# NanoMeter

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The newsletter of the Cornell NanoScale Science & Technology Facility

Winter/Spring 2010 Volume 19, Issue Number 1

#### **Director's Column**

It is with great pleasure we bring you the 2010 Winter/ Spring edition of the *NanoMeter*. In this issue we have collected articles that highlight the leading edge science that is being performed by our CNF users. We also bring you up to date on new equipment acquisitions that are helping to keep the CNF at the leading edge among university nanofabrication laboratories. With our successful proposal to the NSF's Major Research Instrumentation program, we are upgrading our electron beam lithography suite. This will bring some higher resolution lithography capability to the lab. In addition, a one-time stimulus fund (ARRA) infusion of capital upgrades will bring a noticeable update to many of our *workhorse* tools.

Look for individual articles on many of these items in this issue. Our carbon nanotube/graphene furnace was delivered recently and is being installed and our ASML 248 nm stepper is now installed and ready for your use. So it is shaping up to be an amazing year for equipment renewal in the lab! You can also see in several articles, that our staff are busy developing new processes on our tools to make these technologies accessible to our CNF community. I hope you are inspired by their work and that these new capabilities create opportunities for advanced applications.

In addition to advancing the laboratory capabilities, CNF continues to be an engine for Educational Outreach and Professional Growth. We just completed hosting two workshops. One devoted to training NNIN professional staff in the area of "Social and Ethical Issues in Nanotechnology." The other entitled, "Synergies in NanoScale Research and Manufacturing." Hosting such events brings both broader impact awareness to our user community and makes connections among a wide range of interested parties in the Nanotech landscape.

As you know CNF Director, George Malliaras left CNF and Cornell last August, and since then the Office of the Vice Provost for Research has been conducting a search. They are well in to the process and hope to have a new Director announced in the next few months. My thanks go out to Sandip Tiwari, Director of the NNIN, and Daniel Ralph, Chair of the CNF Executive Committee, for meeting regularly with me to be sure that the interim period is managed smoothly.

Please note the dates for upcoming events on the back page and check our web site often for news updates. Please let us know how we can continue to improve CNF!

Don Tennant CNF Interim Director CNF Director of Operations



#### Stimulus Funds to Pay for Equipment at Nanoscale Facility

By Anne Ju Cornell Chronicle Oct. 27, 2009

The Cornell NanoScale Science & Technology Facility (CNF) has received \$1.38 million in federal stimulus funds to help with equipment upgrades. The American Recovery and Reinvestment Act (ARRA) has allocated \$10 million to the NNIN to spread over the 14 sites for various needs. CNF's portion is an add-on to its regular National Science Foundation grant of \$2.68 million per year.

"Replacing old equipment is always a real challenge, and it's not something that people get excited about," said Sandip Tiwari, director of NNIN and Cornell's Charles Mellowes Professor in Engineering.

The grant will cover:

- \$850,000 for an advanced high-resolution pattern generator;
- \$270,000 for a contact mask and bond aligner;
- \$44,000 for expanded range film thickness measure-ment system;
- \$85,000 for an ion implanter computer upgrade; and
- \$127,000 for an atomic force microscope.

Tiwari noted that while economic times have challenged every aspect of the university, CNF has done a good job living within its means. He added that CNF maintains \$74 million to \$100 million worth of equipment. Even if the estimated life span for each piece is 10 years, it is still a sizeable sum to replace each year. The center has reached out to industry partners in the past to help them supplement equipment at CNF. "The ARRA support will make things good for a longer period of time," Tiwari said. "It is just a perfect thing for use within NNIN."

To date, Cornell has received 121 ARRA awards, totaling more than \$99.6 million over two years.



#### A member of the

National Nanotechnology Infrastructure Network (www.nnin.org)

#### Supported by:

The National Science Foundation, New York State Office of Science, Technology & Academic Research, Cornell University, Industry & Our Users.

www.cnf.cornell.edu • information@cnf.cornell.edu



#### Social and Ethical Issues Orientation for NNIN: A Training Workshop

On January 22, 2010, the CNF hosted a one day training workshop on "Social and Ethical Issues (SEI) Orientation at the National Nanotechnology Infrastructure Network (NNIN) Sites." The 24 participants included representatives from all 14 NNIN sites. For this interdisciplinary workshop, participants covered several disciplines such as social sciences, science and engineering, management, communication, education and outreach, environmental health and safety, and also people involved with the user orientation programs at different NNIN nanofabrication laboratories.

