

NanoMeter

Cornell
NanoScale

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Facility
(CNF)

Spring
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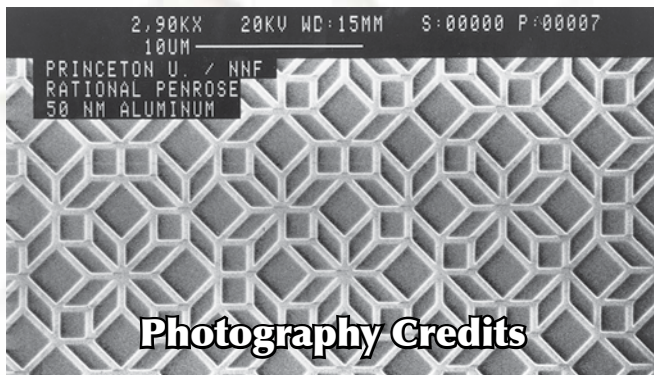
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Our M&M cover was the inspiration of our director, Christopher Ober, made real by Melanie-Claire Mallison's shopping skills and Don Tennant's photography skills. The CNF staff enjoyed eating up the evidence! Yes—those are real M&Ms! (Or... N&Ms..) The background for this issue harkens back to last year's 40th anniversary with a device image from the NNF years, above. The photographs in this book were primarily provided by the author(s), but also by University Photography or CNF Staff.

The NanoMeter is formatted by Melanie-Claire Mallison, and is printed on 30% post-consumer content paper using soy-based inks. We welcome your comments.

Please reduce, reuse, and recycle!



Reminder to Submit to CNF User Wiki

Dear CNF Community: Please remember to share your process and recipe updates on the CNF User Wiki.

wiki.cnfusers.cornell.edu

NNCI Introductory Video

The National Nanotechnology Infrastructure Network (NNIN) has a new video on YouTube that provides an overview of the network and its resources.

<https://youtu.be/72ZXh-EST3U>



Welcome to the 2018 Spring Edition of the CNF NanoMeter

We are very happy to introduce the latest edition of the *NanoMeter*. This year (2018) is set to be an eventful one. We are in the beginning stages of implementing our strategic plan for the next few years and this includes a new focus on heterointegration, a reinvigorated effort in nanobiotechnology, and an enhanced push into two-dimensional (2D) materials. As part of this activity, we are acquiring new tools (more below) including a new AlN deposition system and we are hoping to acquire a flip chip bonder and 3D printer with 2-photon lithography, nanoscale capabilities. Our interest in nanobiotechnology is an ongoing one, and the merger of most of Nanobiotechnology Center (NBTC) into CNF gives us an opportunity to offer additional capabilities, many of which are located outside the cleanroom. Finally our partnership with PARADIM, the new NSF materials innovation platform (MIP) that offers theory, synthesis and characterization capabilities for 2D materials, provides unique nanofabrication expertise based on our long experience with 2D materials.

Before I (CKO) go on, I must mention that this year Don Tennant will be retiring. Don is one of the pioneers of nanofabrication, known for his work on e-beam lithography and his early work on EUV patterning. Anyone who works in this area is well aware of the positive impact that Don has had on this community and we have been fortunate to have him here at CNF. Don deserves our many thanks for helping to lead CNF through its successful membership in the NNCI network following many years as part of NNIN. After a dozen years with Don at the helm of CNF, we are looking for a new Director of Operations. Don will stay connected to CNF through various consulting activities and expects to remain in the Ithaca area.

We want to now return to the merger of the NBTC with CNF. Last spring it became clear that operating the NBTC as a stand alone facility was becoming increasingly difficult. CNF has worked with the NBTC since its beginning and it made sense for CNF to add many of the fabrication and characterization capabilities to its skill set. Beth Rhoades, CNF's Senior Biology Associate and a trained PhD microbiologist, coordinated much of the merger and brought these new capabilities to the second floor of Duffield Hall. Many of these new tools are located outside the cleanroom and training on them takes place in a new course. To continue to make these facilities accessible without barriers, we have developed a "second floor only" safety training. This allows users without need of the cleanroom to rapidly start or resume work on these instruments.

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We have also been on the road to visit other schools in New York State to sing the praises of our capabilities. In the past year we have made stops at Clarkson, RPI, SUNY Poly, SUNY Binghamton, and Syracuse, and we are happy to visit other schools. We see CNF as a regional center welcoming researchers from across the state, country and world. If you would like an information visit or a seminar, please feel free to contact us (information@cnf.cornell.edu).

In the lab we have ordered a new Filmetrics F40-UV and a new F40 thickness measurement microscope suite. These will enhance our thin film measurement capabilities for ultra-thin films such as those deposited in our atomic layer deposition (ALD) tools. We have also seen activity in developing new plasma etch processes for an expanded set of materials. See articles in this issue related to deep etching of germanium, and a reprovisioning of our metal etchers to handle aluminum, aluminum-silicon-copper, aluminum oxide, chrome, titanium, tantalum, niobium, molybdenum, molybdenum di-silicide, and titanium nitride. This is part of an overall scheme to allow a wider range of materials to be processed in the lab. Our plasma process team and CNF Fellows have done a great job of porting and vetting user processes to different etchers to allow us to redeploy our tools to meet this expanding need.

Finally, we were made aware of a new book, "The Long Arm of Moore's Law," written by Cyrus Mody, that spends a couple of chapters devoted to the origins of the micro/nanofabrication centers including the pivotal role and early struggles of CNF (then NRRFSS). It discusses the many and early contributions of the people who shaped the thinking of the funding agencies and the evolution of interdisciplinary research and even the conferences that helped promote this mode of research. We (CKO & DMT) found it riveting.

In late breaking news — Prof. Mody has agreed to be our 2018 CNF Annual Meeting plenary speaker! Join us on Wednesday, October 3rd, to learn more about and from Prof. Mody. And see page 14 to learn more about this year's annual meeting.

***As always, we love to hear from you
and welcome your comments.***



Chris and Don



Understanding Nanocatalysts' 'Talk' Could Better Inform Design

By Tom Fleischman
March 26, 2018
Cornell Chronicle

Enzymes are nature's best nanoscale catalysts, and often show what's known as catalytic allostery—that is, reactions at one site affecting reactions at another site, typically a few nanometers away, without direct interaction between the reactants. Nanoparticles of various materials, such as metal, can also act as catalysts for chemical transformations on their own surfaces, and their surface-active sites can be electronically coupled. Peng Chen, the Peter J.W. Debye Professor of Chemistry in the Department of Chemistry and Chemical Biology, is taking his background in enzyme studies and applying it to his other research specialty: single-molecule catalysis.

His group has published a paper in *Nature Chemistry*, the culmination of years of study, that lays out a conceptual framework for understanding how a nanocatalyst particle works. The work could help inform better design of synthetic nanocatalysts down the road.

"Co-operative Communication Within and Between Single Nanocatalysts" was published March 26. Lead authors are former graduate students Ningmu Zou and Guanqun Chen, and former postdoctoral researcher Xiaochun Zhou. Given his enzymes background, Chen wondered: Can reactions at different surface sites on the same nanocatalyst communicate with each other, similar to allosteric enzymes?

"We'd already developed a way to map catalytic reactions on a single catalyst, in a spatiotemporally [space and time] resolved way," said Chen, whose group published a paper on the subject last year. "For every reaction happening on a catalyst particle, we know where it happened and when it happened. Then I came up with the question regarding whether reactions at different places, on the same catalyst, can talk to each other."

Using single-molecule fluorescence microscopy, Chen and the group found that catalytic reactions on a single nanoscale catalyst—in this case, nanoparticles of gold and palladium—can indeed communicate with each other, likely via movement of positive charge carriers known as holes. The group tested this on two types of nanocatalyst morphologies, and three distinct types of catalytic transformations. They also found that reactions on separate gold nanocatalysts communicate with each other, over even greater distances, through diffusion of negatively charged reaction products.

This communication is analogous to the "spillover" effect in surface science, Chen said.

Both of these represent first-of-their-kind observations involving individual nanocatalysts. "This provides a new sort of conceptual framework for understanding how a nanoscale catalyst particle works," Chen said.

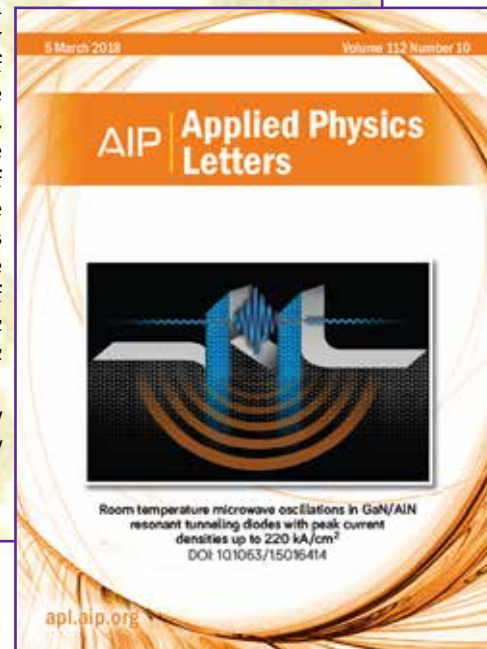
Although applying these observations to real-world development of nonbiological nanocatalysts is still well into the future, this discovery pushes basic science toward that goal, Chen said. "If one can capitalize on that important feature of enzymes in a nonbiological catalyst, maybe there's a way to improve the catalyst's function," he said.

Doctoral student Won Jung of the Chen group also contributed to this work, which was supported by grants from the Army Research Office; the Department of Energy (Office of Science, Basic Energy Sciences, Catalysis Science Program); and the National Science Foundation. Work was done in part at the Cornell Center for Materials Research and the Cornell NanoScale Science and Technology Facility, both of which are supported by the NSF.

Community News: Xing & Jena Cover

The March issue of *Applied Physics Letters* featured in its cover the recent work published by Professors Grace Xing and Debdeep Jena. The cover image shows the arrangement of atomic layers designed to confine electrons within a quantum well made of gallium nitride. This artificial atomic structure, whose number atoms can be counted in the image, was synthesized in Duffield Hall and fabricated in CNF. By injecting a stream of electrons across the tunneling barriers, current oscillations are generated as a result of quantum interference effects. These results pave the way for the demonstration of ultra-fast electronic and photonic oscillators.

URL Link: <https://aip.scitation.org/doi/abs/10.1063/1.5016414>



Novel Semiconductor-Superconductor Structure Features Versatile Gallium Nitride

By Tom Fleischman
March 7, 2018
Cornell Chronicle

Silicon has been the semiconductor material of choice for electronics pretty much since the transistor effect was first observed and identified nearly 80 years ago. There's a valley in California named for it, after all.

