorphous domains. While previous studies have quantified crystallinity using x-ray diffraction, it does not provide a means of analyzing crystalline distribution. Here we use Raman spectroscopy to quantify crystallization percentage and distribution of Kevlar 29 and Kevlar 49 fibers. Both fibers had an amorphous skin and a crystalline core; however, the Kevlar 49 has a larger crystalline core diameter due to its higher percentage of crystallinity. Understanding the crystallinity and distribution of the fibers will help understand the mechanisms of polymer crystallization and fiber failure.

Fabrication and Characterization of Ti_3C_2 MXene Electrodes for Studying Neural Circuits

Escobedo, Lilia
Chemical Engineering, Cornell University

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Implantable electrode arrays, devices which can be used to locate where seizures come from in the brain, are important tools used to study and treat epilepsy. However, their performance is limited by their rigidity and relatively large size. Recently, flexible micro-scaled electrode arrays made with Ti_3C_2 MXene that were fabricated and tested in the Litt lab have shown great promise due to their remarkably low impedance. Nevertheless, incorporating MXene, a two-dimensional metal carbide with electrical properties similar to those of graphene, into micro-patterned electrode arrays has never before been demonstrated and has required significant innovation and optimization of the fabrication process. This study presents the optimization of the dry liftoff step, which uses a sacrificial layer of Parylene C to produce micropatterns of MXene.

Perovskite Solar Cells

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The performance of perovskite solar cells (PSCs) has been rapidly advancing in the past decade, however, most fabrication processes use high temperatures (~500 °C) to sinter TiO2 films. This study focuses on improving the scalability of PSCs by fabricating the TiO2 layers at low temperatures (<100 °C), making them compatible with flexible substrates and roll-to-roll manufacturing. Various TiO2 architectures were compared using different combinations of intense pulsed light sintered compact and mesoporous layers, as well as a thin pore-filling layer, all of which were fabricated at low temperatures on flexible substrates.

Optimal Resolution of Two Photon-Lithography: A Voxel Study

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Two photon-lithography has provided the means for high spatial resolution of three dimensional structures in micro and nanofabrication[1]. This study explores a 3D laser lithography system, the *Photonic Professional GT,* for exposure latitudes on the negative photoresist IP-Dip. Ladder-based structures were printed to test for optimal resolution based on laser power (LP) and scanning speed (SS). Ranging from 10 to 100 % power and 1,000 to 100,000 µm/s, higher scanning speeds have been observed to require higher laser power to obtain better resolutions. Characterization tools like the FEI Quanta 600 ESEM captured line width, height, and aspect ratio for voxel dimensional analysis. Graphs were then plotted to illustrate trends and provide references to future users in maximizing structure quality.

Dielectrophoresis for Particle Subpopulation Analysis

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Dielectrophoresis (DEP) has been well-researched for its purposes in analyzing, manipulating, and separating micro-particles by subjecting such samples to a non-uniform electric field. The disadvantage of traditional designs is that they create a non-uniform dielectrophoretic force ($FDEP$). For the purpose of particle analysis, this factor must remain constant in order to accurately assess a particle's dielectric properties. The work in this study explores a new kind of platform termed isomotive dielectrophoresis (isoDEP), which aims to analyze subpopulations by subjecting particles to a uniform DEP force. A conductive version of this platform has been previously fabricated by our group using doped silicon; however, in order to greatly reduce the device cost, an insulative device was created and will be the subject of discussion. The unique geometry of the device works to keep the gradient of the field-squared ($\nabla E2$) and, thus, DEP force constant. Two devices were fabricated. The first contains a Su-8 microchannel over pre-fabricated electrodes and the second consists of a PDMS/Ag-PDMS structure fabricated using soft lithography where the embedded silver particles serve as an alternative to metal electrodes for the device. The devices were tested using polystyrene particles and phytoplankton (8-15um) to analyze their electrokinetic response.

Articulated Four Axes Microrobot

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The purpose of this research is to investigate a new method of actuation for the AFAM (articulated four axes microrobot). The AFAM is a miniature, millimeter scale device constructed using Micro Electro Mechanical (MEMS) technology, and capable of manipulating micro and nanoscale components in 3 dimensions. In the current design of the AFAM, pitch of the arm is controlled by an electrothermal actuator. The design in this research is an electrostatic rotary actuator that uses four sets of curved combs. The research focused on modeling how the number of fingers, spring length, and spring thickness affect the performance of the actuator, and on the fabrication process of the AFAM on SOI (silicon on insulator) wafers. In the fabrication process, a DRIE (deep reactive ion etching) recipe was used to etch the 50 micron device layer of the SOI wafer. By adjusting the passivation time and duration of etching, we found that we can achieve vertical etching with minimal undercutting.

Bacterial Mechanics on a Chip

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CusCBA is a system of proteins that span the bacterial envelope for efflux of metal ions. Under mechanical stimuli, the diffusion of CusA (the inner membrane portion) is altered, which relates to the assembly of this pump. To better understand bacterial mechanics and its relation to these proteins, we load bacteria into a microfluidic device. At the CNF, we utilize DUV lithography and dry etching to create our fused silica device. The device contains groupings of tapered channels with inlets large enough for cell entry and outlets approximately a quarter of the cell diameter (250 nm). The pressure at the inlet of these tapered channels is higher than the outlet, creating a pressure drop across trapped bacteria cells. This form of extrusion loading creates a non-uniform stress across the cell. Using super-resolution microscopy, the diffusion of CusA was observed in comparison to pressure drop across the channels and how far the cells traveled in the tapers.