The theme of the workshop was to integrate SEI training into existing user orientation programs, in an interactive way. NNIN Director Dr. Sandip Tiwari set the tone by talking about the importance of SEI in any science and technology. The NNIN-SEI Coordinator, Dr. Katherine A. McComas, provided a brief overview of current NNIN SEI activities. Next, participants introduced themselves and shared the current status of SEI orientation at their respective sites. To provide an example of interactive SEI communication, Dr. Debasmita Patra, the NNIN-SEI Postdoctoral associate, presented the SEI training module developed and implemented at CNF over the last one year. To demonstrate the effectiveness of the CNF SEI training, Dr. Patra presented the analyses of the evaluation forms filled out by new users after each SEI training session. Further, frequently asked questions were discussed, and workshop participants debated approaches and responses, with insights from their respective disciplinary backgrounds.

Dr. Domingo A. Ferrer, The University of Texas at Austin, informed the group on his latest findings on risks, regulations and environmental outcomes in nanomaterials at the workplace. Dr. Ethan Allen, University of Washington presented the work of Dr. Deborah Bassett (who was absent) on SEI speech code among scientists and engineers. Dr. Robert McGinn, Stanford University, presented his ethics guide for laboratory users, designed on the basis of an empirical study conducted among 1037 NNIN lab users.

Overall, the active involvement and enthusiasm of all participants, in addition to the workshop evaluations, indicate that the participants liked this kind of workshop and encouraged such workshops in the future too.

For further information about the SEI activities at NNIN, please visit our website: www.sei.nnin.org or contact Dr. Debasmita Patra, patra@cnf.cornell.edu.



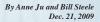
As part of its NNIN research outreach activities, CNF hosted a two day workshop, "Synergies in NanoScale Manufacturing and Research," held on the Cornell University campus, January 27-29, 2010. This was a byinvitation-only working group intended to generate active discussion in the issues related to bringing emergent tools, processes, and materials into commercialization.

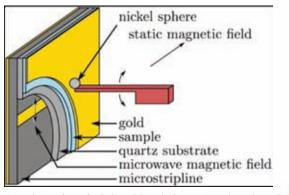
The speakers, moderators, and attendees — selected from across the country and from industry, academia, and government labs — brought a broad range of expertise and experience to the group discussions. The workshop was sponsored jointly by the NSF's National Nanomanufacturing Network (centered at the University of Massachusetts Amherst), the Cornell NanoScale Science and Technology Facility, and the National Nanotechnology Infrastructure Network. The effort was led by Don Tennant, Sandip Tiwari, and Lynn Rathbun from Cornell, and Mark Tuominen and Jeff Morse from UMass.

Topics ranged from roll to roll production of flexible electronics, mass methods of producing bit patterned magnetic media, ways to manufacture in silicon with atomic precision, and groundbreaking methods of making measurements of structural properties in complex materials. We also heard reports on several new means of highly specialized drug delivery and the possibility of mass production of carbon substrates for electronics, new classes of photochemicals, and more.

The program committee will be presenting a summary of the in depth discussions to the NSF.

#### **Researchers are on the Path to Creating Nano-MRI Images**





A schematic of John Marohn's scanned-probe electron spin resonance experiments, with the oscillating cantilever in the middle and the nickel tip that was attached by hand.

#### In biochemistry, shape is everything.

Because of the shape of their binding sites, hormones can attach to cell membranes to send signals inside, and viruses can open up paths to invade. But understanding the structure of complex molecules is no easy task. Existing microscopes, even the world's best electron microscopes, can't resolve atomic details of organic material.

Researchers at Cornell are changing that: They are devising methods to detect the magnetic fields of individual electrons and atomic nuclei, which they hope to use to make 3-D images — a nanoscale version of MRI. Their new approach can detect a tiny force called electron spin to a sensitivity scale of about 400 electrons. Their breakthrough is reported in the December 14<sup>th</sup> online issue of Proceedings of the National Academy of Sciences.

"There is no more longstanding a problem than imaging the structure of membrane proteins," said John Marohn, associate professor of chemistry, who is leading the research and has been working on the problem for nearly a decade. "There is no general approach, and it is backbreaking work." Marohn belongs to a growing community of scientists doing single-molecule imaging. At the heart of his research is the idea that electrons, protons and neutrons create a magnetic field, also called a magnetic moment. When placed next to a stronger magnetic field, all the little "magnets" - electrons or neutrons and protons in the nuclei - line up. Applying a RF field tuned to the electron or nucleus causes the magnetic moment to spin off axis like a wobbly top, a behavior called precession. A medical MRI machine detects voltage induced in a coil by the precession of, typically, a thousand billion hydrogen atoms, which creates enough resolution for 3-D imaging of body parts. Marohn and colleagues are trying to make an instrument sensitive enough to feel the force of a single electron or nucleus in a protein.