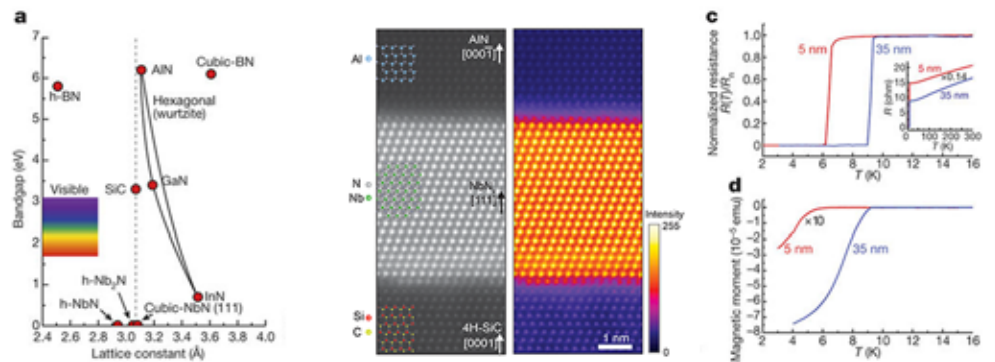
But a relatively new family of semiconductors—group III-nitrides, including gallium nitride (GaN), indium nitride and aluminum nitride—offers greater versatility than silicon with capabilities for ultrafast wireless communications, high-voltage switches and high intensity lighting and photonics.

A team led by Debdeep Jena, professor of electrical and computer engineering (ECE), and David Meyer, head of the Wide Bandgap Materials and Devices section at the Naval Research Laboratory, has successfully devised a semiconductor-superconductor crystal structure featuring GaN grown directly onto a crystal of niobium nitride (NbN), a proven superconductor material used in quantum communications, astronomy and a host of other applications. The group's paper, "GaN/NbN Epitaxial Semi-conductor/Superconductor Heterostructures," was published online March 8 in *Nature*. Former postdoctoral researcher Rusen Yan and current postdoc Guru Khalsa are co-lead authors.

Other key contributors were Grace Xing, the Richard Lundquist Sesquicentennial Professor in ECE and MSE, and David Muller, the Samuel B. Eckert Professor of Engineering in the Department of Applied and Engineering Physics. The method for combining the two materials—molecular beam epitaxy (MBE), essentially spray painting of gallium and nitrogen atoms onto the NbN in a vacuum environment—creates an extremely clean interface and is key to the success of the novel structure. This advance, the group says, opens up a range of possibilities that can

Figure 1: Bandgap, lattice constant, crystallinity and superconductivity in epitaxial NbN_x on SiC.

From: GaN/NbN epitaxial semiconductor/superconductor heterostructures



a, Bandgap versus lattice constant for select nitride semiconductors as well as for SiC. b, Cross-section HAADF-STEM images in black/white (left) and false-colour (right) of 5-nm NbN_x grown on an SiC substrate with a AlN capping layer. c, Resistance versus temperature (normalized to the resistance at 16 K), showing the superconducting phase transition of 5-nm (red) and 35-nm (blue) epitaxial NbN_x on SiC. Inset, resistance measured up to 300 K. d, The Meissner effect measured on the 5-nm and 35-nm samples, showing clear magnetic-flux expulsion accompanying the superconducting phase transition. These measurements are consistent with the T_c obtained in panel c. $\times 10$ and $\times 0.14$ indicate multiplication of the data by 10 or 0.14, respectively, to allow data of different scales to be shown on the same plot.

now combine the macroscopic quantum effects of superconductors with the rich electronic and photonic properties of group III-nitride semiconductors.

"People have tried it with other semiconductors, like silicon and gallium arsenide, but I don't think anything has been as successful as what we've managed to do with GaN," said Jena, who has a dual appointment with the Department of Materials Science and Engineering (MSE).

Gallium nitride-based semiconductors have recently made major inroads in the areas of LED lighting, Blu-ray laser diodes, energy and communications. In fact, the 2014 Nobel Prize in physics was given to a trio of Japanese scientists for their invention of energy-efficient blue light-emitting diodes (LEDs) using GaN.

Technological advances—particularly the type of MBE used in this work, which was developed at the Naval Research Laboratory—has made it possible for scientists to think about semiconductor-superconductor heterostructures such as the one Jena's group has developed. The specialized nitride MBE system includes an electron beam evaporator source, which "melts" the niobium—which has a melting

point of around 4,500 degrees—but not the crucible it's in. Atoms of niobium are deposited onto a silicon carbide wafer, and the GaN semiconductor layers are then grown on top of that, also by MBE.

"This new source allowed us to overcome the temperature limitations of conventional sources, and bring high-melting-point, refractory transition metals like niobium and tantalum into the picture," Meyer said. The team demonstrated for the first time the growth and fabrication of a semiconductor transistor switch, the prototypical gain element in electronics, directly on top of a crystalline superconductor layer. This heterostructure is a kind of "best of both worlds," Jena said, offering a method for devising quantum computation and highly secure communications systems.

"There are some things that we would love to do with quantum systems—quantum computation and

cryptography—things that are not possible in classical systems," he said. "On the other hand, there are things that classical systems are much better at than quantum systems. And there is this mesozone where you can do wonderful things by mixing and matching the two."

"We think this presents a wonderful opportunity for rapid technology development of next-generation communications and computation systems," Meyer said. Doctoral students John Wright and Suresh Vishwanath from the Jena-Xing Group and Yimo Han from the Muller Group also contributed.

Measurements taken for this work were performed in the Cornell Center for Materials Research Shared Facilities, supported by the National Science Foundation. Some of the heterostructure fabrication was done at the Cornell NanoScale Facility, also supported by the NSF. Additional support came from the Office of Naval Research.



Physicists Take First Step Toward Cell-Sized Robots

By Tom Fleischman, January 2, 2018
Cornell Chronicle

An electricity-conducting, environment-sensing, shape-changing machine the size of a human cell? Is that even possible?

Cornell physicists Paul McEuen and Itai Cohen not only say yes, but they've actually built the "muscle" for one.



Left to right; Itai Cohen, Marc Miskin, and Paul McEuen. Screen capture from video by Charles Walcott.

With postdoctoral researcher Marc Miskin at the helm, the team has made a robot exoskeleton that can rapidly change its shape upon sensing chemical or thermal changes in its environment. And, they claim, these microscale machines—equipped with electronic, photonic and chemical

payloads—could become a powerful platform for robotics at the size scale of biological microorganisms.

"You could put the computational power of the spaceship Voyager onto an object the size of a cell," Cohen said. "Then, where do you go explore?"

"We are trying to build what you might call an 'exoskeleton' for electronics," said McEuen, the John A. Newman Professor of Physical Science and director of the Kavli Institute at Cornell for Nanoscale Science. "Right now, you can make little computer chips that do a lot of information-processing ... but they don't know how to move or cause something to bend."

Their work is outlined in "Graphene-based Bimorphs for Micron-sized, Autonomous Origami Machines," published January 2 in Proceedings of the National Academy of Sciences. Miskin is lead author; other

2018 Dragon Day at Cornell University!



The College of Engineering Phoenix successfully protected the Engineering Quadrangle from the College of Architecture Dragon!

contributors included David Muller, the Samuel B. Eckert Professor of Engineering, and doctoral students Kyle Dorsey, Baris Bircan and Yimo Han.

The machines move using a motor called a bimorph. A bimorph is an assembly of two materials—in this case, graphene and glass—that bends when driven by a stimulus like heat, a chemical reaction or an applied voltage. The shape change happens because, in the case of heat, two materials with different thermal responses expand by different amounts over the same temperature change.

As a consequence, the bimorph bends to relieve some of this strain, allowing one layer to stretch out longer than the other. By adding rigid flat panels that cannot be bent by bimorphs, the researchers localize bending to take place only in specific places, creating folds. With this concept, they are able to make a variety of folding structures ranging from tetrahedra (triangular pyramids) to cubes.

In the case of graphene and glass, the bimorphs also fold in response to chemical stimuli by driving large ions into the glass, causing it to expand. Typically this chemical activity only occurs on the very outer edge of glass when submerged in water or some other ionic fluid. Since their bimorph is only a few nanometers thick, the glass is basically all outer edge and very reactive.

“It’s a neat trick,” Miskin said, “because it’s something you can do only with these nanoscale systems.”

The bimorph is built using atomic layer deposition—chemically “painting” atomically thin layers of silicon dioxide onto aluminum over a cover slip—then wet-transferring a single atomic layer of graphene on top of the stack. The result is the thinnest bimorph ever made.

One of their machines was described as being “three times larger than a red blood cell and three times smaller than a large neuron” when folded. Folding scaffolds of this size have been built before, but this group’s version has one clear advantage.

“Our devices are compatible with semiconductor manufacturing,” Cohen said. “That’s what’s making this compatible with our future vision for robotics at this scale.”

And due to graphene’s relative strength, Miskin said, it can handle the types of loads necessary for electronics applications.

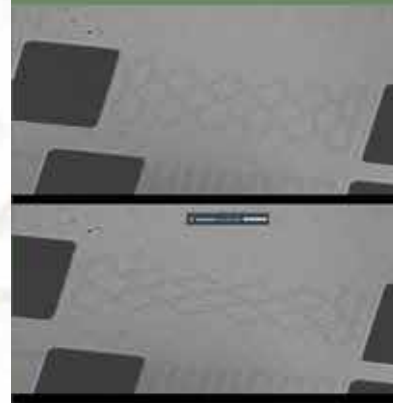
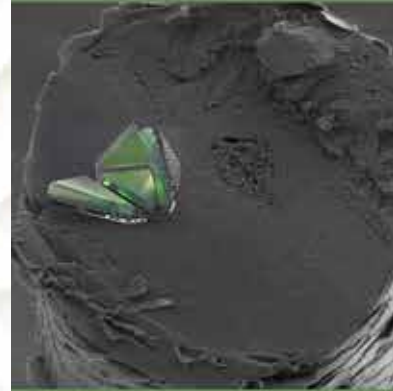
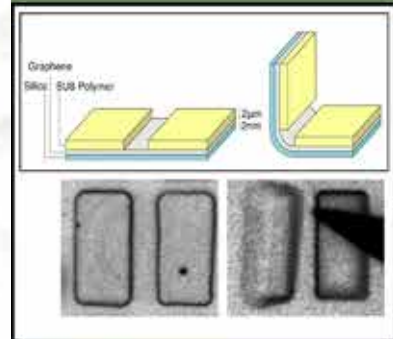
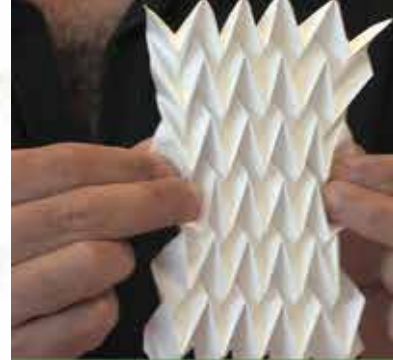
“If you want to build this electronics exoskeleton,” he said, “you need it to be able to produce enough force to carry the electronics. Ours does that.”

For now, these tiniest of tiny machines have no commercial application in electronics, biological sensing or anything else. But the research pushes the science of nanoscale robots forward, McEuen said.

“Right now, there are no ‘muscles’ for small-scale machines,” he said, “so we’re building the small-scale muscles.”

This work was performed at the Cornell NanoScale Facility for Science and Technology and supported by the Cornell Center for Materials Research, the National Science Foundation, the Air Force Office of Scientific Research and the Kavli Institute at Cornell.

Figures at right: Screen captures from video by Charles Walcott. Find the video at <http://news.cornell.edu/stories/2018/01/physicists-take-first-step-toward-cell-sized-robots>





What goes into making smaller, faster, and more efficient smart phones and other electronic and research devices? Chris Ober's lab has some answers.

by Jackie Swift

If you've ever seen the inside of a smart phone or a piece of wearable tech like an Apple Watch, you may have been amazed at the tiny size of the semiconductor chips that power the device. They aren't small enough, though, for the electronics industry. The industry seeks to produce components at ever decreasing size in an effort to increase speed and efficiency, and at the same time, lower power consumption and weight.

