Cornell NanoScale Science & Technology Facility

NanoMeter

SPRING 2014 Volume 23, Issue 1

welcome

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2013 CNF Annual Meeting Corporate Sponsors

The CNF is grateful for the support of the following corporations that sponsored the 2013 CNF Annual Meeting. If your company would like to sponsor this year's meeting (being held Thursday, September 18, 2014), please contact Don Tennant, CNF Director of Operations, at tennant@cnf.cornell.edu.

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The cover image for this NanoMeter is an example of 3D printing, performed for S.H. Cheong, DUM Ph.D., Diplomate of the American College of Theriogenologists, Department of Clinical Sciences, College of Veterinary Medicine, Cornell University. The interior background image is from 2013 CNF REU Intern Alice Perin. Alice worked with PI Prof. Daniel Ralph and Mentor Greg Stiehl on "Spin Manipulation in Antiferromagnetic Devices." All the NNIN REU Program reports are online at http://www.nnin.org/reu

Photographs in this issue were taken by CNF staff, especially Don Tennant and Sam Wright. Research photographs are credited as noted.

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Welcome to the 2014 Spring Edition of the NanoMeter

The big news over the last few months has involved decisions by National Science Foundation (NSF) about the competition for the Next Generation National Nanotechnology Infrastructure Network (NG-NNIN), which had been planned as the means by which NSF would fund shared open user facilities for nanotechnology over the next ten years. Most of you have already heard this news via our emails to you. Following more than two years of work, the NSF has announced that they will be not be funding either of the proposals that were submitted as part of this competition. However, they have reaffirmed their support of shared facilities which provide nanotechnology infrastructure, and to that end, the NSF has provided an extra year of bridge funding for the existing network of facilities, including CNF. It appears that they are planning to start a new competition all over again from the beginning.

The official NSF announcement is available at http://www.nsf.gov/news/news_summ.jsp?cntn_id=131012.

After some reflection, we have come to think that this cancellation of the NG-NNIN competition may not be a bad thing. We expect that we may not have been told all of the reasons for the cancellation, but from our point of view the original call for proposals was flawed in that it outlined a greatly-expanded mission without providing sufficient funding to achieve that mission. We have suggested to the NSF that they should engage with the national nanoscience and nanoengineering community to set more-realistic priorities about what should be the mission of the NSF-supported user facilities in this field, priorities that are consistent with budget constraints. We hope that all of you will take advantage of your opportunities, if they come, to help the NSF set appropriate goals. During the recent debate within NSF about the NG-NNIN program, hundreds and hundreds of you wrote to the NSF, and we have been told that these communications had a big effect in making clear to the leadership at NSF that shared facilities are critical for progress in nanoscience and nanotechnology throughout the USA.

We will continue to keep you up to date via email about any news concerning NSF funding. If you would like to be added to our email distribution list, please send a note to Ms. Melanie-Claire Mallison at mallison@cnf.cornell.edu.



Photograph by Christopher Hallman, University Photograph

New Equipment:

In somewhat happier news, the CNF has been able to acquire many new instruments and upgrades over the last year, that are now in various stages of installation or availability.

We acquired an Oxford Cobra ICP etcher to provide HBr etching of silicon nanophotonics and methanol etching of magnetic materials; a new Ion Mill from AJA that is being installed to replace the Veeco; a new IRise infrared imaging microscope from Schott-Moritex for inspecting wafer bonds; a Heidelberg DWL66 Mask Maker that will complement our DWL 2000; an Anatech Resist Asher that will eventually replace our Branson; a dedicated KOH bath for anisotropic silicon etching; spin and bake capabilities for 300 mm wafers for e-beam lithography; a dedicated pre-alignment microscope for the JEOL 9500FSZ e-beam lithography system; and new beam scanners and power supplies for the odd-hour and even-hour evaporators. Installation will soon begin on new facilities for LPCUD TEOS and a new Arradiance Atomic Layer Deposition system that will expand our array of ALD materials and will allow ALD on powders.

Please mark your calendars for our next *Annual Meeting* of users and vendor partners on *September 18*, where we will highlight some of the research being done at CNF and the new capabilities of our toolset. Please also take note of the other upcoming events listed in this NanoMeter, and check our web site often for news updates.

Dan Ralph Lester B. Knight Director, CNF

Don Tennant CNF Director of Operations

Picture of Health: A Selfie That May Save Your Life



Engineering graduate students Matthew Mancuso, left, and Vlad Oncescu demonstrate their smartCARD system on a iPhone. These devices were printed using the new 3D Printer at the CNF. Jason Koski/UP

With a new smartphone device, you can now take an accurate iPhone camera selfie that could save your life – it reads your cholesterol level in about a minute.

Forget those clumsy, complicated, home cholesterol-testing devices. Cornell engineers have created the Smartphone Cholesterol Application for Rapid Diagnostics, or "smartCARD," which employs your smartphone's camera to read your cholesterol level.

"Smartphones have the potential to address health issues by eliminating the need for specialized equipment," said David Erickson, Cornell associate professor of mechanical engineering and senior author on a new peerreviewed study. Thanks to advanced, sophisticated camera technology, Erickson and his colleagues have created a smartphone accessory that optically detects biomarkers in a drop of blood, sweat or saliva. The new application then discerns the results using color analysis.

When a user puts a drop of blood on the cholesterol test strip, it processes the blood through separation steps and chemical reactions. The strip is then ready for colorimetric analysis by the smartphone application.

The smartCARD accessory – which looks somewhat like a smartphone credit card reader – clamps over the phone's camera. Its built-in flash provides uniform, diffused light to illuminate the test strip that fits into the smartCARD reader. The application in the phone calibrates the hue saturation to the image's color values on the cholesterol test strip, and the results appear on your phone.

Currently, the test measures total cholesterol. The Erickson lab is working to break out those numbers in LDL ("bad" cholesterol), HDL ("good" cholesterol) and triglyceride By Blaine Friedlander, Cornell Chronicle December 11, 2013

measurements. The lab is also working on detecting vitamin D levels, and has previously demonstrated smartphone tests for periodontitis and sweat electrolyte levels.

The article, "Cholesterol Testing on a Smartphone," appeared online Nov. 28 in the journal Lab on a Chip, also co-authored by Vlad Oncescu and Matthew Mancuso, Cornell graduate students in the field of engineering. This study was funded by the National Science Foundation, the Engineering Research Council of Canada and Cornell's David R. Atkinson Center for a Sustainable Future.

In a related study, the Atkinson Center provided academic venture funding on an application that analyzes micronutrients on a smartphone – so that phones can track micronutrient deficiencies for world populations.

Working on this project are Erickson; Saurabh Mehta, Cornell assistant professor of nutritional sciences; Julia Finkelstein, researcher in nutritional sciences; and Joe Francis, associate professor in development sociology.

"By 2016, there will be an estimated 260 million smartphones in use in the United States. Smartphones are ubiquitous," said Erickson, adding that although smartCARD is ready to be brought to market immediately, he is optimistic that it will have even more of its advanced capabilities in less than a year. "Mobile health is increasing at an incredible rate," he concluded. "It's the next big thing."



David Erickson, associate professor of mechanical engineering, tests the smartCARD, which uses an application system to read cholesterol levels in about a minute. Jason Koski/UP

'Shaken, Not Stirred': Oscillator Drives Electron Spin

By Anne Ju, Cornell Chronicle December 3, 2013

Contrary to many textbook illustrations, electrons aren't just balls floating around an atom. In quantum theory, they're more like little tops, exhibiting "spin," and each creating its own tiny magnetic field. Learning how best to manipulate these spins could open up technological advances in everything from quantum computers to encryption protocols to highly sensitive detectors. Usually, scientists exert control over electron spins by applying magnetic fields. It is the same concept that gives us magnetic resonance imaging: A strong magnetic field influences the spins (in MRI's case, of the nuclei) inherent in billions of hydrogen atoms in the body, enough of which can be converted into medical images.



Above: A schematic drawing of the device, which includes a transducer that produces gigahertz-frequency standing waves within diamond. Evan MacQuarrie

Right: The researchers call their electron spin resonance demonstration "shaken, not stirred." While ordinarily spin resonance is induced by "stirring" a spin with a rotating magnetic field, in this work it results from "shaking" a diamond crystal with a mechanical oscillator. Evan MacQuarrie



A collaboration of physicists and

engineers has found a new way to control electron spins not with a magnetic field but with a mechanical oscillator -ademonstration of electron spin resonance that's "shaken, not stirred," said lead researcher Gregory Fuchs, assistant professor of applied and engineering physics (AEP).