In their paper, they describe their new approach, which involves detecting the spin of electrons in a molecule called a nitroxide — rare because it contains a radical, or an unpaired electron. Their technique opens up the possibility of imaging, at sub-nanometer resolution, the locations of nitroxide "labels" attached to individual protein molecules. By creating a sample of nitroxide molecules dissolved in a thin-film polymer and bringing the sample close to a 4  $\mu$ m nickel magnet attached to a 350 nm silicon cantilever, they can detect the electron spin by measuring the frequency of the cantilever as it wobbles, like a diving board. The cantilever is similar to those used in scanning probe microscopy, a type of imaging that involves a cantilever scanning a surface and recording the probe-surface interactions.

To improve their frequency readouts and get more accurate measurements, the group must learn, among others things, how to make their magnets smaller, Marohn said. "The dream is to take that signal, run it through some sort of complicated algorithm, and get a picture," Marohn said.

The paper's first author is graduate student Eric Moore. The research was funded by the National Institutes of Health and the Army Research Office's Multi-University Research Initiative, and the work was performed in part at the CNF.



by Bill Steele Cornell Chronicle



Cornell Nanophotonics Group

SEM of two thin, flat rings of silicon nitride, each 190 nm thick and mounted a millionth of a meter apart. Light is fed into the ring resonators from the straight waveguide at the right. Under the right conditions, optical forces between the two rings are enough to bend the thin spokes and pull the rings toward one another, changing their resonances enough to act as an optical switch.

With a bit of leverage, Cornell researchers have used a very tiny beam of light with as little as 1 milliwatt of power to move a silicon structure up to 12 nanometers. That's enough to completely switch the optical properties of the structure from opaque to transparent, they reported.

The technology could have applications in the design of micro-electromechanical systems (MEMS) and micro-

optomechanical systems (MOMS) which combine moving parts with photonic circuits, said Michal Lipson, associate professor of electrical and computer engineering.

The research — by postdoctoral researcher Gustavo Wiederhecker, Long Chen, Ph.D. '09, Alexander Gondarenko, Ph.D. '10, and Lipson — appears in the online edition of the journal *Nature* and is forthcoming in print.

Light can be thought of as a stream of particles that can exert a force on whatever they strike. The sun doesn't knock you off your feet because the force is very small, but at the nanoscale, it can be significant. "The challenge is that large optical forces are required to change the geometry of photonic structures," Lipson explained.

But the researchers were able to reduce the force required by creating two ring resonators — circular waveguides whose circumference is matched to a multiple of the wavelength of the light used — and exploiting the coupling between beams of light traveling through the two rings.

A beam of light consists of oscillating electric and magnetic fields, and these fields can pull in nearby objects, a microscopic equivalent of the way static electricity on clothes attracts lint. This phenomenon is exploited in "optical tweezers" used by physicists to trap tiny objects. The forces tend to pull anything at the edge of the beam toward the center. When light travels through a waveguide whose cross-section is smaller than its wavelength, some of the light spills over, and with it, the attractive force. So parallel waveguides close together, each carrying a light beam, are drawn even closer, rather like two streams of rainwater on a windowpane that touch and are pulled together by surface tension.

The researchers created a structure consisting of two thin, flat silicon nitride rings about 30  $\mu$ m in diameter mounted one above the other and connected to a pedestal by thin spokes. Think of two bicycle wheels on a vertical shaft, but each with only four thin, flexible spokes. The ring waveguides are 3  $\mu$ m wide and 190 nm thick, and the rings are spaced 1  $\mu$ m apart. When light at a resonant frequency of the rings, in this case infrared light at 1533.5 nm, is fed into the rings, the force between the rings is enough to deform the rings by up to 12 nm, which the researchers showed was enough to change other resonances and switch other light beams traveling through the rings on and off. When light in both rings is in phase — the peaks and valleys of the wave match — the two rings are pulled together. When it is out of phase, they are repelled. The latter phenomenon might be useful in MEMS, where an ongoing problem is that silicon parts tend to stick together, Lipson said.