Optimal Strength Nano-Cellular Materials

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As most brittle materials decrease in thickness, their rupture strength will increase. A beam bending test is most common way to find a specimen's rupture strength by supporting a horizontally laid specimen across 2 low friction contact points and applying a load to the top and center of the beam. The beam bending test is an excellent test at the macroscale, but our project is to characterize the rupture strength of a thin, brittle material at the nanoscale. In this project, we will be mimicking the macroscale beam bending test with our own microscale beam bending test using the Nanoscribe to design our structures and Atomic Layer Deposition(ALD) to coat the structures with a thin film of alumina. After structures are constructed and coated, the internal polymer will be removed using an O2 plasma etcher leaving us with a hollow structure of alumina. A load will be applied to the top, center of the structure allowing us to characterize the rupture strength.

Can We Increase Concrete Durability with Nanoparticles?

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Previous studies have well demonstrated that photocatalytic cement, titanium dioxide-doped (TiO_2), can reduce nitrogen oxide species(NO_x) in air. In the presence of UV light, TiO_2 catalyzes reactions that oxidize NO_x to nitrite (NO_2^-) and nitrate (NO_3^-) species on the surface of the cement. Calcium nitrite or nitrate have previously been added to cement to work as corrosion inhibitors. This project aims combine to these concepts and examine these converted species can act as corrosion inhibitors.
The cement was exposed to three different conditions: 1) Without NO gas or UV light; 2) With NO gas, but not UV light; and 3) With both NO gas and UV light. Little information could be discerned about the fate of the nitrogen with the following tests: fourier transform infared spectroscopy (FTIR) paired with thermogravimetric analysis (TGA), x-ray diffraction (XRD), and x-ray photoelectron spectroscopy (XPS). Samples also were subjected to ultraviolet-visible spectroscopy (UV-Vis) to measure for nitrites and/or nitrates. Unexposed samples were free of either nitrite or nitrate, while both compounds present in the exposed samples. However, the concentration of the species found in those exposed samples did not match theoretical calculations. It is hypothesized that these discrepancies are due to the samples absorbing the NO even when not exposed to the UV light. Further tests must be conducted to discover the true fate of the nitrogen.

Modification of Signal Propagation Velocity through Printed Circuit Boards by Using High Dielectric Constant Materials

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Signal propagation velocity through a PCB (printed circuit board) is a direct function of the dielectric constant of the material surrounding the conductor. The typical signal velocity for common FR4 PCB substrates is approximately 15 cm/ns. Conductor lengths and dielectric constants therefore determine the time delay from the output of one device to the input of another device on a PCB. Currently, timing skew between signals is minimized by using serpentine patterns to lengthen shorter traces to match longer traces. This practice sacrifices valuable PCB area that could have been used for other components or signal routing.

The work here tests the concept of modifying the dielectric near a conductor to reduce the signal velocity of shorter traces rather than physically lengthening them. A reference microstrip trace on standard FR4 dielectric is compared to a trace placed over a trench filled with various materials of different dielectric constants. The timing skew between the reference trace and test trace is then compared. Measured results indicate that the signal velocity can be altered using a "trench-and-fill" method of replacing standard PCB substrate with higher or lower dielectrics. The ratio of: Velocitytest/Velocityref = 0.68 to 1.67 was obtained by reducing or increasing the dielectric constant. The standard PCB manufacturing process is then considered to determine possible entry points for a "trench-and-fill" dielectric modification process.

Transistor Process Optimization for Micro-fabrication Courses

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Consistency and reliability are key points that are sought in the micro-fabrication process. For micro-fabrication courses such as the one at Montana State University this concept is important, since being able to consistently produce products that work as desired helps to create a better learning experience. The transistors that are produced in these courses are affected by the doping concentration and dopant profiles of the fabricated wafers, which are made using the method of diffusion. In order to ensure that the profiles and therefore the transistors are created properly, we studied the impact of diffusion and drive-in temperatures on the process. Phosphorus diffusion and drive in temperatures were varied, and the dopant concentration and depth profile were subsequently measured using Time-of-Flight Secondary Ion Mass Spectrometry. This was compared to measurements of previously made transistors, and to theoretical calculations, to quantify the dependence on these temperature parameters and identify better target values for the transistor process. As a result of the analysis and tests, a more reliable transistor process can be used to improve course learning, lab troubleshooting, and circuit testing.

Laser Induced Bubble on Plasmonic Topography for Nanoparticle Assembly

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Plasmonic substrates with gold nanopyramids fabricated through photolithography have been shown to be efficient at generating bubbles under femtosecond irradiation. By using the Nanoscribe 3D laser setup, we systematically examine how laser intensity, scanning parameters, and scanning geometry affect bubble properties, including the maximum bubble size, lifetime, and generation rate. By tuning the laser intensity and the scanning speed, we have found the optimal settings for bubble size. This permits us to control bubble formation to eventually control particles near surface.

Probing the Potential of Nanoscale Polymer Brushes

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Polymer brushes have been of interest because of their unique ability to act as a functional coating. The unique structure of polymer brushes allow them to be used for several applications including antifouling, resistance to nonspecific binding, and biosensing. With the realized applications of polymer brushes, it has become more important to understand their fundamental structure and the resultant stimuli-responsive behavior. This project aims to create a way to observe the polymer brushes at the molecular level by putting polymer brushes onto the sidewalls of diffraction gratings. Once done observation can be done on the macro scale by observing the diffraction of light through the gratings, and on the molecular scale by observing the reflection of neutron/x-ray beams across the sides of the diffraction gratings. By utilizing photolithography patterning on a sandwich structure on top of a fused silica wafer, the process to reaching this goal has been furthered.