Working with materials at the microscopic level, Christopher K. Ober, Materials Science and Engineering, and his lab may have the answer. They produce an array of thin films and surfaces that have the potential to revolutionize the world of electronics. In addition, the Ober lab creates innovative surface materials for other industries, such as the biomedical and even the shipbuilding industries.

Creating the Tiniest Chip, Smallest Features

Recently, Ober joined with department colleague Emmanuel P. Giannelis to invent the world's most sensitive (fastest) extreme ultraviolet (EUV) photoresist. A photoresist is a coating used in photolithography to map out a pattern on a surface—say, of a silicon chip. The chip is coated with the photoresist and exposed to a light shone through a mask. The photoresist's solubility changes where the light strikes. A solvent then removes the more soluble sections, before the silicon chip is subjected to a subsequent process, such as etching or dopant diffusion, which transforms the silicon wherever the photoresist is absent, creating patterns on the chip.

Ober and Giannelis's breakthrough photoresist is made of metal oxide nanoparticle complexes only two nanometers across. (By comparison, the width of a human hair is around 80,000 nanometers.) The coating

is designed to change solubility when exposed to light at the EUV wavelength of 13 nanometers. "A crude rule of thumb for photolithography is that the wavelength you use for imaging is roughly the dimension of the patterned structures you can make," Ober explains. "With EUV imaging, that means you can make structures on the order of 13 nanometers, so down to 10 nanometers. That is awesomely tiny."

For the past 20 years, the electronics industry used various tricks to fabricate ever smaller structures using ultraviolet light with a wavelength of 193 nanometers, but they are finally stumped.

"They chose the EUV wavelength as their next target goal in part because the smaller wavelength makes smaller features," Ober says, "but the revolution in wavelength requires a revolution in materials. Most photoresists today are polymers (plastics) that are almost invisible at EUV wavelengths. They can't absorb the radiation, so the needed solubility change we want can't be induced in the coating. To capture the EUV radiation, we needed to make our photoresist from heavier elements in the periodic table. It took my polymer background and Emmanuel's inorganic materials background to actually make it work. Separately, I don't think we could have done it."

"Right now, we're working with a company to improve these materials. The next step is to get the photoresist to consistently show resolution below 15 nanometers at a quickness that is acceptable for potential manufacturing."

The new photoresist has shown good test results, and the semiconductor industry is very interested. "Right now, we're working with a company to improve these materials," Ober says. "The next step is to get the photoresist to consistently show resolution below 15 nanometers at a quickness that is acceptable for potential manufacturing."

The Cornell NanoScale Facility— Essential to Inventing New Materials and to Biological Studies

Ober, who is the director of the Cornell NanoScale Facility (CNF), points out the crucial role CNF played in the development of the new photoresist. “Without CNF, we could not have done this,” he says. “We have the best facility in the country for nanoscale research. All the basic semiconductor processing tools we need are here.”

He mentions in particular the facility’s JEOL (Japan Electron Optical Laboratory) 9500 scanning electron beam lithography tool, the only one of its kind at an American university. “We’re lucky to have a tool like that at Cornell,” he says. “This is one of the few places in the U.S. you can test at a size scale that small.”

Working at the nanoscale, the Ober Lab also uses other techniques to modify surfaces, including polymer brushes—polymer chains grown from a surface. “We start the growth of the polymer chain by attaching it chemically to a surface,” Ober explains.

“Depending on what we attach to this polymer backbone, we can make it hydrophilic (attracting water) or hydrophobic (repelling water), or we can attach chemical groups that will bind proteins, antibodies, or other biological components to that surface. Over the thickness of just a few nanometers, we can transform glass or silicon so that a living cell thinks it’s in contact with another cell, for instance.”

Using the tools at CNF, the researchers take the brushes and pattern them into shapes that can help them control how biological components interact with the surface, allowing them to study cell surface interactions. For instance, in one project they used pattern brushes to direct the growth of nerve cells on the surface of the brushes.

“We were working with researchers from the George Malliaras group [University of Cambridge], who were designing brain electrodes,” Ober says, “We wanted to provide a surface the nerves were comfortable with, which then led the nerves to the electrode location. We gave the nerve cells a physical cue that directed them to go to the place we wanted them to grow.”

The Tough Problem of Boat Hull Fouling

For years Ober has also been applying his work with polymer brushes to a particularly thorny problem: the fouling of boat hulls by ocean life forms, such as barnacles, seaweed, and bacteria. Fouling is a natural process with a huge cost both environmentally and monetarily.

“All that stuff growing on the hull makes the surface very rough, and ships have to burn extra fuel to push their way through the water,” Ober explains. “For an ocean-going ship, that can mean several million dollars extra in fuel costs a year. And that fuel creates carbon dioxide, which pollutes the atmosphere.”

The traditional solution has been to poison the sea creatures by loading toxic copper metal into the paint used on ships’ hulls. The paint is designed to wear away, constantly exposing fresh copper, and the copper ends up accumulating in the ocean. Ober is part of a multidisciplinary international team funded by the United States Navy seeking to solve the problem through surface engineering. “It’s incredibly hard because, given all the years of evolution, there’s always something that will stick to your surface,” Ober says.

So far, the researchers have come up with materials that resist fouling in the lab but fall short when tested in the ocean, especially against sea creatures known as hard foulers—barnacles, zebra mussels, and the like. Hard foulers attach themselves to surfaces with a liquid adhesive that cures to a material similar to an industrial strength epoxy.

“We’ve figured out the optimal surface tension a material needs to prevent most life forms from sticking,” Ober says. “Now if we can figure out how to interfere with the process hard foulers use to cure their liquid adhesive into a solid, we can engineer the material we need. Our goal is to coat the hull with the material. Then as the ship pushes through the water, the critters will just fall off.”



Community News: Victor Aguilar

I graduated from Cornell this past December with a Master of Science in Biomedical Engineering. I have been accepted to the University of Illinois at Chicago to pursue a Doctor of Philosophy in Bioengineering with Prof. David Eddington. I have also been selected to receive the selective Graduate College University Fellowship (provides two years of full funding + a two-year RA appointment). As side news to this, I have submitted and published a master’s thesis incorporating part of the work I did while at CNF.

First Proof of Hydrogen-Bonded Water Wires Could Lead to Better Desalination

By Tom Fleischman
March 26, 2018
Cornell Chronicle

Approximately 70 percent of the Earth's surface is covered by water, but only 2.5 percent of that is fresh water, and less than half of that is easily accessible. Efficient desalination, therefore, is crucial for sustaining life now and in years to come. But a challenge in developing bioinspired synthetic purification systems lies in deciphering the complexity of natural systems like aquaporins (AQPs), nature's most efficient water transport system. These proteins form pores in the membranes of biological cells, facilitating highly efficient water transport.

An international research partnership—co-led by Poul Petersen, assistant professor in the Department of Chemistry and Chemical Biology—has made the first direct observation of the structure of water inside artificial water channels. Understanding the “shape of water,” so to speak, under those conditions will help inform biomimetic purification systems of the future.

Petersen is co-senior author of “Oriented Chiral Water Wires in Artificial Transmembrane Channels,” which published March 23 in *Science Advances*. The paper's other senior authors are Mihail Barboiu of the Université de Montpellier, France; and Georges Belfort of Rensselaer Polytechnic Institute.

Water wires—hydrogen-bonded chains of associated water molecules, which have properties that are very different from bulk water—have been confirmed in theory for years but never actually seen. Petersen and his collaborators, through nonlinear sum-frequency vibrational spectroscopy, claim the first experimental observation of the dipolar oriented (electrically neutral) wire structure of water in nanoscale channels, including supported lipid bilayers, similar to aquaporins. “I would call this the first real observation of a water wire,” Petersen said. “We're not just seeing the oxygen [atoms], we see the protons, as well. It's the first observation of the hydrogen bonding in a water wire.”

This work employs some of the methods Petersen's lab used in work published last year, in which it reported the first observation of a chiral water superstructure surrounding a biomolecule (DNA).

“We can do our chiral method, just like we did for the DNA research, and see that they also form a chiral structure,” Petersen said. “They have a helicity to them—they sort of spiral through.”

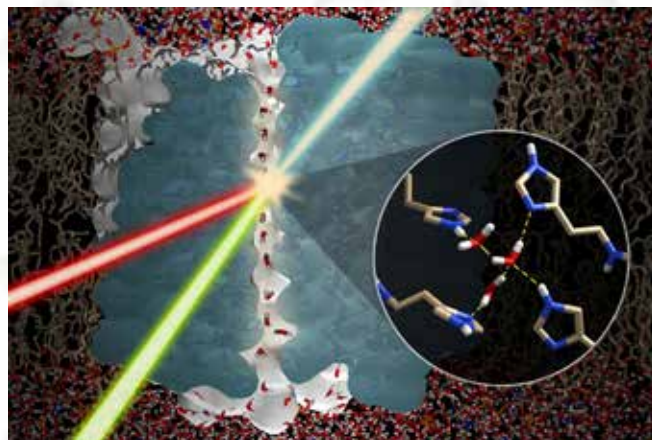


Figure 1: An illustration of the first direct observation of a dipolar oriented chiral water wire inside a transmembrane channel. Provided.

One of the key findings: The net dipolar orientation of water molecules in confined channels induced specific polarization of the channel, which likely acts as a driving force for water permeation through the membranes. The best current method for desalinating seawater is reverse osmosis—fluid passing through a membrane in the direction opposite of naturally occurring osmotic pressure. The group feels that identifying the chirality of water structures within these artificial channels will inspire design of more efficient purification technologies in the future.

“Scientists are trying to make structures that mimic aquaporin—that's the gold standard for water transport,” Petersen said. “And for aquaporin, we know it's all about the alignment of the water molecules, and their helical nature, that is important for water transport.”

Also contributing was Heather Vanselow, Ph.D. '17, and doctoral student Stephanie Sanders, both from the Petersen group. Sample preparation included work conducted at the Cornell Center for Materials Research, which is supported through the National Science Foundation's Materials Research Science and Engineering Centers program, as well as the Cornell NanoScale Science and Technology Facility, also supported by the NSF.

Petersen's contributions to this work were supported by a Young Investigator grant from the Arnold and Mabel Beckman Foundation, and through his NSF Career Award grant.

Controlling Ultra-High Speed Electron Tunneling Waves

Wednesday, November 22, 2017

Cornell Engineering,
School of Electrical and Computer Engineering

Resonant tunneling diodes are semiconductor devices which enable electrons to travel at ultra-high speeds. However, engineering this quantum transport regime within the III-Nitride family of semiconductors has remained challenging for almost twenty years. Now, advances in epitaxial growth and device fabrication at Cornell, have led to groundbreaking observations into the resonant tunneling physics of III-Nitride semiconductors. These results not only raise hopes for future practical applications, but also reveal fundamental properties of semiconductor quantum heterostructures.

Resonant tunneling diodes (RTDs), being the fastest active electronic devices to date, are candidates for developing compact solid-state sources that will be at the heart of future ultra-high speed communication networks. The versatility of these devices has already been demonstrated by achieving wireless data transmission rates 100 faster than current standards [1].