An application in photonic circuits might be to create a tunable filter to pass one particular optical wavelength, Wiederhecker suggested. The work is supported by the National Science Foundation (NSF) and the Cornell Center for Nanoscale Systems. Devices were fabricated at the Cornell NanoScale Science and Technology Facility, also supported by NSF.

(Find the full research report on pages 206-207 of the 2008-2009 CNF Research Accomplishments, http://www.cnf.cornell.edu/cnf\_2009ra.html)

#### Watching Crystals Grow May Lead to Faster Electronic Devices:

#### Research could improve manufacture of defect-free, thin films needed to make semiconductors

News From the Field NSF Press Release 10-012 Copyright 2010 AAAS

Conventional theory says when films are being formed at the atomic scale, atoms land on top of each other and form mounds or "islands" and feel an energetic "pull" from other atoms that prevents them from hopping off the island's edges and crystallizing into smooth sheets. The result is rough spots on the thin films used to produce semiconductors. Cornell University-led researchers eliminated this pull by shortening the bonds between their particles. But they still saw particles hesitate at the island's edges.

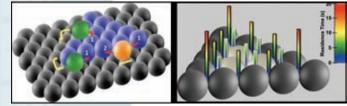


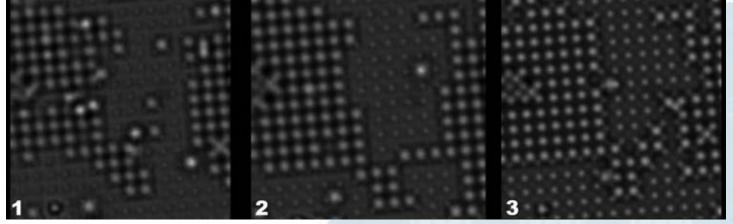
Figure 1

On the left side of Figure 1, green particles are the ones that encounter a step edge or corner barrier. The orange particle encounters smaller barriers as it moves from site to site. The #1 indicates the bond being broken. The #2 indicates the bond that is forming. Near a step edge or corner the atoms do not have a new neighbor to form a bond with (so no #2 particle). This is what sets up the barrier.

Using a solution of tiny plastic spheres 50 times smaller than a human hair, scientists at Cornell University discovered the thin, smooth crystalline sheets needed to make semiconductors can be grown more smoothly by managing the random darting motions of the atomic particles that affect how the crystals grow.

Researchers reproduced the conditions that lead to crystallization on the atomic scale by using particles much bigger than atoms, but still small enough that they behave like atoms to watch how particles crystallize. Additionally, with special laser beams known as "optical tweezers," researchers placed an individual particle (atom) on top of a growing crystal island and determined how easy it was for the particle to hop off that island. They found the random darting motions of a particle are a key factor that determines how long it spends on the island. When particles can hop off islands more easily, smooth crystals can be grown.

Video Images 1, 2 and 3, show a colloidal crystal freezing onto a square lattice template. (Full video can be found at http:// www.nsf.gov/news/news\_images.jsp?cntn\_id=116259 &org=NSF) The time an atom spends on top of a mound or "island"



Video Images 1, 2 and 3

often determines whether a rough spot will form during a thin film's crystallization process. Rough spots, bumps and defects are a serious problem for thin-film manufacturing.

The right side of Figure 1 shows how long on average a particle remains in the same location. The bars correspond to how long (on average) a particle placed on top of this triangular island resided at each lattice location.

The researchers' findings appear in the January 22, 2010, online issue of the journal Science.

All Images: Rajesh Ganapathy, Sharon Gerbode, Mark Buckley, and Itai Cohen. Video: John Savage also.

#### **Fabrics that Fight Germs and Detect Explosives go to Market**

By Sheri Hall Cornell Chronicle September 21, 2009

Fabrics with embedded nanoparticles to detect counterfeiting devices, explosives and dangerous chemicals or to serve as antibacterials for hospitals, law enforcement or the hospitality industry are just a few of the products that a new company, launched by two Cornell researchers, will produce. iFyber LLC, begun in fall 2008, uses technology developed through a cross-campus collaboration by fiber science professor Juan Hinestroza and Aaron Strickland, a research associate in the Department of Food Science. The company, which will commercialize this research, was launched and funded by KensaGroup LLC in collaboration with the Cornell Center for Technology Enterprise and Commercialization.

The key to iFyber's technology is the ability to deposit nanocoatings on natural and synthetic fibers with nanoscale precision, Hinestroza explained.

"We're using a chemical process to uniformly deposit nanoscopic particles onto the surface of a fabric," he said. "These particles can change the properties of the fabric."