Fuchs and the research team showed that an oscillator – a transducer moving at extremely high frequency – can drive the transitions of electron spins (a phenomenon called spin resonance), within defects commonly found in the crystal lattice of a diamond. Their results were published online Nov. 27 in the journal Physical Review Letters.

In conventional magnetic resonance, a rotating magnetic field twirls around at the same rate as the electrons "spin" - the magnetic field is "stirring" the spins. Instead, the Cornell researchers used an oscillator to "shake" the diamond lattice to directly flip the spins. Their experiment involved looking at electrons spins within a naturally occurring defect in the crystal lattice of a diamond, called a nitrogen-vacancy center. Spins found within these defects are a promising platform for studying quantum spin control. To complete the work, Fuchs collaborated with Sunil Bhave, associate professor of electrical and computer engineering, whose expertise is in high-frequency microelectromechanical systems (MEMS). They used a transducer, which acts like a small speaker, to vibrate the lattice by applying AC voltage. This created a standing wave inside the diamond, and the stress from these waves on the defect created the electron spin

resonance. They measured the values of the spin using a microscope that senses the fluorescence output of the spins. From the gigahertz-frequency oscillations, they demonstrated direct coupling of spin states within the diamond defects without the use of a magnetic field.

A device built around such a system could be useful in MEMS or for a precision sensor, Fuchs said. Their research is a breakthrough in the field of spin-based quantum technologies, in which scientists are trying to understand and control physical phenomena at the most fundamental scales. "We are just taking baby steps to try to understand these interactions, quantify them and see what can be done," Fuchs said. "What we've done is the very first thing – showing that you can do spin resonance using mechanical oscillations."

The paper is titled "Mechanical Spin Control of Nitrogen-Vacancy Centers in Diamond." Its first author is Evan MacQuarrie, a graduate student in physics, and included work by Tanay Gosavi, a graduate student in the field of electrical and computer engineering, and Nicholas Jungwirth, a graduate student in physics.

The research was supported by a seed grant from the Cornell Center for Materials Research, which is funded by the National Science Foundation (NSF). The Department of Energy also contributed to the research. Device fabrication was performed at the NSF-supported CNF.

Thing Power[™] - BOLT[™] Power Cell Energizes 'Internet of Things' Applications

Rochester, NY (PRWEB) November 19, 2013

MicroGen Wins 2013 MEMS Technology Showcase® with Piezo-MEMS Vibrational Energy Harvesting



Two form-factors for MicoGen's BOLT Power Cell that contains an MPG (purple unit), efficient AC-DC conversion and DC voltage regulation electronics, and energy storage.

MicroGen Systems, Inc. (MicroGen) has developed the first commercially available piezoelectric microelectromechanical systems (piezo-MEMS)-based vibrational energy harvester; a micro-power generator (MPG). MicroGen has created a DC power source by integrating its MPG with electronics and energy storage. The result is the BOLT[™] Power Cell (see above), which was one of two winners in the third annual MEMS Technology Showcase[®] at 2013 MEMS Executive Congress[®] in Napa, CA (MEMS Industry Group 11/14/13 press release).

MicroGen's BOLT Power Cells provide "Thing Power" – mechanical energy (e.g. vibration) converted to electrical power for electronics and sensors in 'Internet of Things' (IoT) applications. Potential IoT uses include, but are not limited to, industrial and building wireless sensors, transportation systems (e.g., plane, train and automobile sensors), asset tracking (e.g., livestock management and monitoring cargo), wearable electronics (e.g., health monitoring and gaming) and other consumer applications (e.g., humidity sensors in clothes dryers, toys) and for analogous DoD/Homeland Security purposes.

MicroGen's CEO, Dr. Robert Andosca, presented the MEMS Technology Showcase winning presentation and live on-stage power harvesting demonstration using mechanical vibration from a flying Parrot AR Drone 2.0 Quad Rotor Helicopter (see award winning video including demos, http://www.prweb.com/releases/2013/11/prweb11344903.htm).

"I am honored to have MicroGen and its BOLT Power Cell chosen by our industry peers to have the best live demo." He further added, "We are excited to help enable the 'trillion sensor world' vision."

"We were very pleased with all the finalists' live demos at MEMS Technology Showcase," said Karen Lightman, Executive Director, MEMS Industry Group, the host organization of MEMS Executive Congress. "We especially congratulate MicroGen Systems, which so impressed attendees with the energy harvesting capabilities of its BOLT Power Cell that it tied for the number one spot to earn one of two crowns awarded during the Showcase."

About MicroGen Systems, Inc.:

MicroGen is bringing to market a suite of products based on its proprietary piezo-MEMS platform technology that is in production at X-FAB MEMS Foundry Itzehoe located north of Hamburg, Germany. This includes actuators, energy harvesters and sensors that can be designed and fabricated individually or as a system-onchip (SoC). MicroGen is an early stage start-up backed by strategic investment from semiconductor holding company Xtrion N.V., which is the majority owner of X-FAB. In addition, the New York State Energy Research Development Authority, The University of Vermont (UVM) Ventures Fund, Cornell University's Energy Materials Center, CenterState CEO and NY-based angels have provided funding. MicroGen located in Rochester, NY is a UVM spin-out that developed its core piezo-MEMS platform technology at Cornell University. MicroGen is led by a strong international management team, Board of Directors and Advisory Board with significant semiconductor and MEMS industry and start-up company experience. For more information, visit http://www. microgensystems.com. "BOLT" and "Thing Power" are in the process of being registered trademarks of MicroGen Systems. All other product and company names are trademarks or registered trademarks of their respective holders.

About MEMS Industry Group:

MEMS Industry Group (MIG) is the trade association advancing MEMS across global markets. More than 160 companies comprise MIG, including Analog Devices, Applied Materials, ARM, Bosch, Broadcom, Freescale Semiconductor, GE, Honeywell, HP, Infineon, Intel, InvenSense, Murata Electronics Oy, OMRON Electronic Components, Qualcomm, STMicroelectronics and Texas Instruments. For more information, visit http://www. memsindustrygroup.org.

November 4, 2013

Making Electric Energy Efficient at Bargain Prices

So Long, Kilowatt-Hours Lost. Hello, Energy Found.

Kevin Matocha, president of Monolith Semiconductor, captures a self-portrait in the mirror surface of a silicon carbide wafer at the Cornell NanoScale Facility.

To enhance efficiency in electric energy transfers from high-voltage grids to your home's toaster and television, the U.S. Department of Energy's ARPA-E program awarded a \$3.2 million grant in October to Monolith Semiconductor, an Ithaca-based startup company.

Monolith Semiconductor exclusively uses the Cornell NanoScale Science and Technology Facility (CNF) to make state-of-the-art silicon carbide metal-oxidesemiconductor field-effect transistor (MOSFET) switches, which can amplify or reduce large volumes of power during energy transfer. The MOSFET switches step down electricity from the grid to your home and office - with almost no energy loss.

This means, for example, that future electric cars plugged into your carport likely will be easier to charge, travel longer distances on cheaper electricity and become less expensive to manufacture, said Kevin Matocha, Monolith Semiconductor president.



By Blaine Friedlander, Cornell Chronicle

Monolith Semiconductor's silicon carbide wafers being loaded into a high temperature furnace at the CNF.

Within four years, the company hopes to sell performance silicon carbide at today's cheap silicon prices. "With the help of the CNF we're developing the manufacturing processes to dramatically reduce the cost of silicon carbide switches - which makes them accessible and ideal for renewable energy inverters, power supplies, industrial motor drives, electric vehicles and a smarter electrical grid," says Matocha.

ARPA-E, the Department of Energy's Advanced Research Projects Agency-Energy, awarded 14 projects nationwide to develop next-generation power conversion devices. The agency believes these projects will influence how the electrical energy of tomorrow is controlled on the grid.

"To transform America's energy infrastructure, we will need innovative technology options that can radically improve how we convert and use energy," said Cheryl Martin, deputy director of ARPA-E. "[These projects] could result in some of the critical components needed to update our aging infrastructure and reduce power losses from the grid."

Monolith Semiconductor started using the NSF-supported CNF in December 2012 and the company created its first prototypes in May 2013. Matocha said his company uses CNF for its etchers, furnaces, and deposition and photolithography tools.

"The main reason we are working at the CNF is their collection of tools and - equally important - the wellqualified engineering staff that keeps the tools up and running and available for use," Matocha said.

cnf in the news

New Micro Water Sensor Can Aid Growers

Crop growers, wine grape and other fruit growers, food processors and even concrete makers all benefit from water sensors for accurate, steady and numerous moisture readings. But current sensors are large, may cost thousands of dollars and often must be read manually.