Prevention of Occlusion and Cell Adhesion in 3D-printed, Liquid-infused Tympanostomy Tubes

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Tympanostomy tubes, or ear tubes, are the most common solution to relieve the symptoms of otitis media (OM), or middle ear infection, in the U.S. Most commercial ear tubes are made from silicone or fluoroplastic. While about one million ear tubes are implanted annually, approximately 7% to 37% of them fail due to occlusion caused by the adhesion of mucus, keratinocytes, or bacterial biofilms. Utilizing three-dimensional (3D) printing techniques, and a surface modification technique termed *slippery, liquid-infused, porous surface* (SLIPS), this project aims to create ear tubes that will prevent occlusion by cells and biofilms while enabling fluid flow comparable to that through commercial tubes. Ear tubes and surfaces for characterization were 3D-printed using a two-part curing polydimethylsiloxane (PDMS) elastomer and cured at 80°C for two hours. SLIPS surfaces were created by infusing the tubes with silicone oil of viscosities ranging from 10 to 100cSt at room temperature for 48 hours. The SLIPS sheets were characterized with a sliding angle test. The SLIPS surfaces showed significantly lower sliding angles than control silicone and fluoroplastic surfaces as well as 3D-printed PDMS sheets without SLIPS surfaces. Keratinocytes cultured on the SLIPS surfaces were characterized using live/dead stain for cell adhesion at day 4, and cytotoxicity assay for cytotoxicity at day 7. The SLIPS surfaces showed significantly lower cell adhesion and similar cytotoxicity compared to control surfaces. Fluid flow through the SLIPS ear tubes was tested by measuring the pressure drop across the ear tube at a constant fluid flow rate of 20 mL/min. SLIPS ear tubes showed improved or comparable performance compared to commercial ear tubes. These results show promise for 3D-printed, liquid-infused tympanostomy tubes to provide a more effective solution for OM patients.

Optical Characterization of Epitaxially Encapsulated Submicron SiO_2 Gratings in MBE-grown GaAs

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High-contrast photonics is a re-emerging field that has demonstrated a host of interesting optical phenomena, such as broadband perfect reflection and transmission, ultra-thin lensing, and plasmonic-like local field enhancement. The core engineering in high-contrast photonic structures integrates two or more optical materials with a low- and high-index of refraction, respectively. Shaping these materials into subwavelength structures, such as gratings, enables high-contrast photonic structures that can achieve the aforementioned phenomena via coherent phase and/or polarization interference. Most current approaches use air as the low-index "material" or non-encapsulated gratings.

Recently, we expanded these approaches to epitaxial III-V semiconductors and demonstrated single-crystal lateral overgrowth and planarization of all-MBE -grown GaAs over sub-micron SiO_2 gratings. While demonstrating good structural quality, the optical quality of the MBE-grown GaAs over encapsulated SiO_2 gratings remains largely unexplored. With optoelectronic and photonic application as the goal, high optical quality material growth is paramount.

Current 1.4 micron pitch encapsulated silica grating structures were optically characterized using Fourier transform infrared (FTIR) spectroscopy. FTIR showed interference fringing between the grating and the planar top surface. To test material quality a single quantum well, AlAs/$In_{.15}Ga_{.85}As$/GaAs, was embedded in the GaAs overgrowth layer in both the control and the grating sample. Photoluminescence (PL) spectroscopy indicated carrier recollection as a method of enhancing PL in the sample because the bulk GaAs peak suffered a 5-fold reduction but the quantum well peak improved by 40% with gratings. This indicated that tailored sub-wavelength encapsulated metastructures could enhance the emission rate and efficiency of a quantum emitter.

Development of Automated Opto-Thermal Characterization System

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The measurement of thermal conductivity (κ) in materials can be divided into two methods: contact and non-contact based. Both methods have their drawbacks when used on nanomaterials or materials with high κ. Contact based methods become very difficult to use with high-κ materials as the contact resistance often surpasses the low intrinsic resistance of the sample, giving inaccurate measurements. Non-contact based measurements such as optical measurements often have large uncertainties, poor temperature sensitivity, or complex analysis. The purpose of this effort was to develop a reliable form of non-contact based measurement of thermal transport using Raman spectroscopy and Brillouin Light Scattering (BLS) in conjunction with one another. The system relies on a continuous wave 532 nm probe laser with constant intensity and a variable modulated heating source. The modulated heating – either from electrical or optical sources – provides a signal to which the Raman and BLS measurements can be locked onto in order to improve their sensitivity. This project provided the resources to develop programmatic control over sample positioning, ambient temperature, and probe and heating laser intensity. In addition, a LabVIEW program was developed to automatically measure the laser spot size using a modified knife edge method with suspended carbon nanotubes (CNTs). The improvements made to the opto-thermal characterization system can greatly assist in the research and development of high-κ materials, such as BAs, BN, and CNTs.