Among the custom properties of the treated fibers are simultaneous water and oil resistance, antimicrobial behavior and electrical conductivity. The company's proprietary coating process allows the nanoparticles to adhere to curved fiber surfaces and crevices with a uniform distribution of particles using traditional textile processing equipment.

"There is significant potential to use this technology in a

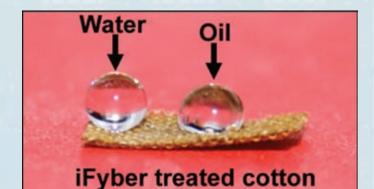


Figure 1: The technology developed at Cornell imparts unique properties such as simultaneous water and oil repellency to fabrics while preserving their air permeability and comfort properties.

wide range of applications," said Strickland, director for research and development of iFyber.

To date, the company has received two Small Business Innovative Research Grants from the U.S. Department of Defense for developing custom fabrics using nanotechnology. One project is to develop a material that can detect and identify leaks in chemical warfare suits used by the U.S. Air Force. The second is to create novel antibacterial wound dressings and surgical sutures for the U.S. Navy.

In addition, undergraduate students from Cornell's School of Hotel Administration, under the direction of Professors Robert Kwortnik and Michael Sturman, conducted a preliminary market analysis on using the technology to create antibacterial bedding and linens for the hospitality industry. "We are awaiting further market analysis before moving into the linen market," he said.















#### LEGO Models Show How Things Move

By Sarah Perdue, Feb. 1, 2010, Cornell Chronicle

The national anthem plays. The teams, wearing matching uniforms, are announced to a cheering crowd. The music starts pumping. It's your typical ... science fair?

It was the Junior FIRST LEGO League Expo held on January 30 in Duffield Hall, where 18 teams of elementary school students from central New York displayed their science and technology research projects — and their working LEGO models — on such diverse topics as how luggage gets from a ticket counter to an airplane, how mail gets delivered and how amphibious vehicles move.

The expo was developed by FIRST, which stands for For Inspiration and Recognition of Science and Technology. FIRST is a nonprofit organization started in the early 1990s by Segway founder Dean Kamen. "Kamen got frustrated seeing kids get excited about sports heroes but not about scientists and engineers," said Dan Woodie, who organized the day's event. "The whole idea is to get that sports atmosphere, that excitement, and show we can get excited about science and technology."

The teams build a LEGO model and make a poster that they present to a panel of reviewers at the expo. The theme of this year's expo was Smart Move. The Rockin' Red Rapid Rockets from Victor Primary School in Victor, N.Y., picked chocolate.

"Our model shows how chocolate gets made, from the bean to the bar," said team members as they presented their poster, which showed how cocoa beans get to the Hershey factory. Then they demonstrated their LEGO model, "There's a battery in it, and it moves the gears to move the box with the chocolate in it along the conveyor belt to the truck, which takes it out of the factory." The team agreed that working together and making new friends was the best part of participating. That, and they got to eat lots of chocolate.

# New Tools & Upgrades at the CNF

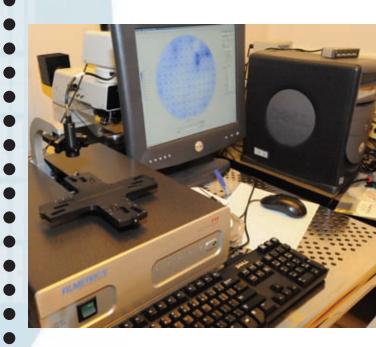
#### **FilMetrics**

We have replaced our older F50/F20 FilMetrics tool with a FilMetrics F50-EXR that brings some new features to the CNF.

In addition to making measurements about 5X faster, the tool uses light from 380 nm to 1700 nm to measure films up to 250  $\mu$ m thick. Additionally the tool has increased accuracy, and the ability to map refractive index and roughness over the wafer surface in addition to thickness.

Our F50/F20 system will be moved to our Organic Electronics lab on the 2nd floor to support the Reynoldstech deposition system there.

To learn more about this tool, contact Daniel Woodie, CNF Lab Use Manager, woodie@cnf.cornell.edu.



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The CNF NanoMeter is formatted by Ms. Melanie-Claire Mallison She welcomes vour comments at mallison@cnf.cornell.edu



#### **JEOL JBX-6300FS**



After 13 years of service, the VB6 has now been replaced by a JEOL JBX-6300FS. This tool will have a larger main field with better specifications than the VB6 and complement the capabilities of its big brother, the JBX-9300FS, which was installed back in 2003.