Now, Cornell researchers have developed a microfluidic water sensor within a fingertip-sized silicon chip that is a hundred times more sensitive than current devices. The researchers are now completing soil tests and will soon test their design in plants, embedding their "lab on a chip" in the stems of grape vines, for example. They hope to mass produce the sensors for as little as \$5 each.

In soil or when inserted into a plant stem, the chip is fitted with wires that can be hooked up to a card for wireless data transmission or is compatible with existing dataloggers. Chips may be left in place for years, though they may break in freezing temperatures. Such inexpensive and accurate sensors can be strategically spaced in plants and soil for accurate measurements in agricultural fields.

For example, sophisticated vintners use precise irrigation to put regulated water stress on grapevines to create just the right grape composition for a premium cabernet or a chardonnay wine. While growers can use the sensors to monitor water in soils for their crops, civil engineers can embed these chips in concrete to determine optimal moisture levels as the concrete cures.

"One of our goals is to try and develop something that is not only a great improvement, but also much cheaper for growers and others to use," said Alan Lakso, professor of horticulture, who has been working on water sensing for 20 years.

The sensors make use of microfluidic technology – developed by Abraham Stroock, associate professor of chemical and biomolecular engineering – that places a tiny cavity inside the chip. The cavity is filled with water, and then the chip may be inserted in a plant stem or in the soil where it, through a nanoporous membrane, exchanges moisture with its environment and maintains an equilibrium pressure that the chip measures. By Krishna Ramanujan, Cornell Chronicle October 10, 2013



Vinay Pagay holds a "lab on a chip" that measures moisture levels in soil and can be embedded in plant stems for accurate information on water stress. The researchers hope to mass produce the chips for as little as \$5 each.

Using chips embedded in plants or spaced across soil and linked wirelessly to computers, for example, growers may "control the precise moisture of blocks of land, based on target goals," said Vinay Pagay, who helped develop the chip as a doctoral student in Lakso's lab, using the resources found at the Cornell NanoScale Facility.

Ernest and Julio Gallo Winery and Welch's juice company have already expressed interest in the sensors. And Cornell civil engineer Ken Hover has started working with Pagay and Lakso on using the sensors in concrete.

The researchers seek to understand how values gathered from sensors inside a plant and in soils relate to plant growth and function, so that growers can translate sensor values and optimize management.

The Cornell Center for Technology Enterprise and Commercialization is handling the intellectual property rights and patents.



Tax-Free Business Areas Approved for START-UP NY

Companies that want to advance Cornell's academic mission while creating jobs in the region can apply to receive tax benefits and other support under a new initiative to revitalize the state's economy, called START-UP NY, which stands for "SUNY Tax-Free Areas to Revitalize and Transform Upstate New York."

An economic development program announced by Gov. Andrew Cuomo last year, START-UP NY encourages collaborations between universities and businesses by allowing businesses to establish themselves for ten years in university-designated tax-free zones, as long as the business aligns with an aspect of the university's academic mission. Cornell is joining 64 SUNY campuses across the state in the effort.

The state recently accepted Cornell's application for a campus plan outlining its tax-free zones. Cornell can now begin accepting applicants to become business development partners with the university in these zones.

"As Tompkins County's largest employer, as well as the largest research university in upstate, Cornell is committed to the economic vitality of the state and region," said Mary Opperman, vice president for human resources and safety services, whose office is overseeing Cornell's START-UP NY program. "This is a unique opportunity for Cornell to leverage its strengths in innovation and education to help companies start and grow in the region and to create job opportunities."

Companies eligible to partner with Cornell through START-UP NY could, for example, come from research or student initiatives in the region, or from the university's many incubator programs, including the recently launched By Anne Ju, Cornell Chronicle March 17, 2014

Downtown Ithaca Incubator, according to Caitlin Schickel, regional economic development specialist working on Cornell's START-UP NY campus plan.

Others might include companies relocating to New York or expansions of existing companies generated by their connections to Cornell research and academics.

Businesses selected for the Cornell START-UP NY program must prove links to Cornell's academic mission. For example, they could commercialize research that originated in Cornell labs or establish experiential learning connections between employers and students, like internships and co-ops. They could also collaborate with Cornell's shared research facilities programs including: the Cornell NanoScale Science and Technology Facility, the Animal Health Diagnostic Center, the Cornell Institute for Social and Economic Research, the Cornell High Energy Synchrotron Source, and the Food Venture Center in Geneva.

Applicants will need to demonstrate academic and research benefits to Cornell and prove economic benefits to the community and state, including how many jobs will result and whether they will serve economically distressed regions.

The Cornell START-UP NY campus plan underwent a rigorous public process that involved collaboration with local governments and development councils and a 30-day public comment period.

Tax-free areas under Cornell's agreement are located in the town of Dryden, town of Harford, village of Lansing, city of Ithaca and city of Geneva.

Nanoscale Phonon Spectrometer Studies Phonon Transport in Nanostructures

In a new paper published in Nano Letters and highlighted in Physics Today, the Robinson Group at Cornell University has developed a spectrally precise microscale phonon spectrometer utilizing superconducting tunnel junctions (STJ) fabricated at the CNF. The spectral resolution of their spectrometer is ~10 times better than thermal conductance measurements, and this allows them to explore new heat transfer science.

The Robinson group used this tool to study phonon surface scattering by fabricating silicon nanosheets and determining their surface roughness using atomic force microscopy (AFM).

Then they sent phonons through the nanosheets and measured the phonon transmission rates with their spectrometer. By comparing the measured transmission rates to rates predicted by a Monte Carlo simulation of phonon trajectories they were able to assess the validity of a five-decades-old theory called the Casimir-Ziman theory. Until this point this theory hadn't been rigorously tested.

One of the grand challenges of nanoscience is to develop the experimental tools to understand the basic mechanisms of heat flow at nanoscale lengths. Phonons, a fundamental unit of heat, are the major heat carriers in semiconductors and insulators. Research on phonon thermal transport in nanostructures has generated great excitement recently due to the potential impact on thermoelectric materials and microelectronic cooling. It is generally believed that surface scattering is the primary is the dominant scattering mechanism of phonons in nanostructures, which can influence the ability for heat flow through the structure. However, precise experimental techniques for probing phonon surface interactions - which depend on the surface roughness and the phonon wavelength - are lacking. The fundamental science of heat flow is as not as well understood in nanostructures as it is in bulk materials.

To study this, the Robinson group developed a microscale phonon spectrometer to precisely assess phonon scattering rates in silicon nanosheets. Al-Al_Oy-Al superconducting tunnel junctions are utilized for emission and detection of phonons with frequency ~ 100 GHz to ~ 870 GHz. The emitters and detectors were deposited on sidewalls of etched mesas (7 μ m wide and ~ 0.8 μ m high) using double angle evaporation. Electron-beam lithography and unpulsed Bosch etching was used to pattern and etch the nanosheets - 0.6-0.8 µm high and 0.12-0.38 µm wide - into the mesas. Such geometry allows for lineof-sight propagation of phonons from the emitters, through nanosheets, and to detectors. While transiting the nanosheets, phonons interact with the surface and

Totally Diffusive signal Scattering SQUID Detector leasured phonon **Ziman Theory** Predictions Nanosheets Source STJ Mesa 3 µm Simulated nanosheet transmission

> undergo partial scattering and transmission depending on the surface roughness.

> The Robinson group measured the phonon transmission rates with the spectrometer and compared measured transmission rates to rates predicted by a Monte Carlo simulation of phonon trajectories, with work done in collaboration with CNF's Derek Stewart. They were able to assess the well-known Casimir limit for totally diffusive phonon scattering using Ziman theory. The Casimir-Ziman theory determines the probability of specular or diffusive surface scattering by the phonons based on surface roughness and the phonon's wavelength. A perfectly smooth surface will specularly reflect phonons, resulting in a 0% probability for diffusive scattering, and a perfectly rough surface will have a 100% probability of diffuse scattering. A real surface will have a scattering probability between these two values (0-100%) and will depend on the surface roughness and the phonon wavelength.

> The Robinson group carefully determined their surface roughness to be 1 nm, and then used the Ziman theory to find a prediction of diffusive surface scattering probability of $\sim 40\%$. However, their measurements and simulations showed that scattering occurred at 100% probability. This means that the totally diffusive scattering occurs at much lower frequencies than had been previously believed. Since diffusive scattering effectively lowers phonon transmission, this excessive phonon scattering rates have implications for thermal conductivity in nanostructures: the actual thermal conductance will be much lower than predicted using the standard Casimir-Ziman theory.