This milestone has been possible due to the intrinsic fast speed of these devices which are capable of generating high-frequency signals within the terahertz (THz) band. Ultra-high speed data transmission is not

the only important application enabled by RTDs. THz Imaging [2] and spectroscopy [3] systems have also been envisioned for security scanners and explosives detectors. Furthermore, non-destructive quality control systems for medical and pharmaceutical industries have also become an increasingly important application. However, despite the broad range of practical applications enabled by these devices, their limited output power remains the major technical limitation, preventing field deployment.

In this scenario, the III-Nitride family of semiconductors has emerged as a potential solution to the limited output power available from current RTD-sources. This revolutionary family of semiconductors are routinely synthesized by a group of researchers led by Cornell engineering professors Debdeep Jena and Grace Xing. III-Nitride semiconductors, characterized by its efficient light emission, have already garnered the Nobel prize in physics three years ago. However, its versatility could be further expanded with the demonstration of light-emitters within the infrared and THz bands. These applications, however, rely on engineering artificial electronic levels and devising electron transport mechanisms to populate them. Nonetheless, for almost twenty years, resonant tunneling, a quantum transport process capable of solving these limitations, has remained elusive in III-Nitride semiconductors.

Now, in a recent study, the Cornell research group, has shown that III-Nitride semiconductors are capable of robust resonant tunneling transport which persists even at room temperature. These results, described by a reviewer as a “highly significant achievement”, have been recently published in Physical Review X [4].

Advances in crystal growth quality combined with the use of state-of-the-art tools, has enabled the observation of unprecedented quantum transport signatures. These unique results not only raise hopes for future practical applications, but also reveal fundamental properties of semiconductor quantum heterostructures which have remained masked over decades.

Resonant tunneling relies on quantum mechanical effects which dictate that electron waves are allowed to tunnel across a double-barrier structure such as the one shown in Figure 1. This image shows the atomic layers that comprise the device under study, across which, electrons tunnel at ultra-high speeds.

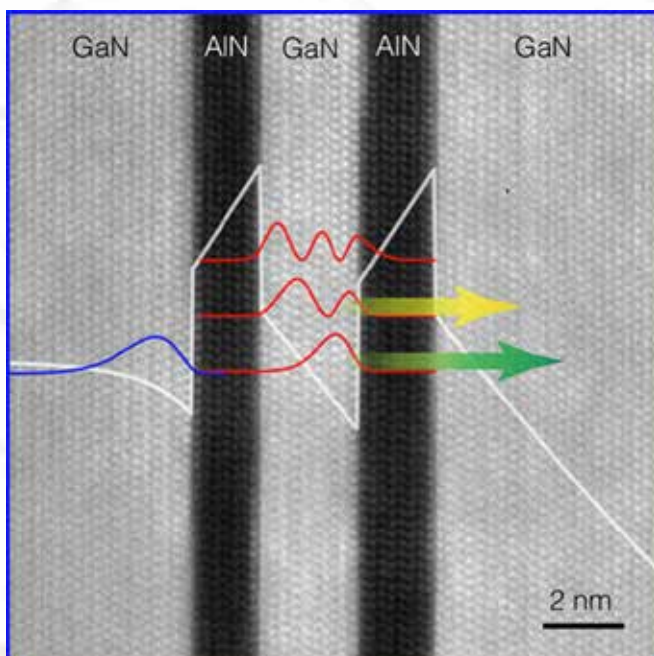


Figure 1: Scanning transmission electron microscopy (STEM) image of the thin layers within a resonant tunneling diode (RTD). An overlay of the electron waves shows the quantum states (in red) that enable resonant tunneling transport.

Quantum confinement introduced by the barriers generates artificial electronic levels in the central layer which enhance electron transport. Nonetheless, these artificial levels, depicted by the red wavefunctions, are extremely sensitive to thickness variations and structural defects. The high density of these imperfections have prevented previous research groups to engineer resonant tunneling in III-Nitrides.

Using molecular beam epitaxy (MBE), a technique which is the specialty of the Jena-Xing laboratory, precise control over the layer thickness and high quality interfaces were attained. This has led to robust quantum transport effects that allowed researchers to detect and measure several unexpected tunneling features. It turns out that these features are intimately connected with the crystal structure of these materials. By uncovering this connection, the team came up with a new metrology scheme which can be employed to probe internal electric fields with unprecedented highest precision. Such internal fields, known as polarization fields, play a crucial role in the performance of photonic and electronic devices. Thus, this novel technique, pioneered at Cornell, might enable the design of more efficient light emitting diodes (LEDs) and transistors.

Looking forward, these findings pave the way for realizing III-Nitride-based high-speed electronic oscillators and room temperature THz quantum cascade lasers which can reach wavelengths that have remained unachievable by other semiconductors. Finally, by introducing a novel metrology tool, researchers have further expanded the versatility of III-Nitride semiconductors, which could potentially span yet unexplored fields in the near future.

This work was performed in part at the Cornell NanoScale Facility, a member of the National Nanotechnology Coordinated Infrastructure (NNCI), which is supported by the National Science Foundation (Grant ECCS-1542081).

References

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- 2 T. Miyamoto, A. Yamaguchi, and T. Mukai, "Terahertz imaging system with resonant tunneling diodes," *Jpn. J. Appl. Phys* 55, 032201 (2016).
- 3 S. Kitagawa, M. Mizuno, S. Saito, K. Ogino, S. Suzuki, and M. Asada, "Frequency-tunable resonant-tunneling-diode terahertz oscillators applied to absorbance measurement," *Jpn. J. Appl. Phys* 56, 058002 (2017).
- 4 J. Encomendero, F. A. Faria, S. M. Islam, V. Protasenko, S. Rou-vimov, B. Sensale-Rodriguez, P. Fay, D. Jena, and H. G. Xing, "New tunneling features in polar III-Nitride resonant tunneling diodes," *Phys. Rev. X* 7, 041017 (2017).

Community News: Lynden Archer

The Cornell Energy Systems Institute, formerly named the Cornell Energy Institute, has a new director and a new focus as one of the university's main hubs for energy research and education.

Lynden Archer will serve a five-year term as the institute's David Croll Director, succeeding Jefferson Tester, who is now the chief scientist for Cornell's Earth Source Heat project. Archer is a professor and the former director of the Smith School of Chemical and Biomolecular Engineering, and a CNF principal investigator, where he has researched transport processes for energy storage and carbon-capture technologies since 2000.

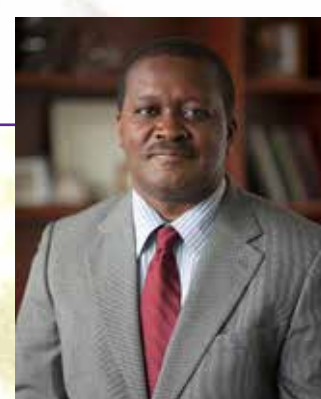
"He's one of the leading researchers in the area of energy, so Lynden brings great credibility in his ability to lead by example," said Lance Collins, the Joseph Silbert Dean of Engineering. "He's also an extraordinarily strategic thinker and an incredible fundraiser."

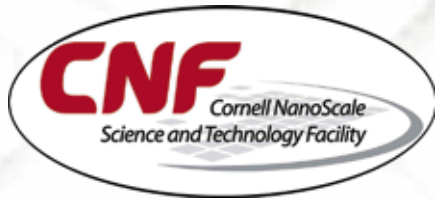
Archer's work with lithium-metal batteries and nanoscale organic hybrid materials earned him a spot on the "World's Most Influential Scientific Minds" list by Thompson Reuters in 2015, and his demonstration of an electrochemical cell capable of sequestering carbon from the atmosphere and turning it into electricity was named one of the "10 Ideas That Will Change the World" by Scientific American in 2016.

The Energy Systems Institute is run by the College of Engineering, but organizes energy engineering-based research and education across the university. While it will remain focused on the challenge of reducing carbon emissions without lowering quality of life, Archer said under his leadership the institute will more narrowly focus on enabling innovations in materials, technology and energy systems designs for impacts in transportation, manufacturing and the electric power grid.

In collaboration with the College of Engineering, the institute will identify and recruit faculty research stars to Cornell with a specific purpose: to make transportation and manufacturing systems data-driven, dynamic components of electric power networks. Through cross-college collaborations, the institute will build Cornell expertise, including in agriculture and chemical synthesis, for converting carbon dioxide emissions to useful products such as biofuels and plastics.

With a sharpened focus on three research themes, the institute also expects to play a stronger role in commercializing technologies developed at Cornell. There aren't many U.S. companies investing heavily in emerging technologies such as carbon-capture and fuel cells, and as a result, researchers must bear more of the responsibility for bringing their ideas to market, according to Collins.





Announcing the CNF-Fraunhofer IPMS Partnership



The Fraunhofer Institute for Photonic Microsystems (IPMS) in Dresden, Germany and the Cornell NanoScale Science and Technology Facility (CNF) at Cornell University in Ithaca, NY are pleased to announce a partnership to accelerate nanotechnology commercialization. The two partners have completed a tool map that allows small companies to plan the growth of their R&D prototyping activities into commercial foundries by developing their micro/nanofabrication process in a planned way that minimizes technology disruptions.

The Cornell NanoScale Facility at Cornell University is known for its flexible prototyping facilities and engineering support during product research and development stages. CNF now adds the Fraunhofer Institute to its list of partners that can provide a significant commercial ramp-up and volume manufacturing for clients who wish to grow their silicon-related and MEMS businesses beyond what CNF can provide.

Fraunhofer offers microelectronic R&D and pilot manufacturing solutions for various application fields and at institutes across Germany. In this initial partnership, the CNF-Fraunhofer related tool map is focused on silicon-related processes available at Fraunhofer IPMS facilities in Dresden, which make two clean room facilities available:

1) A 200mm wafer MEMS cleanroom (1500 m² / 15,000 ft²; ISO 14644-1 class 4) in which they offer complete solutions for micromechanical and photonic microsystems from conception to component up to complete systems. Furthermore, Fraunhofer IPMS provides established links to X-FAB Dresden, a leading analog / mixed signal foundry in close vicinity, for seamless transfer to high volume production.

2) A 300mm wafer advanced CMOS cleanroom (1000 m²; ISO 14644-1 class 6) at the Center Nanoelectronic Technologies (CNT) that offers a wide range of state-of-the-art industry standard processing and analytic equipment including a complete Copper Back-End-of-Line Module. The cleanroom is also qualified for wafer exchange (short loops) with Global Foundries Fab 1 Dresden and Infineon Dresden and so further scale-up solutions for advanced technology nodes and lithography access can be offered in cooperation with these industry partners.

Don Tennant, CNF Director of Operations, stated, "We are excited to have a partner where we can refer our small business customers to get the assistance they need to fully commercialize their concepts and prototypes in the CMOS, Silicon Photonics, and MEMS space. We also hope that cross referrals to CNF will allow us to provide services to Fraunhofer clients who are not yet at the scale-up stage and require the flexibility to explore a variety of approaches."

Martin Landgraf, Program Coordinator for the Fraunhofer Institute for Photonic Microsystems (IPMS), added, "We are delighted to have this cooperation in place. With our recent multi-million investments in new process equipment and tool upgrades we have enlarged our wafer processing capacity significantly to serve fabless or fablite customers worldwide.

Clients also benefit from our vast experience in lab-to-fab services, our strong network and the quick availability of our semiconductor consumables screening platform capabilities."