Low-loss Zero-index Metamaterials Induced by a Bound State in the Continuum

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Metamaterials with zero index of refraction exhibit remarkable optical properties, such as infinite phase velocity and refraction to the interface between zero and non-zero index medium. These properties provide many potential applications, including optical cloaking, super-coupling, and alternative methods for phase-matching in nonlinear optics. Such materials can be achieved by arranging an array of silicon pillars on a silica glass substrate. However, this set up may lead to radiative loss as light propagates through the material. Such loss can be eliminated by adjusting the pitch, radius, and height of the pillars to achieve a bound state in the continuum at the desired operating wavelength. Using this method, we attempt to fabricate a low-loss, zero-index metamaterial at an operating wavelength of 1550 nm from a 1.5-micron silicon on insulator (SOI) wafer. The fabrication process consists of using thermal oxidation to achieve the desired thickness of silicon on the SOI wafer, then using electron beam lithography to construct the silicon pillar array. We plan to characterize the real part of the effective index by measuring the effective wavelength of the interference light when there are two light sources incident from two opposite directions, and measure the loss of the metamaterial by imaging the decay in the out-of-plane radiated light as it propagating through the medium.

Graphene Liquid Cell for Live Bacterial Imaging using Scanning Electron Microscopy

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Producing nanometer resolution visuals of living biological cells has been restrained by harsh vacuum conditions used in electron microscopy (EM). Traditional procedures used to prepare biological cell samples for EM involve dehydration, fixation, and coating samples with conductive materials; all of which render biological cells nonviable. Microfabricated liquid cells utilizing SiNx membranes as an electron window have been used to encapsulate biological samples in liquid for wet environment EM. However, the use of SiNx usually results in low contrast and sample stability due to its relatively high thickness and low thermal and electrical conductivity. Graphene, an atomically thin, liquid impermeable, and conductive material is a potential alternative to SiNx liquid cells. The focus of this project was to produce a graphene liquid cell, where multilayer graphene acts as an encapsulating window material for enabling wet environment EM of live bacterial cells. E. coli and S. marcescens cells were transferred on to various substrates and covered in multilayer graphene in attempts to encapsulate the bacterial samples within liquid. Scanning electron microscopy was used to visualize samples and determine the effectiveness of graphene as a window material. After undergoing EM, samples were removed from their substrates and incubated to gauge whether the cells were still viable.

Nanostamp Optimization for Single-molecule DNA/Protein Array Studies

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Over the past two decades, biophysical single molecule DNA, RNA and motor protein studies have increasingly demonstrated the important role that the structural and mechanical properties of single molecules play in gene replication and expression. Specifically, genetic processes can be significantly affected when DNA or RNA experiences torque or protein interaction forces, which often happens in vivo. These studies use DNA/RNA "tethered" between a protein anchor on a surface, and a microbead that can be twisted or pulled with optical or magnetic tweezers. Nanostamping of protein spot anchor arrays, compared to blanket coating a substrate with protein for DNA/RNA anchoring, aids in single molecule studies by precisely controlling the DNA/RNA anchor position, which improves resolution and throughput of the technique. Thus we are exploring a nanostamping method called the "Ink Substract Print" method

by optimizing the dimensions of electron beam-patterned nanostamps that can selectively pattern arrays of 100nm wide circles of protein on a glass surface.

Understanding the Fundamentals of Hydrogen Evolution Reaction

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Clean-burning hydrogen fuel is an attractive alternative to fossil fuels, which currently dominate the energy economy. Currently, most hydrogen production is carried out via steam methane reformation and coal gasification. These processes produce carbon oxide pollutants and depend on finite and non-renewable fossil fuels. A small portion of industrial hydrogen gas is produced via water electrolysis – an environmentally clean process, which depends on Earth's virtually unlimited supply of water as feedstock. However, platinum metal, the current state-of-the-art catalyst for water electrolysis, is very rare and expensive making it unsuitable for large-scale hydrogen production operations. An emerging class of materials, transition metal dichalcogenides (TMDs), have been shown to possess promising properties in catalyzing the hydrogen evolution reaction (HER). However, TMDs currently suffer from relatively poor efficiencies and long synthesis times. The activities of these materials can be further improved by coupling with reduced graphene oxide (RGO). Here we attempt to shorten the hours-long synthesis of a TMD-RGO hybrid material, MoS_2-RGO, by replacing the lengthy high-pressure, high-temperature reductive treatment of the precursor materials with a facile intense pulsed light (IPL) treatment which can be carried out in minutes.

Optimization of CMOS-Compatible Zero-Index Metamaterials

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Light moving through a material with a refractive index of zero experiences an infinite wavelength and zero phase advance. Such exotic behaviors can be accessed toward a variety of applications, especially when these materials are integrated with silicon photonic devices. This behavior is highly desirable for photonic applications such as supercoupling, cloaking, and beam-steering. Our group has designed a metamaterial consisting of a square matrix of holes patterned using electron beam lithography onto a silicon substrate, which exhibits zero-index behavior at a wavelength of 1630 nm. However, the metamaterial is designed to exhibit zero-index behavior at a wavelength of 1550 nm. The discrepancy between theory and experiment is hypothesized to arise from deviations of the fabricated device from an ideal simulated model. This project systematically analyzes the fabrication procedure to determine the cause of the wavelength deviation, paying particular attention to the etch geometry and accuracy of two different reactive ion etching processes. Characterization of such features will help with the development of an improved theoretical model so that the obtained experimental zero-index wavelength can be reliably predicted.

Atomic Force Microscopy-based Mechanical Testing Reveals the Mechanisms of Plasticity in Disordered Nanoparticle Packings

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The plastic deformation mechanisms of disordered nanoparticle packings (DNPs) is investigated using the atomic force microscope. The atomic force microscope was used to perform nanoindentation and scratch tests on the surface of the DNPs. Topography scans of the surface were completed before and after the tests were executed. These tests were instrumental in determining how variations in environmental humidity level and bulk volume fraction affect the mechanics of plasticity of the samples. It was found that high humidity results in an increase in energy dissipated during the indent as well as an increase in the number of load drops. Load drops are believed to correspond to particle rearrangements such as shear transformation zones or T1 processes. Samples with higher volume fraction exhibited a higher hardness and effective modulus.