Both tools will operate at 100kV, have the same software and operating system, and use the

same pattern format, making the process of alternating between the two systems much easier than in the past. However, the 6300 is more than just a copy of the 9300.

The key feature of the new tool will be its ability to write at two different field sizes. In the "4<sup>th</sup> lens" mode it will have a 500  $\mu$ m field size, which will be upgraded to 1 mm sometime next year, and will perform similar to the 9300. However, the "5<sup>th</sup> lens" mode is what makes the 6300 truly unique.

In this mode, the beam spot will be less than 3 nm and the minimum address grid will be 0.125 nm. This combination will allow the routine patterning of features less than 8 nm, while still maintaining the overlay accuracy, alignment tolerance, and stability of a dedicated ebeam system. Additionally, line edge roughness and other artifacts that tend to show up on curved features, rings, and ellipses will be minimized.

The 6300 has not simply replaced an aging tool but has added a whole new capability.

Many users will find that both e-beam systems will suit the needs of their project. However, for those who are pushing the limits of lithography, the 6300 will be the tool of choice.

Contact Alan Bleier for more information on this tool, bleier@cnf.cornell.edu.

#### **Specifications:**

Writing Speed	25 MHz	(50MHz upgrade later this year)	
Field Size	4 <sup>th</sup> Lens, 500 µm	(1 mm upgrade next year)	
	5ª Lens, 62.5 µm		
Sample Size	Pieces to 6" wafer		
Field Stitching	< 30nm	(4 <sup>th</sup> lens)	
<b>Overlay Accuracy</b>	< 35nm	(4 <sup>th</sup> lens)	
Beam Diameter	< 3nm	(5th lens)	
Minimum Linewidth	< 8nm	(5th lens)	



#### **One Staffer's Moving Experience....**

Why do all the big tools come in when it's cold? The JBX-9300, the ASML, now the JBX-6300. If it's big and expensive, CNF will get it when there is snow on the ground and frostbite in the air. This was a bad week to quit drinking coffee.

Moving the new tool in only took one day, but preparations began months in advance. The outer wall of the VB6 room had to be removed and two new, larger doors installed for the installation of the JEOL. *This was done while the VB6 was still in operation*.

As the installation date approached, and the snow continued to fall, the Cornell grounds crew kept the path from the loading dock to the side of Duffield Hall clear of ice and snow, for the rigging crew from Al Smith rigging. The most critical — and heaviest — component, the column, had to be unloaded at the loading dock and then driven around the building on a forklift to the other side of Duffield. Here it was transferred to air pucks and moved into a service chase to be uncrated. Once uncrated, it could then be moved down the west hall of Duffield and into the cleanroom to its final home.

Meanwhile, other members of the rigging crew and JEOL were busy at the loading dock unloading the remaining eight crates and bringing them into Duffield.

Inside, the climate controlled comfort of the cleanroom space was needed for all the various components of the 6300. The AC Box was installed into the ebeam resist room. The wall was then moved to fit around it. (The resist room is just a little bit smaller now.) The chiller was installed into the 6300 service chase. However, to do this, the Zeiss Supra SEM first had to be deinstalled in order to make room for the chiller. A new transformer was also installed in the basement and power was run to the new AC Box, thanks to work with Richardson Brothers.

A lot of work was done by a lot of people and the installation is still currently in progress. But JEOL hopes to have a beam down the column by the third week of February and we should begin doing exposures by early March.

The Cornell NanoScale Facility has a long history of pushing the limits of nanofabrication. This tool will help us continue that tradition. Hopefully this will not be the last advanced lithography system the CNF acquires.

One thing is for certain.

Whenever we do get another system... it will be cold outside...







#### ASML PAS 5500/300C 248 nm DUV Wafer Stepper





175nm l/s in 420 nm UV82

200 nm l/s in 600 nm UV82

Lens		Field Size	Overlay	Throughput
NA	Resolution	Max X & Y	2 pt. Global Alignment	200 mm Wafers 70 Exp., 30 mJ/cm <sup>2</sup>
Variable 0.40 - 0.63	< 200 nm	22 × 27.4 mm	<u>≤</u> 45 nm	$\geq$ 80 wph

PAS 5500/300C DUV Stepper

CNF is pleased to announce the completion of installation and qualification of our ASML PAS 5500/300C 248 nm DUV wafer stepper. The addition of this tool adds greater capability to CNF and also fills a gap in our lithography tool set. Users of the lab will now be able to pattern feature sizes down to 200 nm and below using photolithography on any wafer size from 3" to 200 mm diameter.