The two sites plan to post the tool map on their customer websites to allow small businesses to plan their path to product introduction earlier in the prototype stages of their work.

Fraunhofer-Gesellschaft is the leading organization for applied research in Europe. Its research activities are conducted by 72 institutes and research units at locations throughout Germany. The Fraunhofer IPMS is located in Dresden, Germany where maintains two cleanroom facilities. For specific requests at Fraunhofer IPMS solutions on 200mm or 300mm, please contact: Martin Landgraf, phone +49 351 2607-3004, martin.landgraf@ipms.fraunhofer.de

The Cornell NanoScale Science and Technology Facility (www.cnf.cornell.edu) operates a 17,000 sq. ft. cleanroom facility at Cornell University in Ithaca, NY. CNF is an open multi-user facility operated with funding from the National Science Foundation, NYSTAR Empire State Development, and Cornell University. For further information, contact: information@cnf.cornell.edu



Cornell NanoScale Facility :: 2018 Annual Meeting ::

Wednesday, October 3, 2018

The 2018 Cornell NanoScale Facility Annual Meeting will be held Wednesday, October 3rd. Please note this is a very different month and day from the usual!

**Registration is now open!
Please plan to join us!**

The CNF Annual Meeting is a special day-long event and an excellent opportunity for our colleagues to learn of the exciting research carried out by CNF users over the past year. The day will start with a plenary speaker and presentations from over a dozen experienced CNF users. We'll follow up the presentations with a Poster Session & Corporate Soiree. During this time, researchers will present posters of their work at the CNF, while our sponsoring companies will have display space in order to meet users, review resumes and talk about their job options and internships.

**We look forward to
welcoming you to the CNF!**

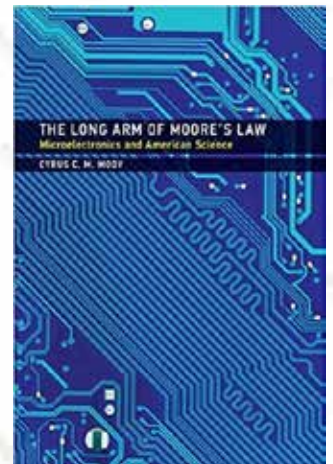


**2018 CNF Annual Meeting
Plenary Speaker**

**Professor
Cyrus Mody**

We are very pleased to announce our plenary speaker, Professor Cyrus Mody (C.C.M.): Chair in the History of Science, Technology, and Innovation; History Department / MUSTS Research Cluster; History, Faculty of Arts and Social Sciences; Maastricht University

Prof. Mody is the author of "The Long Arm of Moore's Law; Microelectronics and American Science" (MIT Press), which covers the pivotal role and early struggles of CNF (then NRRFSS).



http://www.cnf.cornell.edu/cnf_2018am.html



Scenes from the 2017 CNF Annual Meeting & 40th Anniversary Celebration. University Photo.

CNF Short Course: Technology & Characterization at the Nanoscale (CNF TCN)

Tuesday - Friday, June 5 - 8, 2018

This intensive 3.5 day short course offered by the Cornell NanoScale Science & Technology Facility, combines lectures and laboratory demonstrations designed to impart a broad understanding of the science and technology required to undertake research in nanoscience. TCN is an ideal way for faculty, students, post docs and staff members to rapidly come up to speed in many of the technologies that users of the CNF need to employ. Members of the high tech business community will also find it an effective way to learn best practices for success in a nanofab environment. Attendance is open to the general scientific community and is not limited to CNF users or Cornell students. It is suitable for both new and experienced researchers interested in nanoscale science. An emphasis will be placed on CNF laboratory resources, however, the concepts and techniques discussed are generally applicable to research in this field and do not require use of CNF.

PLEASE NOTE #1: The short course augments but does not replace the three part training required to become a user of our facility, nor does it replace tool trainings. To become a CNF user, one must follow the instructions under the "Getting Started" link online.

PLEASE NOTE #2: Thanks to funding from the National Science Foundation, this course is free for graduate students from U.S. institutions outside Cornell, up to five students per external university. (Travel and lodging are not included.) Students who wish to attend the course for free should contact Rebecca Vliet to ensure comp registration is available PRIOR to registering. When registering please make sure to indicate this by checking the appropriate box on the registration form.

COST

Academic Rate (including students and faculty) and Government Rate: \$425. Industrial Rate: \$850. As noted above, the course fee will be reimbursed for up to five research graduate students per university for grads from U.S. institutions outside Cornell.

OVERVIEW of COURSE

Tuesday, Day 1: Registration & Orientation (5:30p-7:30p)

Wednesday, Day 2: Introduction & Microfluidic Systems (8:45a-4:00p)

Thursday, Day 3: MEMS Cantilever Fabrication (8:45a-4:00p)

Friday, Day 4: Nanoelectrode Fabrication (8:45a-4:00p)

REGISTRATION

http://cnf.cornell.edu/cnf_tcn_june_2018.html



2018 CNF, KEP & PARADIM REU PROGRAMS

During the summer of 2018, Cornell NanoScale Science & Technology Facility (CNF), Keeping the Ezra Promise (KEP), and Platform for the Accelerated Realization, Analysis, and Discovery of Interface Materials (PARADIM) will sponsor Research Experience for Undergraduates (REU) programs from June through August. Engineering and science students with broad interests across disciplines focusing on nanotechnology applied – and our twelve interns are now locked in!

Mr. Vivek Anil, 2018 CNF REU Intern

The Pennsylvania State University

PI/Mentor: Gennady Shvets

Project: Topics in nano-biophotonics: Fabrication of plasmonic metasurfaces that attract & spectroscopically interrogate cancer cells

Mr. Miguel Castro-Rivera, 2018 PARADIM REU Intern

Interamerican University of Puerto Rico

PI/Mentor: Betül Pamuk

Project: Understanding the structure of IV-VI semiconductors

Mr. Anthony Coleman, 2018 PARADIM REU Intern

Chicago State University

PI/Mentor: Hanjong Paik (1 of 2)

Project: MBE growth/system development and XRD characterization

Ms. Stephanie Eberly, 2018 PARADIM REU Intern

North Carolina State University

PI/Mentor: Luca Moreschini

Project: Development and commissioning of a novel ARPES system

Ms. Iryna Glushchenko, 2018 PARADIM REU Intern

Princeton University

PI/Mentor: Hanjong Paik (2 of 2)

Project: MBE growth/system development and XRD characterization

Mr. Jordan Howard-Jennings, 2018 CNF REU Intern

Harvey Mudd College

PI/Mentor: Christopher Ober

Project: New Photopatterning Materials for Advanced Lithography

Mr. Cesar Lema, 2018 PARADIM REU Intern

New York University

PI/Mentor: Don Werder

Project: Growing 2D Transition Metal Dichalogenide (2D-TMDC) Materials for PARADIM Users

Mr. Marcus Marracci, 2018 PARADIM REU Intern

CSU Fullerton

PI/Mentor: Betül Pamuk and Don Werder

Project: Growing and Designing 2D Transition Metal Dichalogenide (2D-TMDC) Materials for PARADIM Users

Mr. Joshua Romo, 2018 KEP REU Intern

University of Dayton

PI/Mentor: Tobias Hanrath

Ms. Sierra Russell, 2018 CNF REU Intern

SUNY College of Nanoscale Science and Engineering (Albany)

PI/Mentor: Paul McEuen

Project: Magnetic Film Characterization for Actuating Origami Devices

Mr. Kody Whisnant, 2018 CNF REU Intern

Wayne State University

PI/Mentor: Abraham Stroock

Project: Development of MEMS tool to study the physics of water and ice

Mr. Reed Yalisove, 2018 CNF REU Intern

University of Michigan-Ann Arbor

PI/Mentor: James Engstrom

Project: Fabricating atomic membranes using ALD



The 2017 KEP REUs presented at the Robert Frederick Smith Chemical and Biomolecular Engineering Graduate Research Symposium in January, and Jadel got the chance to make his first ever snow-man! Photographs provided.



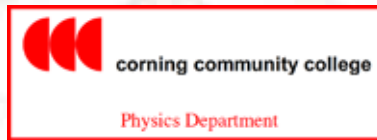
Scenes from the 2017 REU Program. Photos by Don Tennant.



OAOC NVE
 1914 County Hwy 35
 Maryland, NY 12116
 December 11, 2017



**2018
 CNF
 LEGO
 EXPO**



Professor Lawrence Josbeno
 Physics Department

April 1, 2018

To those who made our tours spectacular:

The NVE Class of 2018 would love to warmly thank you for such a wonderful tour of Cornell University. Reputably, Cornell's campus and classrooms easily utilize various technologies to ensure that their students gain the highest understanding of their choice major and beyond. Each engineering department tour resonated what to expect when attending an Ivy League college. As high school seniors soon to graduate, our visit to Cornell has been valuable in regard to our future plans. Not only has our visit been such a beneficial college insight, the learning experience was just as great.

scientific tools almost guarantees enjoyable and successful research. The Nano Tech Engineering presentation was beyond intriguing and the nano related activities, like exploring materials with the high resolution cameras, using the electron beam microscope, and manipulating the ferrofluid. (Hopefully, our instructor can be convinced to purchase a bottle for the class!) The virtual clean room tour, where all the nano related creations are built, was quick and focused and not to mention humorous.

Thank you again to the staff and students for the wonderful tours.

Sincerely, Amanda Pickwick, David Morell
 and the OAOC Engineering Class of 2018

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Dear Melanie-Claire,

Thank you for the tours of your facility for the Engineering Physics class from Corning Community College. These incredible experiences began for my Physics III class on Wednesday, March 7, 2018, when Michael Skvarla visited and gave a brilliant presentation. He demonstrated outstanding intellectual ability, academic achievement, a promethean depth of understanding, and a sense of humor. The level of his presentation was appropriate and entertaining. This is no mere presenter but a veritable tour-de-force.

The CNF tours were informative, entertaining, motivating and exciting. Edward Camacho and Dan McCollister, Jeremy Clark and Aaron Windsor, demonstrated outstanding ability, patience and cheerfulness. They were also entertaining and able to involve the students — in short they were outstanding. The students were talking about this experience and asking questions the entire trip back.

The trip to your lab has been an integral part of the physics program here for several years. Congratulations to all members of your facility for providing this excellent outreach experience. Thank you again for what was a rewarding experience for my students, and a very special thanks to Michael and Melanie-Claire who have been doing this for many years.

Sincerely yours, Lawrence Josbeno

Professor of Physics, Corning Community College

Yield Engineering Systems Image Reversal Oven



The new Yield Engineering Systems Image Reversal oven is now operational. New training is required. The new system implements pressure monitoring and safety features that were unavailable in the old tool.

The YES 58-TA oven uses NH_3 (ammonia) gas to reverse the tone of positive photoresist. This can be used to create an undercut profile in the photoresist for lift-off processing. In the reversal process, the chamber is purged of oxygen using vacuum and heat. It is then filled with 500 Torr of NH_3 (ammonia) vapor. The NH_3 reacts with the acid in the exposed resist rendering it insoluble in developer. The proceeding flood exposure causes acid to form in the previously unexposed areas allowing them to be removed in development, leaving behind the negative image of the first exposure. A descum process using O_2 plasma is recommended after lift-off to promote good adhesion of deposited material.