Printed Nanocrystal $CuInSe_2$ Photovoltaic Devices

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$CuInSe_2$ (CIS) nanocrystals are being investigated to fabricate low-cost photovoltaic (PV) devices using relatively mild fabrication processes. CIS is an ideal material for photovoltaics because it has a high absorption coefficient and contains no toxic elements like Cd or Pb. Photovoltaic devices using polycrystalline CIS have reached record efficiency of 19%. The methods used to form these polycrystalline CIS layers require high temperatures process, so we are developing methods to make CIS PV devices using CIS nanocrystal inks that can be solution processed. We synthesized CIS nanocrystal inks using previously reported methods and fabricate photovoltaic devices using relatively mild conditions [1]. However, the efficiency of our CIS nanocrystal thin film PV devices is relatively low, with a record PCE near 3%. The reduced efficiency is likely due to a low mobility lifetime product in the nanocrystal film. Therefore, we aim to investigate methods for improving the PV performance of CIS nanocrystal films to improve the efficiency of CIS PV devices.

First, we compared the efficiency of CIS PV devices before and after heating by using the furnace and intense light pulse treatments. As a result, we found that the efficiency was changed by heating. We then studied the effects of heating on the CIS layer by measuring the Raman spectroscopy and X-ray diffraction. In consequence, we found that heating the CIS nanocrystals is removing organics and changing the crystal structure of the material. It is likely reducing defects which is either surface defects or cation disorder.

[1] Panthani G. Matthew et. al *J. Am. Chem. Soc.,* **2008***, 130 (49),* 16770-16777.

Exploring Strategies to Effectively Fabricate Conductors within 3D Printed Plastic Components

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Many advances have been made in the field of additive manufacturing (AM) over the last two decades. The ever-evolving field of AM is no longer confined to simply printing demonstration and prototype parts out of plastics. New materials have been explored and novel 3D printing technologies have been developed so that it is now possible, for example, to fabricate fully functional parts and components out of metals using AM. However, in spite of all these amazing advances, no one has successfully addressed the challenges associated with printing “smart AM components” with embedded electronics, sensors, and conductive interconnects. This research addresses that need by focusing on novel strategies for embedding metal conductors inside 3D printed parts and components using an interesting post-print process. Our overall strategy involves printing plastic components with micro-fluidic channels where the conductors are desired. Then, using a series of post-print techniques, we filled these channels with conductive material to form the required electronic traces. Custom test coupons were manufactured out of acrylonitrile-butadiene-styrene (ABS). Fused filament fabrication (FFF) was the AM process. A series of tests were conducted to fill the micro-fluidic channels using a variety of strategies ranging from electroless plating, seed layer formation of various materials, electroplating using both in-channel and out-of-channel electrodes, and direct metal injection. The conductivity properties of the various materials were experimentally characterized for each process using 4 Point Probe resistivity analysis. Our best results were with a nickel seed layer followed by copper electroplating using an in-channel electrode. All of our tests are presented on the poster along with the advantages and disadvantages of each.

Transition Metal Sulfide Heterostructures for Hydrogen Evolution Reaction

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Transition metal sulfides are promising new materials that could replace commonly used and expensive platinum derivatives as electrodes for hydrogen production by water splitting. Layered molybdenum disulfide (MoS_2) has drawn great interest because it can be easily synthesized and exceptionally stable while possessing excellent mechanical, electronic, and catalytic properties. One challenge in utilizing MoS_2 is that the catalytically active sites occur predominantly on edges of the layers. This project focused on manipulating the morphology and structure of transition metal sulfide heterostructures composed of MoS_2 flakes grown on copper sulfide (Cu_xS_y) nanowire bases using a facile vapor deposition method. The controlled synthesis of these heterostructures was determined by altering growth parameters and finally identifying the best morphologies in promoting the concentration of edge active sites while maintaining the desired structure. Scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDS) were used to study morphology; X-ray diffraction (XRD) was used to demonstrate the phase transformation; Raman spectroscopy, X-ray photoelectron spectroscopy (XPS), and transmission electron microscopy (TEM) were used to further analyze the compositional characteristics of the heterostructure. The samples with desired morphology and structure will be finally subjected to a three-electrode electrochemical system for a hydrogen evolution test.

Vacuum Spray Deposition of ITO Nanoparticle Buffer Layer for Suppression of Sputtering Damage on Perovskite/Si Tandem Solar Cells

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Perovskite/Si tandem solar cells have attracted much attention because of their high theoretical power conversion efficiency. The top perovskite solar cell absorbs visible light from ~300-800 nm and the bottom Si solar cell absorbs near infrared light from ~800-1200 nm. A film of indium tin oxide (ITO) is often deposited by sputtering on the top perovskite solar cell to collect generated electrons but the sputtering process damages the perovskite solar cell, resulting in a decrease of cell performance. In this study, we

have tried to suppress the sputtering damage by depositing a buffer layer of ITO nanoparticles (NPs) by vacuum spray deposition before the ITO sputtering. Si wafers with a buffer layer of ITO NPs showed a smaller decrease in lifetime after the sputtering step than that without ITO NPs, which suggests that the buffer layer of ITO NPs is effective in suppressing the sputtering damage. A buffer layer of Si NPs was also tested, and those results will be reported as well.