A key component in the PAS 5500/300C's imaging is the AERIAL illuminator, which ASML introduced, and the optical system which was developed by Carl Zeiss, ASML's optical technology partner. AERIAL delivers conventional and off-axis illumination using continuously variable coherence and annularity settings. Zoom lenses in the illuminator maximize the exposure intensity at any setting. The third-generation deep-UV reduction lens, also a Zeiss product, has a fully automatic variable numerical aperture adjustable from 0.40 to 0.63, which allows the operator to select optimal resolution and depth of focus for each job and process layer. The combined lens and illuminator settings are continuously adjustable and can be automatically

set from the stepper's process recipe to suit the needs of each process layer. The magnification, field curvature and distortion of the projection lens are automatically servocontrolled for optimal imaging.

Through-the-lens phase grating alignment technology and precision stage accuracy achieve global overlay of 45 nm or less, with alignment through the lens allowing direct reticle reference and the elimination of baseline drift.

Using a 10-watt, 1,000-hertz excimer laser light source from Cymer Laser Technologies and Zeiss' double telecentric deep-UV optics, the PAS 5500/300C delivers more than 220 milliwatts per square centimeter at the wafer's surface. This high intensity reduces exposure times, enabling throughput of more than 80 200 mm wafers per hour at productionlevel doses of 30 millijoules per square cm.

Contact Garry Bordonaro if you are interested in being trained on this tool, or have questions on its capabilities. bordonaro@cnf.cornell.edu



#### **First Nano Carbon Nanotube Furnace**

The CNF has received shipment of a First Nano Easy Tube 3500 furnace system. This system will be configured with hydrocarbon gases for the growth of carbon nano-tubes and graphene on full 100 mm wafers. The supplier has demonstrated the growth of single wall tubes on lithographically defined pads prior to shipment of the unit. The system is load locked and has a low pressure vacuum option that will allow growth pressures to be adjusted from a few hundred mTorr up to atmospheric levels. The tool is currently in the process of being installed and is anticipated to be available for users in late spring 2010. Contact Phil Infante for more information, infante@cnf.cornell.edu.

#### ASML Update, Part 2

In 2009, CNF acquired and installed an ASML PAS 5500/300C DUV stepper which operates at 248 nm. The lens column has a numerical aperture NA = 0.63 to provide a 4:1 reduction with a variable

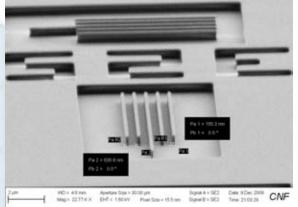


Figure 1: Post BARC removal etch using a low pressure/ low power O, RIE process in the Oxford 82 system.

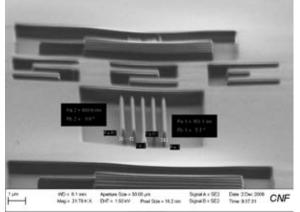


Figure 2: Post CHF/O<sub>2</sub> etch into thermal oxide (600 nm) using the Oxford 100 ICP system.

field size of up to 21 mm<sup>2</sup>. It can accommodate wafer sizes from 75-200 mm, as well as smaller size pieces.

The system specifications include linewidth resolution < 200 nm with an overlay accuracy of 45 nm. This capability successfully bridges the gap between i-line (365 nm) projection lithography and electron beam lithography.

Recently CNF staff members Garry Bordonaro, John Treichler, and Vince Genova performed a detailed characterization of the stepper's resolution and pattern transfer capabilities.

As device design rules continue to decrease, the advances in lithographic systems, such as the ASML DUV stepper, require new schemes for pattern transfer and process integration. The higher numerical aperture of the lens column not only provides higher resolution but also a reduced depth of focus at lower wavelengths. This reduced depth of focus requires that the photoresist become thinner which then limits the dry etch pattern transfer capability depending on the selectivity of etch process.

Therefore, nanoscale etching may require new and more complex schemes such as multi-level resists or etch masks. So in addition to the thin upper resist imaging layer and antireflection coating, a spin on hard mask, or a deposited hard mask such as amorphous carbon may be required. High numerical aperture tools can induce high reflection from ordinary substrates such as silicon (Si) and hence require the use of a bottom antireflection coating (BARC).