> With this spectrally resolved measurement of phonon transport, the understanding of phonon propagation and scattering in nanoscale structures will be greatly improved; hence, the exploitation and engineering of phonons for thermoelectric devices, microelectronic coolers, and phononic devices should become more feasible.

> > continued on page 11



The Robinson Group, continued

The success of this project was only possible due to access to the Cornell NanoScale Facility. The CNF's worldclass fabrication facilities and support staff help to over a number of technical challenges to make the nanoscale phonon spectrometers, such as placing tunnel junctions on a mesa and etching vertical sidewalls for nanosheets.

J.B. Hertzberg*, M. Aksit*, O.O. Otelaja*, D.A. Stewart, and R.D. Robinson, "Direct Measurements of Surface Scattering in Si Nanosheets using a Microscale Phonon Spectrometer: Implications for Casimir-Limit Predicted by Ziman Theory," Nano Letters 14, 403 (2014) (*equal author contribution) http://dx.doi.org/10.1021/nl402701a

Featured in Physics Today, print magazine, February 2014 issue: http:// scitation.aip.org/content/aip/magazine/physicstoday/article/67/2/10.1063/ PT.3.2262 Featured in Physics Today Online: http://scitation.aip.org/content/aip/magazine/physicstoday/news/10.1063/PT.5.7037 Cornell Chronicle article: http://www.news.cornell.edu/stories/2014/02/tiny-tool-measuresheat-nanoscale

Harmful Algal Blooms Project

Thanks for your interest in our Harmful Algal Blooms project. It is a new project for us, and we are very excited about it. We are in the construction stage of our device we don't have a device in hand yet. We want to mention that we do have a model bacteria strains from Pasteur Institute in Paris (it gives florescent light under microscope).

The first generation of the device will have less numbers of micro-habitats. It is about 320 of micro-habitats on one chip. *Below* are some pictures of PCC7806 cells in our current microfluidic device with 400-µm width and 167-µm depth. The cells are fluorescent under Cy5 (red filter).

Reported by: B.J. Kim (senior research associate), Chih-Kuan Tung (postdoc), Prof. Mingming Wu, Associate Professor, The Biological and Environmental Engineering Department, Cornell University.



Cornell Among Recipients of State Awards for Energy Storage Tech

Cornell University is among the six recipients getting a total of \$1.4 million from New York state to create new technologies in battery and energy storage that could one day harden the state's electric grid and diversify transportation fuels.

"Investing in New York's cleantech economy will revolutionize the way we store and transfer energy while creating jobs and supporting our state's clean energy businesses," Governor Andrew M. Cuomo said in a statement. "This funding will help to create new opportunities for manufacturers and researchers around the state to commercialize their products, help the environment by reducing energy use, and ultimately continue to grow our state's green economy."

The majority of recipients were awarded \$250,000. Cornell will work to develop and demonstrate a regenerative fuel cell energy storage system, using a membrane designed

Ithaca Journal Staff Report March 5, 2014

at the university to produce hydrogen. The project could reduce the cost of hydrogen production and pave the way for hydrogen-powered vehicles. Widetronix will work with the Cornell NanoScale Facility to enhance the power density of the Widetronix betavoltaic platform.

Other recipients include Custom Electronics in the Mohawk Valley working with Binghamton University; Columbia University seeks to scale-up electrochemical reactor technology developed at the school; Rensselaer Polytechnic Institute in Troy working with Finch Paper of Glens Falls and JNC of Rye; Con Edison and the Battery and Energy Storage Testing and Commercialization Center of Rochester working with Ambri Inc.

Funding is provided through the New York State Energy Research and Development Authority and New York Battery and Energy Storage Technology Consortium Bench-to-Prototype solicitation.



The 2013 NNIN REU Interns at the network-wide convocation in August, held at the Georgia Institute of Technology. Photograph by James Griffin.

2014 Nanotechnology Network REU Program

2014 NNIN iREU Program

Even with the unknown resolution of our proposal to the National Science Foundation for network funding, 741 undergraduate students took a chance and started an application for the 2014 Nanotechnology Network Research Experience for Undergraduates (N-REU) Program! And NSF funding was secured in time to hire 58 N-REU interns.

The interns have been assigned to a specific research project at one of our eleven participating sites, and over the ten-week summer program, they will make a meaningful research contribution to their research group.

Six students will be here at the CNF for the summer, and we look forward to getting to know them and introducing them to our nano-world!

Find out more about the program at http://www.nnin.org/reu

Once the NSF funding was secured for the summer, we also hired eleven of the 2013 NNIN REU interns to join us this summer as part of our International (iREU) Program.

Two interns will be traveling to the Centre Microélectronique de Provence, Ecole Nationale Supérieure des Mines de Saint Etienne, France, and nine interns will be traveling to the National Institute for Materials Science (NIMS), Tsukuba, Ibaraki, Japan.

Our own 2013 CNF REUs, Gabriel Lopez Marcial and Connie Wu, are part of the NIMS Program. Read more about Connie and Gabriel in the CNF Community section.

cnf events





CNF Junior FIRST° LEGO° League

Our Expo on Saturday, January 25th was a huge success! Twenty-four teams and 126 youth — from the central NY area displayed their work on this season's challenge: *Disaster Blaster*. Many thanks to the Ithaca Sciencenter and the Red Cross of Central NY for providing such fun activities for everyone! We also appreciate the hard work of the student volunteers from the Cornell Chapter of the Society of Women Engineers!

Please contact Dan Woodie to sign up for an email reminder for next season (daniel.woodie@cornell.edu).

There's always something new to learn, as Junior FIRST[®] LEGO[®] League teams will find out this season. In the 2014 Jr.FLL THINK TANKTM Challenge, over 26,000 children ages 6 to 9 from fourteen countries will take a closer look at where and how learning happens every day.

Get ready to dive into the Jr.FLL THINK TANK!



isaster P

cnf events



Japan's Nanofabrication Platform

On Tuesday, December 3, 2013, the CNF hosted a visit with Japan's Nanofabrication Platform, a visit organized by MEXT (Ministry of Education, Culture, Sports, Science and Technology in Japan). The Nanofabrication Platform started in 2012 and consists of sixteen Japanese universities and organizations, and Kyoto University is the lead organization. The mission to open Japan's facilities to researchers, much like the NNIN does here in the U.S.A. In December, MEXT visited several members of the NNIN to learn more about our operations and cooperations; Cornell University, University of Washington, Stanford University. and Harvard University. Here are the representatives during their visit to the CNF!

Want to learn more about how we can help with your nanotechnology Research & Development or commercialization plans?

Next fine you come to libece or Cornell — Errenes for a CNF Visit and Touri

Contact Ms. Melanie-Claire Mallison, mallison@cnf.cornell.edu

CNF User Wiki

We are pleased to announce CNF's own user wiki! The wiki provides a central location for up-to-date recipes, tool manuals, and tips that will help you reach your fabrication goals.

We strongly urge you to submit recipes of your own using the "contact" link. Put your trials and errors to good use, and pass on those hardearned tricks that might help someone else!

A mobile version of the wiki is available for cell phones and tablets. Users logged in with a CNF ID can also subscribe to receive emails when specific topics are updated.

Explore the wiki at http://wiki.cnfusers.cornell.edu/

Follow @CNFComputing on Twitter

"Digital Bits from the CNF IT World."

What's on the minds of the CNF IT Staff? What's trending in the CNF Computing Arena? Follow us @CNFComputing!

cnf events

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, graduate 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, but class size is limited.



Tuesday – Friday, June 3 – 6, 2014

> Register today!

Details and registration are now available online at: http://www.cnf.cornell.edu/cnf_tcn_june_2014.html



Cornell NanoScale Facility

:: 2014 Annual Meeting ::

Thursday, September 18 :: Presentations & Posters

CNF's 2013 Nellie Yeh-Poh Lin Whetten Memorial Award Winner: Carol Newby

Carol Newby is a graduate student in the Department of Materials Science and Engineering, and is the recipient of CNF's 2013 Nellie Yeh-Poh Lin Whetten Memorial Award.

Carol grew up in Devon, England, and completed her undergraduate studies in Materials Science and Metallurgy at the University of Cambridge.



The opportunity to spend her junior year on exchange at MIT exposed her to the benefits of conducting research in the USA and she decided to return for her doctorate. Before heading to graduate school, she spent a year at Consiglio Nazionale delle Ricerche (CNR) Bologna, Italy, gaining experience as a research assistant in a lab studying organic spintronics. Carol joined Cornell MSE in 2008 and began work in the Ober Group on developing patterning processes for organic electronic materials.