For more information and to be trained on the tool, contact Garry Bordonaro, bordonaro@cnf.cornell.edu



New CNFUsers Website

On Wednesday, March 21, 2018, CNF debuted its new CNFUsers website replacing the current ten-plus years old site.

The new site is hosted in a modern Drupal environment on Pantheon and uses modern web design paradigms. Mobile devices can use the responsive design elements to render the site on most any screen size including smart phones. New high quality tool pictures, the @CNFUsers twitter feed, the Cornell Events Calendar featuring CNF events, and a standardized maintainable layout are all parts of the new site. Contact CNF IT at computing@cnf.cornell.edu with questions or concerns.

Recommissioning of the Plasma-Therm 720/740 RIE System

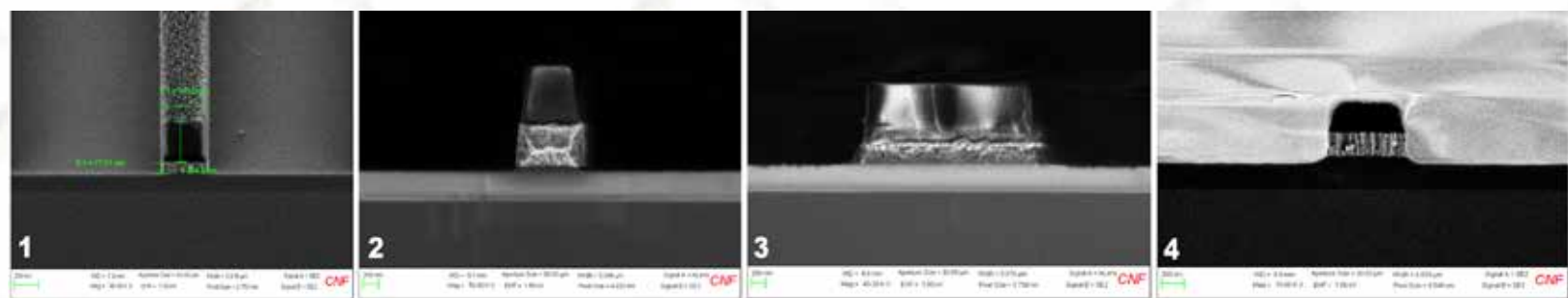
In response to the evolving needs of the CNF user community, we have recently recommissioned the chlorine based dual chamber Plasma-Therm 720/740 RIE system. For many years the PT740 has served as a dedicated metal etch chamber, while the PT720 was used for shallow silicon etching. The Plasma-Therm 770 left chamber ICP is now the dedicated metal etch system for the CNF. This has allowed us to rededicate the usage of the PT 720/740 RIE.

The PT720 RIE is being used to etch 2D transition metal di-chalcogenides (TMDs) including MoS_2 , SnS_2 , WSe_2 , NbSe_2 , GaSe , InSe , CdSe , and others. In addition, 2D metal oxides such as WO_3 , V_2O_5 , MnO_2 can be etched as well as layered 2D perovskites such as SrRuOx , and SrLaAlOx whether pure or mixed phase. These novel materials are used in the fabrication of high performance electronic and optoelectronic devices by an increasing number of faculty research groups here at CNF. We at CNF are fortunate to have a multitude of etch systems to allow us to dedicate systems for specific uses, including unconventional materials such as these, to avoid cross contamination and to ensure the integrity of the fabricated device. Even in these systems, we have adopted protocols to guarantee suitable chamber conditions for repeatable results.

The PT740 is now an RIE system which will accommodate the presence of gold on a substrate or wafer. Traditionally gold is not allowed in etch systems because of its high sputter yield and its high contamination potential which ultimately leads to the detriment of the fabricated device. Users are now allowed to etch down to a gold interface and have gold exposed in the course of an etch. The PT740 is being facilitated with CF_4 and CHF_3 in addition to chlorine based chemistry which has traditionally been there. The CF_4 and CHF_3 gases will allow the etching of silicon based dielectric films down to a gold interface. Of course all process flows must comply with the CNF Tool and Compatibility Policy to ensure that process restrictions are upheld for other tools once etching is performed in the PT740 or PT720.

The missions of the PT720 and PT740 will continue to evolve to meet the changing needs of the CNF user community. We will keep you informed of all the updates and if you have questions regarding usage of the PT720/740 or the CNF Tool and Compatibility Policy, please contact Vince Genova and Phil Infante respectively.





Newly Developed Metal Etch Processes at CNF

Submitted by Vince Genova
CNF Research Staff

With the arrival of the Oxford Cobra ICP system and the development of HBr based silicon etch processes for photonics applications, the Plasma-Therm 770 ICP left chamber has been designated the chlorine (Cl)-based metal ICP etch chamber.

Historically, the PT770L was the chlorine-based silicon etch chamber for shallow silicon etching and was used predominately by photonics-based researchers, along with the Plasma-Therm (Unaxis) 770 based SF_6/C_4F_8 mixed etch. There has been a long term need for ICP based metal etching at CNF, which up to now has had to use the conventional parallel plate Plasma-Therm 740 RIE system for all of its metal etch needs.

The addition of ICP metal etch capability enables users to etch high aspect ratio features of up to 20:1 at higher etch rates and higher selectivity due to the many benefits of the ICP. Because chamber wall conditions are critical for reproducible Cl-based metal etches, we have decided to segregate Cl-based metal etch processes from fluorine-based metal etches. Therefore, those metals that preferably etch in fluorine chemistry, such as tungsten and TiW, now have etch processes established on the Oxford Cobra ICP.

Thus far we have developed etch processes on the PT770L for the following materials: aluminum, aluminum-silicon-copper, aluminum oxide, chrome, titanium, tantalum, niobium, molybdenum, molyb-

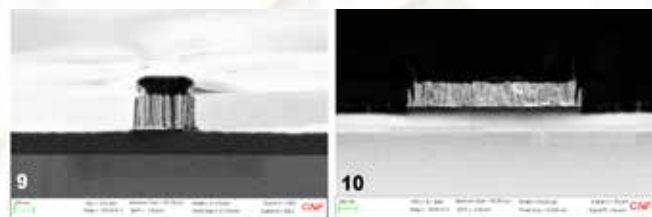
denum di-silicide, and titanium nitride. Processes for tantalum nitride and aluminum nitride are pending.

Results for some of the PT770L metal etch processes are illustrated above and below.

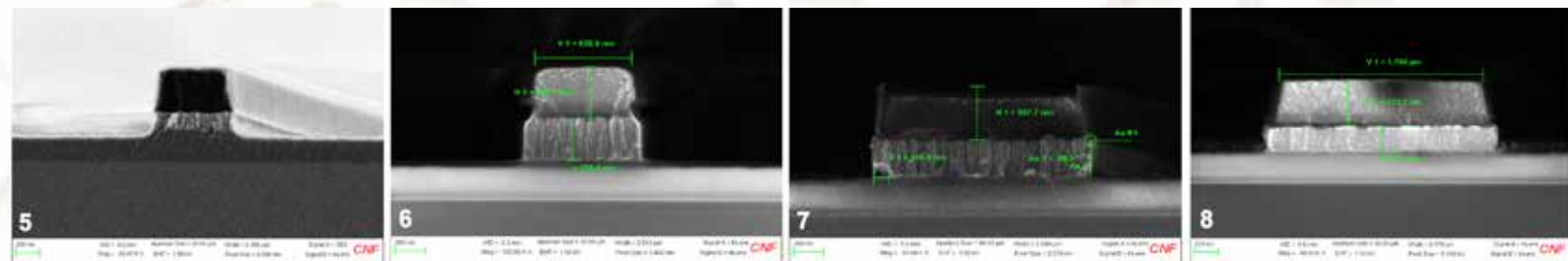
- Figure 1: Chrome etch ($Cl_2/O_2/Ar$)
- Figure 2: Al etch ($BCl_3/Cl_2/CH_4/Ar$)
- Figure 3: Al-Si-Cu etch: $BCl_3/Cl_2/CH_4/Ar$
- Figure 4: Ta etch $Cl_2/BCl_3/Ar$
- Figure 5: Ti etch $Cl_2/BCl_3/Ar$
- Figure 6: Nb etch ($Cl_2/BCl_3/Ar$)
- Figure 7: Mo etch ($Cl_2/O_2/Ar$)
- Figure 8: $MoSi_2$ etch- $Cl_2/O_2/Ar$

The following metal etch processes are available in the Oxford Cobra ICP:

- Figure 9: W etch $SF_6/CH_3OH/Ar$
- Figure 10: TiW etch $SF_6/BCl_3/Cl_2/Ar$



For more details on ICP based metal etching at the CNF, please contact Vince Genova at genova@cnf.cornell.edu



A Comparison of Silicon and Germanium Deep Reactive Ion Etching and its Application to X-Ray Optics

Vincent J. Genova (CNF)
David N. Agyeman-Badu (MSE/CHES)
Arthur R. Woll (CHES)

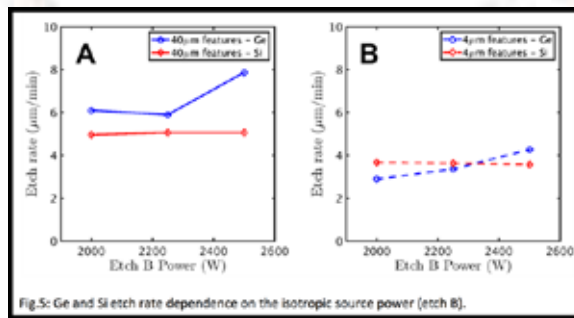
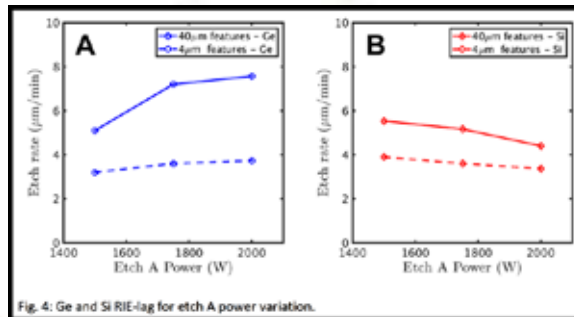
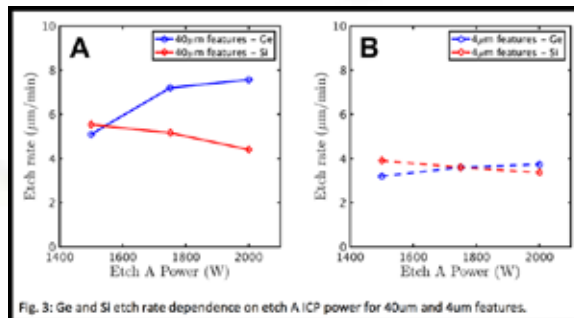
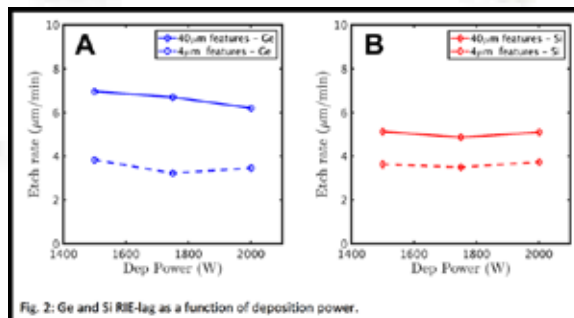
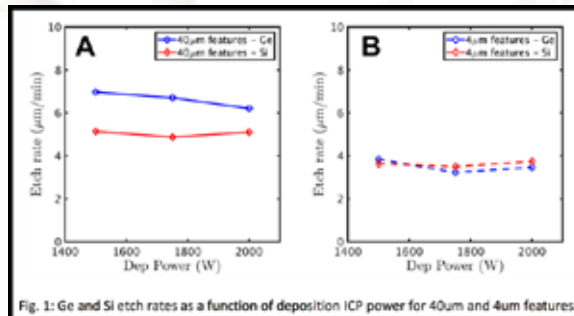
A collaborative research effort between CNF and CHES scientists has resulted in publication in the Journal of Vacuum Science and Technology B 36(1), Jan/Feb. 2018. This work was also presented by Vince Genova at the 61st International Conference on Electron, Ion and Photon Beam Technology & Nanofabrication in Orlando, FL, in May 2017.