Raman Spectroscopy and Transmission Electron Microscopy of Si_xGe_{1-x}-Ge-Si Core-Double-Shell Nanowires

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One-dimensional semiconducting nanowires are attractive components for nanoscale electronics and photonic devices due to their reduced dimensions and the ability to engineer their electronic properties. In this study, we investigate the Raman spectra, transmission electron microscopy (TEM), and energy dispersive x-ray spectroscopy (EDX) of epitaxial Si_xGe_{1-x}–Ge–Si core-double-shell nanowire heterostructures. The nanowires are synthesized using a combination of vapor-liquid-solid (VLS) growth mechanism for the core, followed by in situ epitaxial growth using ultra-high vacuum chemical vapor deposition for the shell. Gold droplets are used to catalyze nanowire core growth. Raman spectroscopy is used to probe the elastic strain associated with lattice mismatch between the core and shells. The Si_xGe_{1-x} core has a larger lattice constant and can be used as a non-planar substrate for depositing compressively strained Ge layers for high hole mobility, and tensile strained Si layers for high electron mobility. Planar and cross sectional TEM are used to probe the crystal structure, shell morphology, and to measure the shell thickness. The nanowires reveal a cylindrical core and faceted shells. Planar view TEM is used to determine the nanowire orientation by investigating diffraction pattern through Fast Fourier Transforms. The appearance of the diffraction pattern reflects the crystalline nature of the sample. Additionally, to determine the shell thickness and elemental composition, EDX measurements were acquired using line scans across the nanowires.

Design and Fabrication of Micro-Tip Arrays for Nano-Scale Atom-Probe Tomography

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A lack of optimization in specimen preparation currently limits widespread use of atom-probe tomography characterization. Guided by ideal APT specimen parameters and flaws in commercially available products, this study explores methods and processes involved in fabricating a micro-tip array for atom-probe samples. Construction of a two-tiered design composed of a tapered needle tip atop a sturdy base structure was attempted using microfabrication techniques including photolithography, vapor deposition, dry etching, and wet etching. Deep reactive-ion etching and wet KOH etching were investigated for effectiveness in producing the needle geometry required for field evaporation. While consecutive deep-trench and isotropic DRIE processes—in that order—were found to carve out the base of a cylindrical micro-post rather than sharpening it to a needle, reversing the two procedures is hypothesized to result in the desired geometry. SEM and profiler imaging demonstrate that anisotropic KOH etching can fabricate 120-µm-tall base structures beneath the constructed needle tips with sufficient SiN masking. These results illustrate that microfabrication techniques can produce advantageous complex-geometry microarrays for customizable use in APT analysis.

Nucleation Studies of Thin Film Oxides using Atomic Layer Deposition

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Atomic Layer Deposition is a nanoscale fabrication technique used to coat materials in thin film oxides for the semi-conductor industry. It is typically thought these oxide films show complete and conformal atomic level growth as soon as the process has started. Samples of Al_2O_3 and HfO_2 were chosen as precursors due to their heavy prevalence of use in industry wide applications. These oxides were then deposited on holey carbon support film (Cu substrate) TEM grids, and analyzed at 1, 5, and 10 cycles. These samples were then characterized using Scanning Transmission Electron Microscopy (STEM), and were found to have incomplete and non-conformal growth. Additionally, the growth rate was studied for these two oxides under pressure giving mixed results.

Bioceramic-Based Biomaterial Products for 3D-Printed Orthopedic Screws

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The body can safely absorb nontoxic, biodegradable materials over a period of time. Currently, orthopedic implants are often composed of titanium due to its high tensile and compressive strength, but this often requires a second surgery for removal. To avoid an additional procedure, biodegradable orthopedic screws allow fractured bones to heal completely while slowly dissolving in the body. The focus of this project is to determine the mechanical characteristics of a biocompatible, biodegradable composite material that will be utilized as orthopedic bone fixtures. Initially, polylactic acid (PLA) and hydroxyapatite (HA) were combined to create five samples of 10-50 vol % HA composite mixtures. Compression and tensile bar molds were then printed from acrylonitrile butadiene styrene (ABS) to create compression-molded test specimens of the composite mixtures. These specimens will then be utilized for testing and analyzing load-bearing properties. Finally, the composite material will be used to create orthopedic screws from a 3D printed compression mold. Current work is being done to determine the mechanical and structural properties of the materials.

Decreasing the Defects in Free-Standing Nickel Inverse Opal Cellular Solids

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High strength, low weight materials are important for improving the fuel efficiency of vehicles, performance of robots, and even bone replacements. A promising method for achieving high strength and low weight materials has been to develop foams with nanoscale pores fabricated from self-assembled spherical particles and electrodeposited metal; however, scaling the fabrication of these nanoscale foams causes cracking and defects which introduce weaknesses into the material. In this work, we study how adding 0.05 wt% to 5 wt% concentrations of polyethylene glycol into colloidal suspensions of PS affects the defect density of the final self-assembled PS opals. In addition, we develop techniques for removing the nanoscale foams from hard substrates so that the foams can be mechanically tested.