Organic materials cannot be processed with conventional methods that use organic solvents as these would damage or remove the organic material. The Ober Group therefore developed an approach they called *orthogonal processing* that uses fluorinated polymers and solvents to address this problem. Due to significant differences in intermolecular bonding, fluorinated materials are chemically orthogonal to organic materials. This means they don't interact with organics – fluorinated solvents are immiscible with organic solvents (as shown in the vial in Figure 1), and do not dissolve or damage organic materials. Carol was tasked with extending the concept of orthogonal processing into the realms of inkjet printing, imprint lithography and photolithography.

One of Carol's first projects was to conduct a study of the inkjet printing of fluorinated solutions using CNF's Dimatix DMP 2800 printer. Using a model system consisting of a fluorinated methacrylate polymer (PFMA, structure shown in Figure 1), dissolved in hydrofluoroether (HFE) she found that despite their unusual properties (low surface tension, high density) fluorinated solutions could still be deposited by inkjet printing. As inkjet printing is a low-cost, direct-write technique that is also compatible with roll-to-roll manufacture one plausible application of inkjet printed fluoropolymers is low-resolution (>50 µm) patterning of organic semiconductors. To demonstrate this Carol fabricated and tested arrays of bottom-contact P3HT transistors, shown in Figure 1, in which the P3HT organic semiconductor is patterned using inkjet printed fluoropolymer drops as an etch mask.



CNF NanoMeter V23N1

Fluorinated materials systems have also been shown to be biocompatible so orthogonal processing can be adapted to pattern biomolecules for applications such as biosensors. As well as avoiding the use of organic solvents, an ideal patterning process for biomolecules needs to also be conducted at room temperature and avoid the use of UV light. For this reason Carol helped develop a *nanoimprint* process that uses PFMA as a resist on CNF's Nanonex NX 2500. Patterns in PFMA resist on silicon are shown in Figure 2 with features ranging from 100 to 2 μ m. In collaboration with the Baird Group in Chemistry this process was used to demonstrate two-component protein patterns as shown in the fluorescence images in Figure 2.

As photolithography is the industry standard for highresolution, high-throughput patterning of inorganic materials it would be useful if this technique could be adapted to be compatible with organic materials without losing the benefits of using a highly-developed commercial photoresist. Carol did this by using PFMA as an inert barrier layer to protect light emitting polymers during patterning with conventional resists like Microsposit[™] 1805 as illustrated in Figure 3. This approach is very versatile and can be adapted to the required process allowing additive or subtractive patterning down to 1 µm resolutions. Carol demonstrated patterns of unprecedented complexity in



Figure 3

organic materials including two- and three-component patterns as shown in Figure 3.

Besides research, Carol was involved with various other endeavors on campus. She served as a Graduate Resident Fellow on West Campus for three years and was a TA for two MSE classes allowing her to get truly involved with undergraduate life and really appreciate the wonderful undergraduates Cornell attracts. She was also a CNF Fellow for nearly three years helping to develop standard processes for the Nanometer. Carol is exceedingly grateful to all those who helped her along her way at Cornell, especially the CNF staff, her advisor and the Ober Group.

The skills she learned at CNF will undoubtedly be helpful in her next chapter as a Process Engineer at Intel working on certain 14 nm devices.



Carol accepted her award at the 2013 CNF Annual Meeting; she is pictured here with Dan Ralph, CNF Lester B. Knight Director, and Don Tennant, CNF Director of Operations.

The CNF Nellie Yeh-Poh Lin Whetten Memorial Award

"This award is given in fond memory of Nellie Whetten, a CNF staff member from 1984 to 1987, who died March 24, 1989. This award recognizes outstanding young women in science and engineering whose research was conducted in the CNF, and whose work and professional lives exemplify Nellie's commitment to scientific excellence, interdisciplinary collaboration, professional and personal courtesy, and exuberance for life."

(From the Whetten Award plaque in the CNF main office, which lists all the Whetten Award winners since 1978.)

cnf community news

Robert Austin, a Princeton University professor of physics, was awarded the 2014 Max Delbruck Prize in Biological Physics from the American **Physical** Society (APS) in recognition of work that has "uncovered both new physics and revolutionized the laboratory practice of biology."



The APS cited Austin's work related to the use of nanotechnology and microfabrication as having "wideranging contributions to biological physics encompassing all scales from the molecular to that of organismic populations." The award is presented biennially and includes a \$10,000 prize.

Bob has been a valued member of the CNF Community since 1991 and most recently was our keynote speaker at the 2013 CNF Annual Meeting.



From the SPIE Conference in San Jose: Conference chair Thomas Wallow, at left, presents **Jing Jiang** with the 2013 Jeffrey Byers Memorial Award for Best Poster, sponsored by Tokyo Electron, for "Line edge roughness of highdeprotection activation energy photoresist by using submillisecond post-exposure bake," authored by Jing Jiang, Byungki Jung, Michael Thompson, and Christopher Ober, Cornell University [8682-58] (CNF Project # 2064-11).



At the 2013 Annual Biomedical Research Conference for Minority Students (ABRCMS), **Gabriel López Marcial** won one of the poster awards in his discipline by presenting his 2013 NNIN REU @ CNF research on "Design of a Microfluidic Device to Measure the Deformability of Cancer Cells." Gabriel, who attends the University of Puerto Rico at Mayaguez, spent his summer of 2013 working with Jan Lammerding and Patricia Davidson.

This coming summer, Gabriel will be in Japan as part of our NNIN International REU (iREU) Program, working at NIMS with Dr. Takao Mori on the "Development of Viable Thermoelectric Materials through Atomic Structure Level and Grain-Size Level Control."





Po-Cheng Chen (online)

Po-Cheng Chen won one of the two Outstanding Paper Awards at the 2014 IEEE MEMS Conference. Po-Cheng presented research on "A Silicon Electro-Mechano Tissue Assay Surgical Tweezer"; a project worked on in part by **Connie Wu** (University of Pennsylvania), a 2013 NNIN REU @ CNF, for which Po-Cheng was a mentor. Connie is shown at right presenting her research poster at the 2013 NNIN REU Convocation at Georgia Tech.

On the IEEE award, Connie commented, "Definitely grateful that you gave me this research opportunity or this would have never happened! Best Regards."

Connie is also joining our iREU Program in Japan, working with Prof. Hiroyuki Sakaki and Dr. Takeshi Noda, on "Transport Studies of Semiconductor Nanostructures and Devices Containing Self-Organized Quantum Dots and Wires."

All photographs on pages 18 and 19 provided by the person highlighted in the Community News piece, unless otherwise noted.

cnf community news



Lito de la Rama received the MRS Graduate Student Award (provided, 04/2013)

Lifo de la Rama received the MRS Graduate Student Silver Award in the 2013 MRS Spring Meeting (04/2013). This is an honor for his contribution to nanocalorimetry study of the melting of single and stacked silver alkanethiolate lamellar crystals. He fabricated the nanocalorimetry sensors for his research at CNF in 2009 and 2012. His work is summarized in his publication of L. P. de la Rama, L. H. Allen, et al., JACS, 135, 14286 (2013). He graduated in June 2013 and started his new job as a senior engineer at SanDisk Corporation in July 2013.

Reported by Lito P. de la Rama and his advisor, Prof. Leslie H. Allen. Department of Materials Science and Engineering, University of Illinois at Urbana-Champaign.

Gifts to Cornell NanoScale Facility

...help us to maintain the culture and capabilities of the CNF as an open user facility, dedicated to advancing all fields of nanoscience and engineering and providing hands-on education for the next generation of leaders in these fields. Whether made by an individual or a corporation, gifts both large and small can make a difference.

Here are some ways that your unrestricted gift will be used: Summer Research Internships for Undergraduates, the CNF Fellows Program, New Tools and Processes, Support for CNF Staff Members, Industrial Partnerships, and the CNF Annual Meeting.

> Find out more at http://www.cnf.cornell.edu/ cnf_gifts.html



Connie Wu

Photograph by Melanie-Claire Mallison

Remote Process Work Options at CNF

The CNF is primarily a hands-on facility, offering researchers access and training to use a comprehensive tool set with the support of an experienced technical staff. On a very limited basis though, CNF staff assist with small jobs on a best effort basis that involve just a few, routine process steps.

But did you know that in addition, several independent fabrication consultants offer their services to clients? Although not formally affiliated with the CNF, these contractors are trained CNF users, offer expertise in a variety of fabrication technologies, and provide their own liability insurance. While CNF cannot warranty the work of consultants, we are glad to assist in advertising fabrication opportunities to the community of consultants. We leave it to prospective employers to negotiate the terms of service directly with the applicants.