Despite the technological importance of germanium (Ge) in electronics, photonics, and x-ray optics, relatively little by comparison with silicon, has been published concerning the etch mechanisms in halogen-based chemistry. Furthermore, although deep reactive ion etching (DRIE) has been discussed extensively for decades, very little work has been directed towards understanding the fundamental mechanisms of Ge deep reactive ion etching. By directly comparing silicon (Si) and Ge etching in a time multiplexed DRIE process, we extract significant differences in etch mechanisms from a design of experiment (DOE) and discuss how these differences are relevant to the design and fabrication of Si and Ge collimating channel array (CCA) x-ray optics.

We believe that there are fundamental differences between Si and Ge that are responsible for the observed differences in DRIE using SF₆ and C₄F₈ based plasmas. Among these are the lower bond strength of Ge and its higher overall reactivity with atomic fluorine, which in most cases results in a higher Ge etch rate. In addition, the lower thermal conductivity of Ge plays a key role in the passivation step of the multiplexed DRIE process and its resulting etch profile.

Si and Ge wafers were prepared in an identical manner using photoresist and silicon dioxide as masks for the Bosch etch. The silicon dioxide was deposited by plasma enhanced chemical vapor deposition (PECVD). The wafers were patterned in the ASML 300C deep ultra violet (DUV) stepper resulting in linewidths ranging from ten of microns to less than 0.5 μm. The photoresist masked wafers were patterned with a 3.5 μm thick GKR DUV resist. The Bosch etch was performed in the Plasma-Therm Versaline DSEIII DRIE system. In order to better understand the respective mechanisms of Si and Ge deep etches, a Taguchi L-9 orthogonal partial factorial DOE was executed where the ICP powers of the three steps of the Bosch process, along with the peak-to-peak voltage applied to the wafer electrode were varied. All other etch parameters including flow rates, pressures, and step times were held constant.

Important outcomes of the DOE are the measured etch rates of Si and Ge as a function of the ICP powers of the polymer deposition step (Dep), polymer removal step (etch A), and the isotropic etch step (etch B). Etch rates were also monitored as a function of peak-to-peak voltage (V_{pp}) in the polymer removal step (etch A). The etch rate responses were specifically measured for 40 μm and 4 μm feature sizes. The variations in the Si and Ge etch rates for these feature sizes as a function of the source power in the deposition step are illustrated in Figure 1.



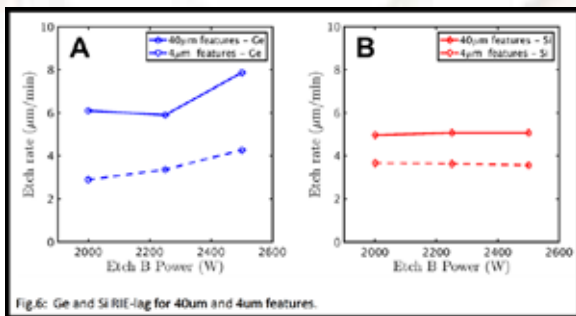


Fig.6: Ge and Si RIE-lag for 40µm and 4µm features.

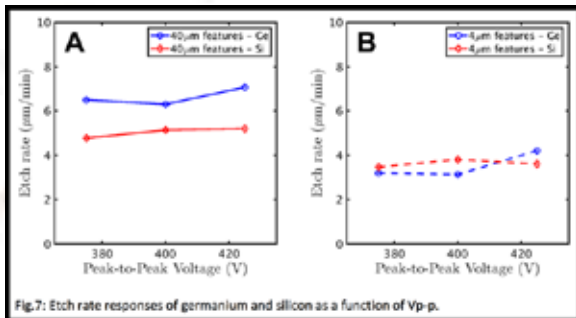


Fig.7: Etch rate responses of germanium and silicon as a function of Vp-p.

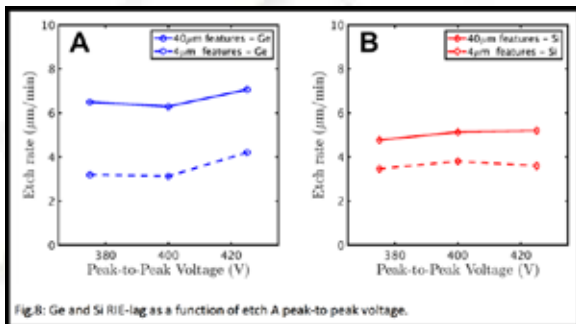


Fig.8: Ge and Si RIE-lag as a function of etch A peak-to-peak voltage.

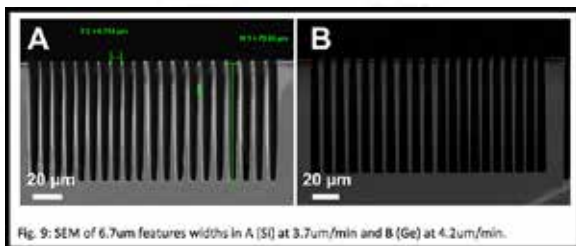


Fig. 9: SEM of 6.7µm features widths in A (Si) at 3.7µm/min and B (Ge) at 4.2µm/min.

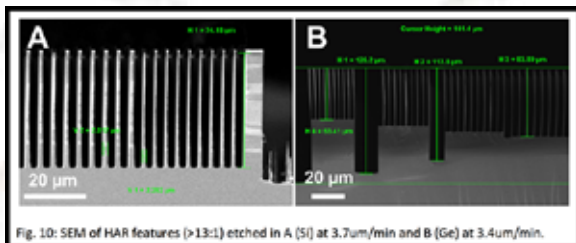


Fig. 10: SEM of HAR features (>13:1) etched in A (Si) at 3.7µm/min and B (Ge) at 3.4µm/min.

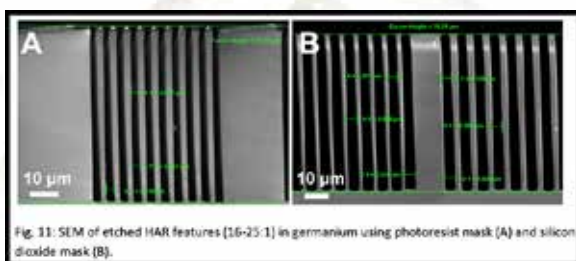


Fig. 11: SEM of etched HAR features [16-25:1] in germanium using photoresist mask (A) and silicon dioxide mask (B).

Increasing the source power in the polymer step increases the dissociation of C_4F_8 into major fragments of CF_2 and CF . The Ge etch rate appears to be more affected by the increasing polymerization for both feature sizes, which indicates that the in-diffusion of atomic fluorine is more constrained for Ge. Figure 2 shows the respective differences in etch rates for Si and Ge for different feature sizes (i.e., RIE-lag). The variation in the ICP power within the polymer removal step (etch A) and its effect on the Si and Ge etch rates is depicted in Figure 3. The decreasing Si etch rate with source power in the polymer removal step (etch A) seems to indicate a more robust polymer boundary on its surface and one that is seemingly unaffected by the increased fluorine radicals. The respective Si and Ge RIE-lags for the variation in etch A source power are illustrated in Figure 4.

RIE-lag, which is a manifestation of the relative etch rates among features of different sizes, is largely due to greater restriction on the diffusion of neutral or radical species within narrower features. Germanium's greater RIE-lag is indicative of its chemical dependence on atomic fluorine, while silicon appears to be less impacted, suggesting a larger ion-enhancement component. The Ge and Si etch rate responses as a function of the isotropic etch (etch B) source power are shown in Figure 5.

The isotropic etch step (etch B) is dependent on the dissociation of SF_6 and increasing the ICP source power results in increasing amounts of atomic fluorine and the generation of volatile SiF_4 and GeF_4 reaction products. The Ge etch rate responds favorably to increases in atomic fluorine and the increasingly chemical nature of the plasma. The respective Ge and Si RIE-lags as a function of etch B source power are shown in Figure 6. The 46% decrease in Ge etch rate at the 4 µm feature size is again indicative of the highly chemical dependence of Ge in fluorine-based chemistry and its response to a decreased transport of chemical species at higher aspect ratios. The etch rate responses as a function of peak-to-peak voltage within the polymer removal step of etch A are shown in Figure 7.

The ion energies within this step are largely responsible for etching the polymer at the base of the trench. The somewhat larger response of the Ge etch rate may suggest a thinner fluorocarbon layer, whereby increasing ion energies would result in greater penetration of reactive ions and the resulting substrate bond breakage making the more reactive Ge surface even more susceptible to chemical attack. Silicon's smaller decrease in etch rate with feature size as shown in Figure 8 would suggest a greater reliance on ion enhancement and may be attributed to the fact that ion transport is less affected by constricted feature size.

Note that consistently higher Ge etch rate for the larger 40 µm feature size. Germanium's higher etch rate is quickly erased as the feature size is reduced by an order of magnitude. This demonstrates the impact of RIE-lag and how the largely chemical nature of Ge etching is impacted by the reduction in the transport of chemical reactants down the constricted trench width. Specifically, we determined that for aspect ratios less than 13:1, Ge etch rates exceeded those of Si under identical DOE conditions. Furthermore, a noticeable difference between Ge and Si is seen in the resulting etch profiles. The Si etch profile shows a very distinct tapered profile with an angle of 88° especially for higher aspect ratio features, while the Ge etch displays a non-tapered anisotropic profile with an angle of approximately 90° as shown in Figure 9.

Notice that the aspect ratio in Figure 9 is less than 13:1 and that the Ge etch rate exceeds that of Si. Figure 10 illustrates aspect ratios greater than

13:1 for a 5 μm feature where the Ge etch rate begins to fall behind the Si etch rate.

Examples of high aspect ratio (16-25:1) Ge etched structures having straight non-tapered profiles with photoresist and silicon dioxide masks are shown respectively in Figure 11-A and 11-B.

Tapered profiles typically result from the build-up of polymer on the sidewall as the etch proceeds with time. This would suggest an enhanced polymerization of the Si sidewall with depth as opposed to the Ge profile, which displays no enhancement with depth. Another very interesting contrast between the Si and Ge profiles is the more noticeable scalloping on the silicon sidewalls. This is apparent in Figure 12 where despite the short 1.5 sec. step time, the scalloping of Si is discernable. We believe that the smoother sidewalls of the Ge etch are due to the thinner polymer formation as a result of the lower thermal conductivity of Ge compared to Si. Thereby the thinner polymer may be more susceptible to attack by the in-diffusion of atomic fluorine radicals, which can serve as smoothing mechanism.