Probing the synthesis and conversion of perovskite single crystals

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Hybrid organic-inorganic perovskites (HOIPs) have recently attracted great attention for numerous applications including field-effect transistors, light-emitting diodes, lasers, and photovoltaics due to their direct band gap, high absorption coefficient, and relatively longcarrier diffusion lengths. Although most of the processing technologies to prepare HOIPs to date are based on solution processed, several different methods have been developed to synthesize HOIPs utilizing chemical vapor deposition (CVD). This latter method usually consists of depositing a lead halide precursor on a substrate, followed by a gas-solidhetero-phase reaction with methylammonium halide. However, a systematic study of the importance of reaction parameters such as carrier gas flow, temperature, and duration in the formation chemistry of individual HOIP crystals has not yet been described. Here, we utilize well-defined, single-crystalline lead iodide microplatelets in a low-pressure vaporphase reaction with methylammonium iodide (MAI) to study which reaction conditions have the greatest impact on the final methylammonium lead iodide (MAPbI3) grain morphology. Electron microscopy observations of vapor-converted MAPbI3 indicate that grain size can be tuned by varying the carrier gas flow and reaction time, with the larger
grain being formed under 100 sccm and 45 minutes at 120°C, respectively. AFM topographic images show that PbI2 crystallites undergo an isotropic absolute expansion and an increase of surface roughness during conversion. Atomic composition measurements by energy-dispersive X-ray spectroscopy (EDS) indicate that the extent of reaction can also be controlled by changing the reaction conditions. Emission spectrum of MAPbI3 obtained by photoluminescence measurements exhibit a strong emission peak centered at 770 nm. Our results suggest the promise of this CVD method in obtaining a clearer understanding of the formation chemistry of HOIPs.

Optimization of Electron Beam Lithography for the Fabrication of Nanostructured Optical Devices

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Polarizer gratings were fabricated using gold coated nanostructures in bulk silicon; the period, fill factor, and etch depth of the gratings must all be optimized for the selection of a desired polarization state and wavelength. To optimize these parameters the dosing of the electron-beam lithography process was varied along with PMGI/PMMA bilayer development times to find the most favorable process for the fabrication of gratings with periods varying from 1 µm to 200 nm. The ultimate application for these devices is to form an array of wavelength and polarization filters to be used in a polarimetric imaging system for atmospheric science applications.

Laser-activated Thermoplasmonic Substrates for Intracellular Drug Delivery

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CRISPR CAS-9 can be instrumental in treating a range of diseases including cancer, genetic cardiac disorders and viral diseases such as HIV. Our goal is to find a cost-effective and repeatable method to deliver large molecules such as the gene-editing tool CRISPR CAS-9 into the cells on a large scale while maintaining cell viability. We have so far been successful in delivering CRISPR-sized florescent molecules(150kDa) into cells with a viability of up to 70% and delivery efficiency of up to 98% of the viable cells. In addition to being repeatable with an average of 70 percent viability, the use of templates to produce multiple substrates improves the cost effectiveness of our method. Our method has relatively high viability compared to previous approaches to this problem because we use gold pyramids for their conductivity and geometric significance, a laser heats up the substrate and due to the geometric properties of the pyramids the temperature at the tips of the pyramids increases, creating bubbles in the surrounding cell medium that burst and perforates the cell membrane, allowing the target molecules to enter the cell. After this process, the cell membrane quickly heals itself in viable cells. The goal this summer was to find the optimum dimensions and spacings of the pyramids on the substrate to further improve cell viability. With the current dimensions of the pyramids there are on average 80 pyramids to one cell in the substrate. The experiment the we have designed will alter the spacings between the pyramids and to achieve a cell to pyramid ratio ranging from 10 pyramids per cell to 70 pyramids per cell in increments of 10. With this experiment, we hope to find the optimum number of pyramids per cell to achieve a higher viability.

Gas Microfluidics using MEMS Micro-pumps

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Gas microfluidics is the study of how gases behave in a submillimeter environment. This has led to the creation of different types of pumps for further gas microfluidic study and applications such as the accommodation, Knudsen, and thermomolecular pumps. Thermal transpiration, describes the mass flow of gas in the free molecular flow regime with a temperature gradient, present. However, more research is required to understand how the thermal transpiration phenomenon affects the flow and mass transport of gas in microfluidic channels. This study investigates the effects of thermal transpiration on the mass transport of air through straight-channel nanoporous membranes in the free molecular and transitional flow regimes. The MEMS micro-pumps are currently being studied for their potential use for Lab-On-A-Chip applications.

Constructing Complex Three-Dimensional Microstructures for Enhanced Adhesion

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In this project, an emerging microfabrication technique known as of two-photon lithography is used to construct artificial, bioinspired fibrillar adhesives with complex 3D geometries. The fibrillar adhesive structures studied have geometries that are inspired by the attachment pads on organisms such as geckos, various insects, and arachnids. Two photon lithography enables the creation of structured surfaces with unique three-dimensional geometry, surpassing traditional methods of photolithography, which offer rather limited opportunities for constructing adhesive structures with complex 3D geometries. As adhesion is strongly linked to the microscale three-dimensional geometry of fibrils, two-photon lithography is a promising method to realize fibrillar microstructures with optimized geometries. Structures were designed, fabricated via two-photon lithography and molding, and characterized during the REU project.

Environmental Effects on SU-8 Flexures in MEMS Devices

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SU-8 is a photoset epoxy frequently used in Micro-Electrical-Mechanical Systems (MEMS) devices. In order to ensure the reliability of devices using SU-8, the impact of temperature and humidity must be investigated due to the diverse environment in which the devices might operate. This project investigates how the mechanical properties of SU-8 are influenced by changes in temperature and humidity, by testing previously fabricated 3-dimensional scanning mirrors. These mirrors have two sets of SU-8 torsional flexures that allow the structure to rotate about two axes, while a deformable SU-8 membrane allows the mirror to adjust beam focus along the third axis. The mirrors have a resonant frequency for torsional oscillation where one gets the greatest range of motion. Devices were exposed to independent variation of both humidity and temperature. After an equilibrium time, the device was scanned to find the resonant frequency, first about one axis and then the other, to investigate the two different sets of flexures independently. We report the observed effect of temperature and relative humidity on these SU-8 flexures, measured as a change in resonant frequency.