We do believe learning the ropes from our great staff is the best way to get the results you need, however, if traveling to CNF is just not practical, check out our listed consultants on our website at: http://www.cnf.cornell.edu/cnf_remotework.html

CorSolutions Fluidic Probe Station

The CNF has a Tool for Testing Microfluidic Devices!

The CorSolutions Fluidic Probe Station makes nonpermanant, leak-tight, low-dead volume connections to ports on PDMS, glass, silicon, plastics or other substrates. Computer-controlled pumps give users the ability to precisely control flow, even program complex flow patterns over minutes to days. A flow-controlled pump delivers flow rates in the nanoliter/minute range, and it can halt flow, a feat not possible with syringe pumps or traditional peristaltic pumps. It is also free of other drawbacks of syringe pumps including pulsation, slow response time, need to refill syringes, and backpressure variations.

A pressure-controlled pump is also available that delivers flows up to 30-40 PSI. The system can be set up under a stereoscope with a wide visual field or a fluorescence microscope, both of which have digital image capture systems. Particle tracking and measurement software allows users to calculate flow rates and to evaluate and document device performance. CNF users have used the station to test flow rates in fluidic prototypes. One group from the veterinary college controlled the flow rate precisely enough to demonstrate the rotation of individual cells by an electric field. More recently, a group from the Materials Science and Engineering Department used the pressure-controlled pump to demonstrate flow through a sub-micron thick channel of novel block co-polymers.

The Shuler group from Biomedical Engineering has also used the system to mimic pulsatile blood flow during the formation of blood vessels in artificial channels.

The station is useful to any fluidics researchers that would like to characterize or functionalize channels.

Contact Beth Rhoades for more information. She is happy to demonstrate the tool using your device. (rhoades@cnf. cornell.edu)



Figure 1: Fluidic probes attached at the inlet and outlet ports of an acrylic fluidic device. The arm are screwed firmly to the table and make compression connections that are plumbed with tubing to a flow-controlled pump (not shown).



Figure 2: Flow controlled pumps have software for programming fluid delivery. A flow rate of 0 nL/min can also be programmed, allowing the user to stop the flow when desired. This is not possible with syringe pumps or traditional peristaltic pumps.



cnf tools & capabilities

Oxford PlasmaPro 100 Cobra ICP Etch System

CNF is pleased to announce the addition of a new PlasmaPro 100 Cobra ICP etch system from Oxford Instruments.

This inductively coupled plasma (ICP)-based reactive ion etch platform is configured for stateof-the-art nanoscale etching. It features a very large plasma generation area 380 mm in diameter combined with a 240 mm electrode diameter for highly uniform etching. The newly designed Cobra ICP is powered with a 3kW rf generator operating at 2 MHz enabling the generation

of plasma densities on the order of $1E+12/cm^3$ by virtue of more efficient ionization and dissociation. Such a high density of ions and radicals enables increased etch rates, while independent control of the electrode power allows for independent control of the ion energies and hence the selectivities to the masking materials and underlying layers. Other features of the system include a wide range temperature (-150°C \rightarrow 400°C) electrode, which will greatly enhance our spectrum of materials that can be etched with volatile chemistries. Mechanical clamping along with high pressure helium backside cooling will allow for additional temperature control for longer etches with resist masking. A low frequency (350 kHz) bias capability of the lower electrode will allow us to more effectively etch high aspect ratio features with minimum RIE-lag effects.

The system is equipped with a 12-line gas pod permitting a wide range of process gases and additives for maximum system versatility. The initial setup will include the following gases: HBr, Cl_2 , BCl_3 , CH_3OH , SF_6 , O_2 , H_2 , and Ar. Future additions may include NH_3 , CO, and C_4F_8 as process gases.

The system is constructed for corrosive halogen based gases and is equipped with a loadlock for sample entry and system isolation. The tool also has an Ocean Optics optical emission spectrometer (OES) that is fully integrated to the control system that will allow users to monitor process chemistry for critical etch termination, i.e., endpoint control, and selectivity to underlying materials.

One of the main process missions of the system will be nanoscale etching of silicon with hydrogen bromide (HBr)-based chemistry. The benefits of HBr etching of silicon have been known for many years and these include moderately fast etch rates with a highly anisotropic etch profile due to its ion enhanced etch mechanisms. HBr chemistry offers high selectivity to resist and the ability to etch high aspect ratio nanoscale features without many



of the artifacts that are present in chlorine based plasmas such as trenching and poor resist selectivity.

There are many differences between chlorine HBr and chemistries that induce differences in feature charging effects, selectivity, faceting of the resist, and composition of the sidewall passivation layers formed during etching. In addition, selectivity to HSQ as a masking layer and to the buried oxide layer (BOX) can exceed 50:1 and 100:1 respectively. These process attributes will

greatly enhance our capabilities to fabricate advanced silicon photonic, MEMS, and electronic devices.

The other principal process objective will be the etching of magnetic based materials. One of the most technologically important areas is the development of magnetic random access memory (MRAMs), which consist of a magnetic tunnel junction (MTJ) and CMOS.

One of the most challenging steps in MRAM fabrication is the etching of the MTJ stack. The stack typically contains a non-magnetic seed layer to promote proper crystalline growth (e.g., Ta), an antiferromagnet such as PtMn or IrMn, an antiferromagnetically exchange based pair of ferromagnets (e.g., CoFe/Ru/CoFe), the insulating tunnel barrier (e.g., Al₂O₃ or MgO), a switchable layer free layer (e.g., CoFeB/Ru/CoFeB), and a suitable hard mask such as TiN or TaN.

The problem is that magnetic materials have difficulty reacting with most chemically active plasma species to form volatile etch products, so users often have to resort to purely physical ion milling processes. However, ion milling suffers from low etch rates, low selectivity, undesirable sidewall redeposition especially for nanoscale features, and damage to the device structure itself. These magnetic materials can be etched in halogenated chemistry (i.e., Cl₂ or HBr), but often electrode temperatures must exceed 190°C to form volatile etch products.

Recently, an alternative process using methanol (CH₃OH) and argon has shown to be effective on Co, Fe, and Ni based alloys. Methanol, as the principal plasma reactant, can form volatile carbonyl compounds (e.g., Ni(CO)₄, Fe(CO)₅, and Co₂(CO)₈) at room temperature. The antiferromagnet IrMn also etches in a methanol plasma. In addition, the selectivity to common mask materials such as Al₂O₃, Ta, Ti, TaN, and TiN is quite high, while leaving no residue on the etched devices.

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cnf tools & capabilities

The Cobra's capabilities will extend to the cryogenic silicon etch used for deep reactive ion etching. The electrode's low temperature range will allow the process to take place at temperatures below -100°C. While the process has been used for MEMs applications for many years, it recently has proven to be an excellent option for nanoscale silicon etching. The process uses SF_c/O_2 at -110°C to etch silicon anisotropically due to the formation of an involatile silicon oxyfluoride on the sidewalls. The process is especially attractive for nanoscale etching since the passivation layer is thin and inorganic. Selectivity to resist masks, including e-beam resists, is greatly enhanced since the low temperature induces less chemical erosion. Furthermore, since the SF₆ and O₂ flow simultaneously, the sidewall profiles are smooth without the presence of scalloping which is characteristic of the Bosch process.

This feature makes fabricating nanoscale photonic structures very attractive.

An additional capability of the new Cobra ICP is the ability to etch nanoscale polymer features at cryogenic temperatures. Using oxygen based chemistry, anisotropic high aspect ratio features can be obtained at cryogenic temperatures due to reduced reactivity of the sidewalls, eliminating the need for a separate passivant additive gas.

Finally with the use of an SF₆ based chemistry, we plan to develop a silicon carbide etch process utilizing the wide temperature range available on the Cobra. With the growing interest in silicon carbide devices among the user community, this will be a nice addition to our increasing etch repertoire. For further information, please contact either Vince Genova or Meredith Metzler.



To supplement our Oxford FlexAl ALD system, we recently purchased a new Arradiance GEMStar ALD system. This compact table top ALD system is ideally suited for research applications in a multiuser facility. The GEMStar 6 can accommodate substrates up to 6 inches (150 mm), and with its unique 300°C hot wall chamber design can deposit uniform, conformal metal, semiconducting, and insulating ALD films on flat substrates, 3D surfaces including high aspect ratio features, 3D objects and powders. Up to eight precursors can be run simultaneously, producing multicomponent films and film stacks.