Because chemical analysis of sidewall polymer can be difficult due to the delicate nature of high aspect ratio DRIE features, we used a small gap structure to simulate a trench etched by DRIE. Structures were constructed with silicon and germanium wafers along with spacers of each material supporting a roof/shadow wafer piece as shown in Fig.14.

Because the base wafer extends beyond the shadowing piece, exposure in this region is by ions and neutrals that interact to form a fluorocarbon layer. This exposed region is representative of the trench bottoms within DRIE. The transition region near the boundary of the shadowing piece has largely exposure to neutrals and some off-axis ions in the formation of polymer that would be representative of the top trench regions of DRIE. The fully shadowed region under the roof piece sees only neutrals and is characteristic of mid-trench regions of a deep etched structure. One gap structure of Si and one of Ge were each exposed to identical standard Bosch etch conditions for 240 cycles. Chemical analysis of polymer present in each of the three regions was performed by X-ray Photoelectron Spectroscopy (XPS) in CCMR. Deconvolution of the high resolution scans of the C1s peak reveal CF_2 , CF, and CF_3 peaks, which are the dominant molecular fragments from the dissociation of the C_4F_8 polymer precursor. The C1s high resolution scans from the completely shadowed region of the gap structure from Si and Ge are shown in Figure 15 A and B respectively.

The polymer in the completely shadowed region is representative of that on the mid-trench sidewall, and although the composition of the polymer on the Si and Ge surfaces is chemically similar, the intensities of the major CF_2 and CF peaks are 25% lower on the Ge surface. High resolution C1s scans from the transition region near the boundaries of the shadow piece are shown in Figure 16 for Si and Ge. The transition region is an area that has exposure to radicals and some off-axis ions and is representative of the top region of the trench. Again compositionally, the polymer on the trench top edge of both Si and Ge is very similar, however, the intensities of the major peaks on Ge are lower by 14.5% for CF_2 and 5.9% for CF. These results support our contention that there is less polymer formation on the Ge surface than the Si surface under identical processing conditions. The fact that Si has a thermal conductivity twice that of Ge can play a major role in this observation. The thinner sidewall polymer would make it more susceptible to

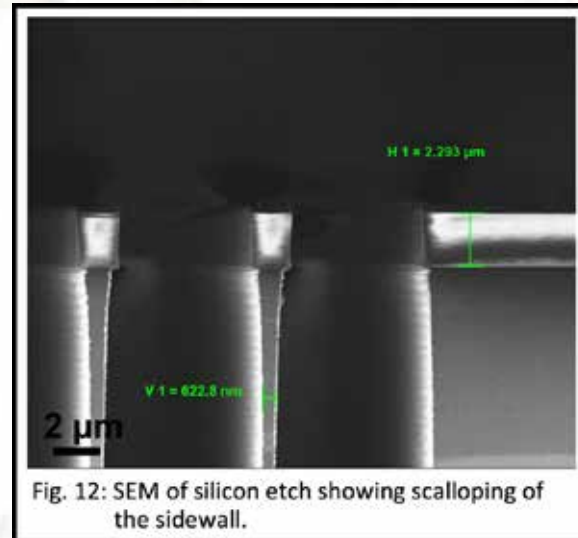


Fig. 12: SEM of silicon etch showing scalloping of the sidewall.

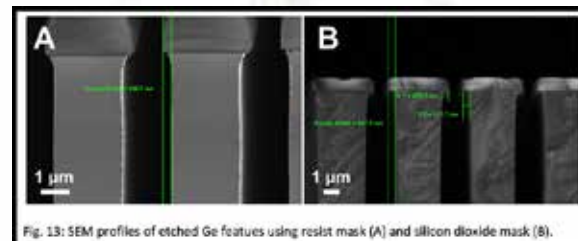


Fig. 13: SEM profiles of etched Ge features using resist mask (A) and silicon dioxide mask (B).

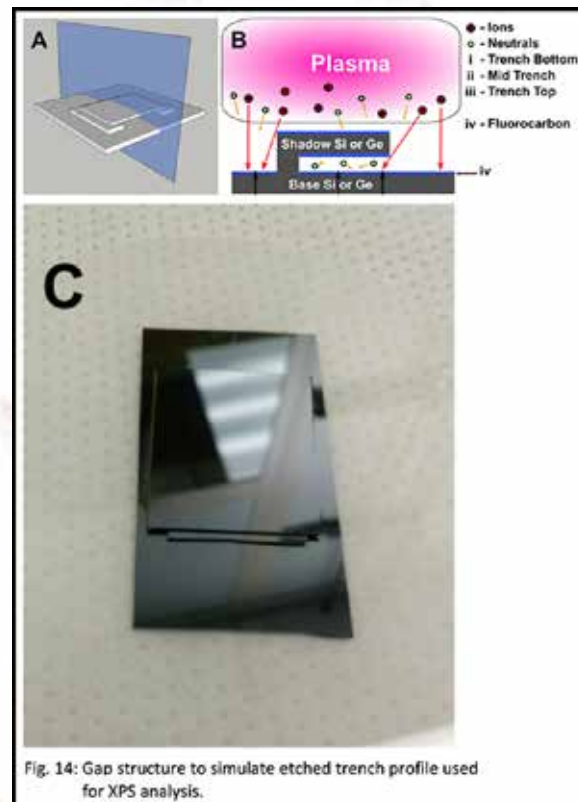


Fig. 14: Gap structure to simulate etched trench profile used for XPS analysis.

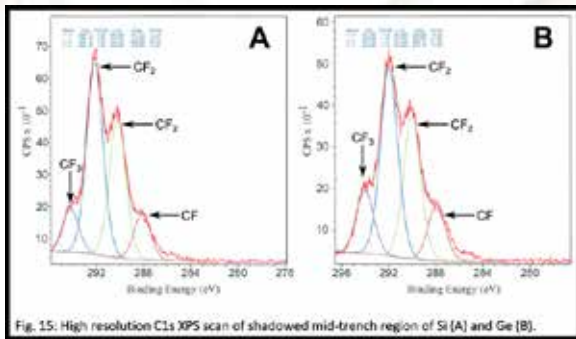


Fig. 15: High resolution C1s XPS scan of shadowed mid-trench region of Si (A) and Ge (B).

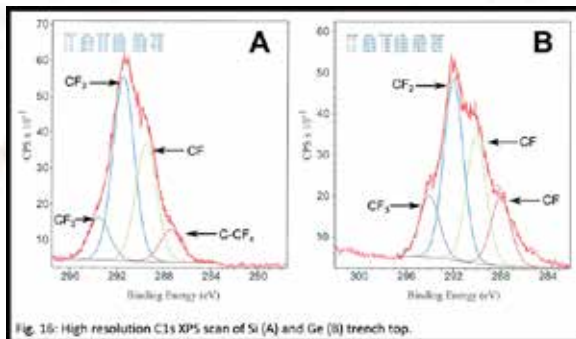


Fig. 16: High resolution C1s XPS scan of Si (A) and Ge (B) trench top.

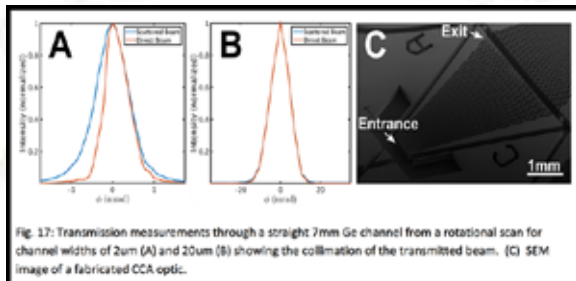


Fig. 17: Transmission measurements through a straight 7mm Ge channel from a rotational scan for channel widths of 2um (A) and 20um (B) showing the collimation of the transmitted beam. (C) SEM image of a fabricated CCA optic.



Acknowledgements:

This work is based upon research conducted at the Cornell High Energy Synchrotron Source (CHESS), which is supported by the National Science Foundation and the National Institutes of Health/National Institute of General Medical Sciences under NSF award DMR-1332208. This work benefited from the facilities and expertise at the Cornell NanoScale Science and Technology Facility, a shared national user facility funded in part by the NSF-NNCI program, grant no. ECCS-1542081, and New York State. This work made use of the Cornell Center of Materials Research Shared Facilities, which are supported through the NSF MRSEC program (DMR-1120296). The authors would like to thank Darren Dale for his expertise in XPS measurements and analysis.

chemical attack by atomic fluorine, and could contribute to smoother Ge sidewall surfaces. Another interesting observation of the Si scans is that the intensity of fluorocarbon peaks increases from the top trench to the mid-trench region. This suggests more polymer deposition descending into the trench and is supportive of the tapered etch profile revealed in the SEM cross sections.

In contrast, the major fluorocarbon peak intensities of the top trench and mid-trench Ge scans illustrate a constant intensity along the depth of the trench. This is in accord with the Ge SEM micrographs showing a highly anisotropic non-tapered etch profile. Scans of the completely exposed areas of the Si and Ge gap structures, which are representative of the trench bottoms, are rather uninteresting and only show the presence of adventitious carbon arising from atmospheric exposure. This is expected since the Bosch process terminates with an etch step and the presence of polymer is not expected.

The goal of CCA x-ray optics development at the Cornell High Energy Synchrotron Source (CHESS) is for 3D high resolution confocal x-ray fluorescence (CXRF) microscopy. There are several advantages in using a lithographically fabricated optic approach. First, the dimensions of the geometry of the fabricated optic define the probe volume of interest. This feature represents a significant improvement compared to the state of the art polycapillaries that have an energy dependent resolution. The energy invariant resolution of CCAs makes it possible to image heterogeneous elemental samples without the loss of detail from the lower energy elements. For CCAs, a given resolution is related to the channel width (w), the geometric collimation (δ) and the working distance (f) as shown by $r = w + \delta f$. Therefore, for a given channel width and working distance constraint, being able to adjust the geometric collimation close to the nominal resolution is desirable.

Secondly, since these optics are etched, optimizing the DRIE process to attain deeper channels increases the solid angle of the fluorescent photons collected and ultimately the efficiency of the optic. While silicon substrates have been used for fabricating CCA optics, germanium has shown great promise. CHESS has demonstrated that the more attenuating germanium CCAs effectively suppress background radiation in CXRF and extend the energy range of operation from 1.7keV to 20keV. This is a huge improvement considering that silicon CCAs begin to fail at 10keV. The design of CCA channels that radially span from a common focal position are defined by pillars as shown in Figure 17C.

Note that for this design, the critical dimensions for the pillars at the front of the optic are smaller compared to the exit. As a result, there is an inherent micro-loading effect imposed on the optic which spans from the front where the pillars are close-packed (8 μm wide pillars, 2 μm spacing) to the exit where they are more sparsely arranged (40 μm wide pillars, 40 μm spacing). Hence for CCAs, RIE-lag is desirable unlike in most MEMS applications where there is considerable effort to minimize this effect. The etch depth is preferred to be shallower at the front and very deep at the exit. The advantage of slower etch rates at the front helps the pillars maintain their delicate structure, while simultaneously being able to etch as deeply as possible at the exit. From the DOE results presented here, the combination of enhanced RIE-lag with the reduced etch rate of the smaller 4 μm features and the higher etch rate of the 40 μm features in germanium, make germanium preferable for CCA optic design and fabrication.

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