Piezoelectric RF SAW-based Energy Detectors

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Extremely low-power electronic devices promise improvements in remote and distributed sensor networks, among other applications. Thus, we have designed and studied mechanisms based on piezoelectric surface acoustic wave (SAW) and graphene properties that detect and manipulate high frequency (200 MHz) electronic signals characteristic of radio frequency (RF) communications intercepted by such a device in hopes of using these signals as a power source. We demonstrate conversion of RF signals to SAWs on a lithium niobate substrate with standard interdigitated transducers. Electrodes of monolayer graphene placed on top of the substrate intercept the SAWs and converts them back to electric current via the acoustoelectric effect.

Use of Sacrificial Polymers in the Manufacturing of Low Dielectric Materials

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Low Dielectric materials are desired in the microelectronics industry to prevent energy loss due to parasitic capacitance of the circuit board. Epoxy resins have been investigated, and while they have the desired low dielectric constant, the need arose for a material with an even lower constant. A sacrificial polymer (in this case polypropylene carbonate or PPC) was used as a porogen to create a nanoporous structure that will preserve the mechanical properties of the epoxy film while lowering its dielectric constant, due to the increased presence of air in the now porous epoxy film. A serial study was preformed to determine the optimal ratio of sacrificial porogen to parent polymer (in this case a styrene maleic anhydride copolymer or SMA) to achieve the lowest dielectric constant without compromising the glass transition temperature or mechanical properties of the epoxy resin to a significant degree. Preliminary results show a 0.08 increase in the dielectric constant opposed to the expected decrease when comparing the nonporous epoxy and 30% PPC epoxy resin, believed to be caused by left over PPC in the film, and the expected decrease in glass transition temperature with increasing amounts of sacrificial polymer.

Stretchable Electronics: Processing and Analyzing Blended P3HT-PDMS Devices

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The creation of flexible and stretchable electronics devices has become possible using semiconducting polymers in junction with polymer substrates and electrodes. In this research, the semiconducting polymer, P3HT (Poly(3-hexylthiophene-2,5-diyl), was mixed with an elastic polymer, PDMS (Polydimethylsiloxane). This blend was then processed and exposed to ultraviolet light for either one, three, five, or eight minutes. The electronic properties, morphology, and absorbance of the polymer blend system were all analyzed. The highest mobility achieved was 0.247 $cm^2 V^{-1} s^{-1}$ after three minutes of UV exposure. In agreement with the electronic mobility, the UV-vis spectrum and AFM morphology of the polymer system showed higher absorbance and larger aggregated polymer fibers, respectively, for three minutes of UV light exposure. The advantage of a blended elastomer and semiconductor is that during UV exposure the P3HT will be able to aggregate within the PDMS and form nanofibers more effectively. P3HT

does not dissolve well in PDMS, so once the solvent, chloroform, evaporates, the P3HT will aggregate and migrate toward the transistor interface allowing for better charge transport and motilities.

Characterization of SU-8 Spin Coating Over Wafer Topography

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SU-8 is an epoxy-based negative photoresist used commonly in microfabrication as a masking material and for permanent micro and nanostructures. SU-8 is particularly important in the fabrication of MEMS deformable membrane mirrors where it can serve as the deformable polymer membrane as well as numerous other structural elements. The fabrication of MEMS mirrors commonly requires SU-8 deposition through spin coating over substrates with high aspect ratio topography. High aspect ratio topography considerably complicates spin coating in comparison to planar and low aspect ratio substrates. A parametric study of spin coating SU-8 over high aspect ratio trenches and vias is presented. The coating of trench and via features is investigated based on the experimental parameters of feature width, depth, radial location on the wafer, angular orientation, resist viscosity, and spin velocity. A profilometer and scanning electron microscope are used to measure and examine coated features. The effects of parameters on feature bridging, non-planarity index, and degree of fill index are presented.

Influence of polymer and nonwoven properties for depositing alumina thin films on poly(vinyl alcohol) nanofibers using flow-through Spatial Atomic Layer Deposition

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Previously the important operating parameters of a novel, flow-through spatial atomic layer deposition (ALD) reactor for deposition on porous nonreactive substrates was evaluated using meltblown polypropylene fibers. In this current study, electrospun poly(vinyl alcohol) (PVA) nanofibers were coated by ALD using the same spatial ALD reactor to evaluate the deposition conditions required for good coatings on a reactive fiber substrate. PVA was selected because it demonstrates a decrease in water solubility with increasing cycles of alumina ALD, allowing for simple evaluation of ALD coating quality.

Solutions of 7 wt% PVA dissolved in water were prepared. Electrospinning was performed at a voltage of 12 kV and pump flow rate of 0.5 mL h-1 onto meltblown polypropylene nonwoven, which served as a support layer to give mechanical stability to the nanofibers during treatment with the spatial ALD tool. Surrounding air was maintained at 22 °C and 30% humidity. Spatial ALD was performed at atmospheric pressure using trimethylaluminum and water for 3, 33, 99, and 198 cycles. The operating temperature was around 70 °C. To evaluate conformality and quality of the coatings, fiber morphology of the PVA mats was evaluated after submersion in water for 1 hr, and 1 day using SEM. X-ray photoelectron spectroscopy analysis was performed to confirm the increased presence of alumina with higher cycle numbers.