A very unique feature of the GEMStar system is the particle ALD coating option. It consists of a 2 μ m particle canister filter mounted on a variable speed (5-95 rpm) 360 degree continuous rotary driven feedthrough to enable conformal coating of the suspended particles. This system has demonstrated successful conformal Al₂O₃ coatings on 30-70 nm diameter CNTs as well as uniform TiO₂ ALD coatings of networked

Arradiance GEMStar ALD System

mesoporous polymeric and carbon films with pore sizes as small as 40 nm. The particle ALD feature will be an invaluable asset to those research groups wishing to conformally coat nanoscale sized media for a variety of applications.

Initially the GEMStar will be configured for platinum, titanium oxide, aluminum oxide, and silicon oxide ALD films. These thermal ALD films will complement our current dielectric film capabilities on the Oxford FlexAL system. XPS analyses of our initial sample evaluations of these four films are illustrated at right and indicate precise stoichiometric and high purity films.

The versatility of the GEMStar will permit us to add additional precursors in the future to quickly meet the changing demands of the CNF user community.

For further information on this system, please contact Vince Genova.



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The CNF has a 3D Printer that is Churning Out Devices

The new ObJet Pro 3D printer from Stratasys prints plastics similar to acrylic or polypropylene in five different colors. One material can resist temperatures up to 75°C, and the others start to soften around 45°C.

CNF users are currently printing prototypes and working devices in a matter of hours. The printer accepts .STL files from a wide variety of 3D CAD software; we provide Autodesk Inventor 2014. The printer slices the CAD files into 16- or 28-micron-tall layers, prints and cures each layer simultaneously. A freshly printed device can be handled immediately, and a gel support material that surrounds the edges and fills in overhangs can be removed by spraying with water or a dilute base.

We are currently characterizing minimum feature sizes (around 300 μ m) and solvent compatibility. While this is not small in our nanofab world, it is right for large fluidic molds. This may be a viable alternative for some SU-8 master molds with heights greater than 300 μ m, which are notoriously difficult. The printed devices can also be treated to cast PDMS or culture cells. Outgassing the printed material followed by parylene coating neutralizes and seals printed surfaces. Such devices can also be treated with FOTS in the MVD 1000 as an effective antistiction treatment.

The Shuler group is using the printer to quickly modify the design of previously machined jigs that hold fluidic cell culture devices. In a matter of days, the jigs underwent three design iterations that would have taken weeks at a machine shop. Staff have also printed tool parts with threaded screw holes or junction boxes. Another group is printing holders for fluidic chips that have one printed piece slide into another to control the injection of samples with glass micro-needles. They are taking advantage of the fact that large tongue-in-groove features and overhangs that would require multiple photolithography steps can be printed in a single run on the 3D printer.

Our new ObJet Pro 3D printer is so easy to use that in addition to the traditional tool training, we offer a printing service. Send the file (.STL format), fill out the online order form, and get your raw device within 1-4 days. We will provide suggestions on how to clean your devices with just soap and water. The printing service is open to existing CNF users or anyone who would like to become a new user. In the latter case, the person would be set-up as a remote user with a remote project that doesn't require access to the facility, so no safety orientation is required. The additional cost of the service is staff time that is generally less than an hour.

Take a look at our web pages for FAQ, the order form, and a gallery of printed devices (http://www.cnf.cornell.edu/ cnf_3dprinter.html).

What would you like to print? Contact the tool managers, Beth Rhoades or Daron Westly, with your ideas.



Figure 1: (a) Jigs with fluidic channels that form a 3-dimensional fluidic device when assembled. (b) A dye highlights the fluidic portion in the assembled one.



Figure 2: Electrical connection box with threaded screw holes for assembly. Other boxes could be printed in opaque material or painted to restrict the amount of light.



Figure 3: PDMS cast on tall fins of DurusWhite polymer. Side walls become more vertical as the height increases. There is always some degree of edge rounding as seen at the tops of the fins. This may be desirable to the biology user looking to replicate rounded channels. CAD can generate any shape.



Figure 4: Multi-part device with a tongue-in-groove feature to control the z-axis of the assembled device. These large features and overhangs would require multiple photolithography or machining steps, but they can be printed in a single run on the 3D printer (printed in acrylic-like VeroClear).



cnf tools & capabilities

Nanonex NX-2500

Nanonex NX-2500 Nanoimprint P-NIL process successfully applied to Si, SiO_2 and Si_3N_4 at the CNF

Nanoimprint lithography (NIL) has the advantage of high throughput with sub-10 nm resolution. NIL is included on the ITRS roadmap for 45 nm and below nodes for advanced electronic devices. In addition to electronics, NIL is a benefit to many applications including displays, nanophotonics, biotechnology, and MEMS.

The NX-2500 has both thermal imprint (T-NIL) and photocurable imprint (P-NIL) capabilities. The thermal imprint module can reach temperatures up to 300°C with rapid heating and cooling rates. The photocuring module uses a narrow band 200W UV lamp with automatic control. It has submicron overlay alignment accuracy and has the ability to handle irregular shaped and sized substrates up to 100 mm diameter.

The efforts of CNF Fellow Carol Newby, along with CNF research staff members Vince Genova and John Treichler, have resulted in the development of an established baselined P-NIL process.

The P-NIL process can be applied to many types of substrates, but has been demonstrated on silicon. The P-NIL process utilizes a bi-layer resist system in which the first resist layer (200 nm) is purely organic and the upper UV resist layer (90 nm) contains silicon. Following the replication of template features into the upper layer of resist, a very critical pattern transfer process must occur in the residual and transfer layers of resist. The amount of residual UV resist remaining is a function of the applied imprint pressure. The pattern transfer consists of a selective fluorocarbon etch chemistry for the residual layer and the use of oxygen plasma to clear the organic resist layer in the Oxford 80. These etches must preserve the critical dimensions (CD) defined by the imprint process. The patterned imprint resist is then used as a mask to transfer the pattern into the silicon using an established "photonics etch" of combined SF₆/C₄F₈ chemistry in the Unaxis 770 ICP system. Linewidths around 180 nm have been etched into 500 nm of silicon with perfect anisotropy and line edge resolution.

The recently established photocurable nanoimprint (P-NIL) process has been demonstrated with pattern transfer into silicon oxide and silicon nitride using an internally fabricated ASML DUV (248 nm) patterned quartz template. The P-NIL pattern transfer process again consists of two separate etches in the Oxford 80 as described above. These etches must preserve the critical dimensions (CD) defined by the imprint process. The imprint pattern is then successfully transferred into the underlying thermal silicon dioxide film using CHF_3/O_2 chemistry using the Oxford 80 conventional RIE system or the advanced ICP based Oxford 100 system with etch rates of 35 nm/min and 127 nm/min, and selectivities to resist of 1.3:1 and 5:1, respectively. This etch was successfully demonstrated with feature sizes as small as 200 nm.

Similarly, CHF_3/O_2 chemistry is used to pattern transfer into underlying LPCVD silicon nitride using both the Oxford 80 and the Oxford 100 ICP systems, with etch rates of 55 nm/min and 142 nm/min, respectively, and selectivities between 1.5-2.0:1. Again features down to 200 nm were successfully patterned as illustrated in the figures below. In future work, we plan to investigate how the new etch chemistries, i.e., CH_2F_2 and C_4F_6 , can be used for successful nanoscale pattern transfer of NIL defined features into silicon based dielectrics.

For further information on these processes, please contact John Treichler or Vince Genova of the CNF research staff.

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cnf tools & capabilities

Nanonex NX-2500 Figure Captions

Opposite Page:

Figure 1. SEM cross-section images of a 1 μ m feature; a) in the bilayer resist after imprinting, b) after descum and underlayer etches, and c) after transfer into silicon.

Figure 2. SEM cross-section images of sub-220 nm features transferred into Si.

Figures 3-5: Nitride Oxford 82 Etches (50/5 sccm CHF_3/O_2 , 150W, 55mT).

This Page:

Figures 6-7: Nitride Oxford 100 Etches (CHF₃/O₂ 52/2 sccm 2500W/50W 6 mtorr).

Nitride etch rate: 142 nm/min Resist etch rate: 94 nm/min Selectivity: 1.5:1

Figures 8-10: *Oxide Oxford* 82 *Etches* (50/2 sccm CHF $_{3}/O_{2}$, 240W, 40mT).

SiO₂ etch rate: 35 nm/min Resist etch rate: 28 nm/min Selectivity: 1.3:1

Figures 11-12: Oxide Oxford 100 Etches (CHF ₃/O₂ 52/2sccm, 2500W/15W, 5 mtorr).

SiO₂ etch rate: 127 nm/min Resist etch rate: 25 nm/min Selectivity: 5:1



IR-MEMS Inspection Tool



The IRise optical setup can be seen above. From: Moritex SCHOTT, catalogue-5-1-ir-inspection.pdf, page 2.

The IR-MEMS Inspection Tool, aka IRise, is a general all purpose semi-automatic inspection tool for front side, buried layers, and backside inspection. The machine can handle substrate sizes up to 200 mm in diameter. It is computer controlled and both software and hardware can be upgraded.

The IRise has two main magnification operational modes; low magnification that ranges from 0.75x to 4.5x and high magnification that ranges from 0.7x all the way to 40x. The field of view for those modes ranges for low magnification between 7.09 x 8.88 mm to 1.18 x 1.48 mm and for high magnification from $0.71 \times .89$ mm to 0.13×0.17 mm.

The IRise currently has three illumination modes; near infrared coaxial illumination, near infrared transmission and near infrared oblique illumination. Other illumination sources can be added in the future. These modes give the machine sub-micron resolution for a variety of applications. This tool supports a variety of materials including; Si-Si, Si-Glass, Si-Au, Si-GaGs, Si-GaP, Si-GaAsP, Si-LiNbO₃, Si-LiTaO₃, Si-Au-PZT, Si-Au-PLZT, and Si-polymer-Si.

The IRise can be utilized for void detection and quality of bonds in wafer bonding. In terms of wafer packaging it can be used to inspect interconnects in single layer or in multilevel designs. It also serves as a good chipping and cracking post-CMP and post-dicing for top and buried layers.

This machine is capable of processing substrates very quickly. Processing time is dominated by image magnification and the size of the substrate. Image stitching and file size are inversely related to those two factors. The IRise's broad optical zoom range makes it ideal for layer characterization pre-processing and post-processing. Image storage is available locally on the machine and on the local CNF network.

Contact Edward Camacho with any questions or to be trained on this tool.

Oxford PlasmaLab 100 System

Silicon Based Dielectric Etching: A Comparison of C_4F_6/O_2 and CH_2F_2/O_2 Chemistries in the Oxford PlasmaLab 100 System

As part of CNF's Cooperative Development Agreement with Oxford Instruments, a significant upgrade was recently completed on our existing PlasmaLab 100-380 ICP etch system. The most significant modification was the installation of a gas ring manifold in close proximity to the substrate electrode. The updated PLC allows one to direct the flow of polymer forming gases to either the chamber showerhead or the gas ring inlet. This is the first Oxford Instruments RIE/ICP system with the unique gas ring configuration. The degree of dissociation and ionization of polymer precursors used in dielectric etching will vary depending on the admission of flow through the showerhead or the gas ring. These fragmented ions and radicals will influence the etch characteristics and selectivity. Because advanced lithographic techniques such as deep UV (DUV) and electron beam lithography require thinner resists to obtain the ever shrinking nanoscale resolution, selectivity becomes paramount. Along with higher resist selectivity, comes the ability to etch to higher aspect ratios.

CNF recently performed a comparative study of advanced polymer precursors hexafluorobutadiene C_4F_6 and difluoromethane (CH₂F₂) applied to the etching of thermal silicon dioxide, LPCVD silicon nitride, and low stress LPCVD silicon nitride with gases directed either to the chamber showerhead or the gas ring. The dielectric films were lithographically patterned with an ASML 248 nm DUV step and repeat system using UV210 resist and AR3 organic bottom anti-reflective coating (BARC). The BARC was etched in the Oxford PlasmaLab 80 RIE system at low pressure (15 mtorr) using a selective Ar/O_2 chemistry in order to preserve critical dimensions (CD). Design of experiments (DOEs) were conducted based on a partial factorial L9 Taguchi matrix varying ICP source power, RIE electrode power, pressure, and oxygen O, flow rate or percentage. Metrics of the DOE included etch rate, selectivity, and profile anisotropy. The C_4F_6/O_2 results indicate that etch rates are consistently higher for SiO₂ and stoichiometric Si_3N_4 when C_4F_6 is directed to the showerhead, while the etch rate of low stress, i.e., silicon rich silicon nitride is higher when C_4F_6 is directed to the gas ring, as shown in Figures 1-3.

Resist selectivity for all three dielectric films is enhanced when C_4F_6 is directed to the gas ring, as illustrated in Figure 4 in the case of SiO₂.

The higher oxide and nitride etch rates via the showerhead and the higher selectivities of all three films via the gas ring suggest that the fluorocarbon layer formed with the gas ring has a lower F/C ratio and has a higher degree of cross-linking, yielding higher etch resistance. These results have been verified by XPS analyses of the polymers. The interaction of the blocking fluorocarbon layer with the three dielectric films is illustrated in Figure 5 in terms of the relative etch rates.



Figures 1 and 2: The etch rate of SiO_2 and Si_3N_4 as a function of RIE and ICP power respectively when C_4F_6 is directed to the gas ring and through the ICP source.



Figure 3, left: Etch rate of low stress Si_3N_4 as a function of RIE power when C_4F_6 is directed either through the gas ring or the ICP source. Figure 4, right: Selectivity of silicon dioxide to resist as a function of RIE power when C_4F_6 is directed to either the gas ring or the ICP source.



Figure 5, left: C_4F_6 etch rates of silicon oxide, silicon nitride, and low stress silicon nitride as a function of RIE power through the showerhead. Figure 8, right: Etch rates of silicon nitride when CH₂F₂ is directed to either the gas ring or the ICP showerhead.



Figure 6, left: Silicon dioxide etch profile using C_4F_0/O_2 through the gas ring. Figure 7, right: Silicon dioxide etch profile using C_4F_0/O_2 through the showerhead.



Figure 9, left: Oxide selectivity to resist and a function of electrode power with CH_2F_2 directed to the gas ring or the ICP source. Figure 10, right: Comparison of silicon nitride selectivity to resist as a function of O_2 flow with CH_2F_2 through the gas ring and ICP source.

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Figure 11, left: Comparison of low stress nitride selectivity to resist as a function of pressure with CH_2F_2 flowing through the gas ring or the ICP source. Figure 12, right: Etch rates of silicon dioxide, silicon nitride, and low stress silicon nitride in CH_2F_2 through the gas ring.



Oxford PlasmaLab 100 System

The relative etch rates in Figure 5 also suggest an inherent silicon dioxide to nitride selective etch using a C_4F_6 based chemistry. The etch profiles are somewhat less anisotropic with C_4F_6 admission through the gas ring, indicating a more aggressive polymer formation on the sidewalls as illustrated in the following SEM micrographs (Figures 6-7).

The results of the CH_2F_2/O_2 DOE are quite different. In this case, etch rates of all three dielectric films are higher when CH_2F_2 is directed through the gas ring, especially in the case of silicon nitride as shown in Figure 8.

The resist selectivity for the oxide and standard silicon nitride is substantially higher when CH_2F_2 is directed through the gas ring, but lower for that of the low stress silicon nitride as illustrated in Figures 9-11.

The etch rate results seem to suggest that the hydrofluorocarbon film (HFC) deposited by the CH_2F_2 via the showerhead is perhaps more dense and cross-linked than that admitted through the gas ring, while selectivity to resist is better accomplished through the gas ring. XPS results seem to indicate essentially equivalent F/C ratios in the respective polymers. The HFC blocking layer interaction with the dielectric films is in contrast to the FC film deposited by the C_4F_6 , again indicative of a compositionally different polymer film, with H_2 content perhaps playing a key role. The relative etch rates of the three films are shown in Figure 12, and indicate an inherent silicon nitride to oxide selectivity using CH_2F_2 based chemistry.

The resulting etch profiles for CH_2F_2 appear to be less sensitive over the studied process range than C_4F_6 , suggesting a wider process latitude. The profiles are highly anisotropic for all of the dielectric films studied for CH_2F_2 admission through either the showerhead or the gas ring, as illustrated in the following SEMs (Figures 13-15).

The addition of the gas ring inlet to the Oxford PlasmaLab 100 ICP has allowed us to achieve enhanced selectivity to sensitive deep UV resists. Future studies will extend the investigation to electron beam resists in the high aspect ratio etching of nanoscale features in dielectrics.

For additional information on these processes, please contact Vince Genova of the CNF research staff.



Figure 13, left: Silicon dioxide etch profile using CH_2F_2/O_2 through the showerhead. Figure 14, middle: Low stress silicon nitride etch profile using CH_2F_2/O_2 through the gas ring. Figure 15, right: Low stress silicon nitride etch profile using CH_2F_2/O_2 through the showerhead.

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