The characterization utilized an organic BARC (AR3) which is spun to a thickness of ~ 80 nm along with a DUV imaging resist (UV210) spun to thicknesses as great as 600 nm optimized parameters derived from a focus-dose matrix yielded linewidths of less than 150 nm, an impressive lithographic aspect ratio of > 4:1.

This result alleviates the need for a more complex hard mask scheme for subsequent pattern transfer. These results were obtained on Si, SiO<sub>2</sub>, SiN, and low stress SiN layers.

The pattern transfer process required the development of the BARC etch which uses a gentle anisotropic oxygen plasma in the Oxford 80 platform. This step needs to preserve the critical dimension (CD) that was defined lithographically and yet retain as much of the imaging resist thickness as possible. Once the BARC was removed, the underlayer films were etched using novel fluorocarbon chemistries and additive gases in the Oxford 100 to maximize resist selectivity along with etch profile control. Etch depths exceeding 1  $\mu$ m were achieved yielding an impressive etch aspect ratio along with CD preservation.

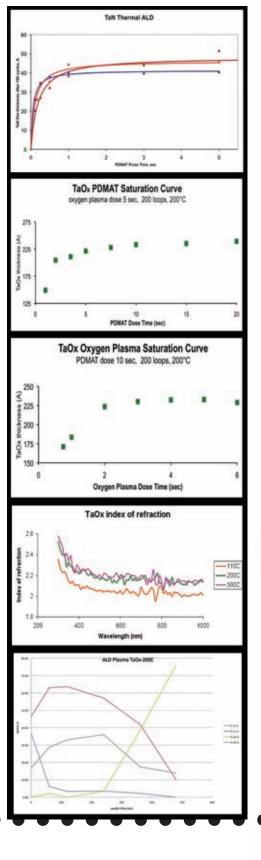
Treichler and Genova also demonstrated a mask fabrication process for the ASML. This process was developed on a Telic (6 in  $\times$  6 in  $\times$  0.250 in) quartz blank with entire in house CNF capability. This included the deposition of 30 nm of chrome in the CVC sputter system. The patterning utilized the aforementioned UV210 resist and AR3 BARC layers and was exposed with the Heidelberg DWL66 using a combination of the 10 mm and 2 mm heads.

Linewidths as small as 600 nm generated on the mask reduced to 150 nm after 4:1 reduction in the ASML. The BARC was etched in the Oxford 80 and the chrome was etched in the Trion Minilock III ICP system. In the near future, we plan to extend mask fabrication capability to phase shifting masks using a newly developed quartz etch in the Trion for the formation of relief structures.

Please feel free to contact Garry Bordonaro, John Treichler, or Vince Genova for details on the above developments.

## Furnace Diffusion Doping Sources Added

The CNF has added a few diffusion doping furnace processes to the existing tool set. A phosphorous oxychloride (POCL<sub>3</sub>) n-type doping furnace is available for non MOS clean substrates, providing sheet resistance values in the 1 to 1000 ohm per square ranges over typical process temps of 900°C to 1100°C. There also are two new solid source diffusion furnaces for boron and phosphorous diffusion using planar diffusion sources (solid sources). These will provide similar sheet resistance values at the POCl<sub>3</sub> process with temperature up to 1100°C for the boron source and 1025°C for the phosphorous source. These latter furnaces are for MOS Clean compatible substrates.



### **Atomic Layer Deposition (ALD) Update**

Since CNF's purchase of an Oxford Instruments FlexAL ALD system in 2008, we have continued to develop new thin film processes. Our ALD system has both thermal and plasma enhanced (PEALD) deposition capability for materials derived from hafnium (Hf), aluminum (Al), tantalum (Ta), and silicon (Si) based organometallic and organosilane precursors. In addition to our present list of processes including  $HfO_2$ , HfN,  $Al_2O_3$ , AlN, and  $SiO_2$ , we are pleased to offer tantalum nitride (TaN) and tantalum oxide (Ta<sub>2</sub>O<sub>2</sub>).

Ta-based thin films have been widely used for many important applications in nanoscale semiconductor device fabrication. TaN has been used as a diffusion barrier for Cu interconnect technology and is a leading candidate for a metal gate material in complementary metal oxide semiconductor (CMOS) devices. Metal gates along with high- $\kappa$  dielectrics allow for optimum device performance at ever decreasing transistor dimensions. In addition, Ta<sub>2</sub>O<sub>5</sub> thin films have been used as a capacitor dielectric material in dynamic random access memory (DRAM) devices, as well as a gate dielectric for nanoscale CMOS devices. These characteristics along with ALD's inherent qualities such as high conformality, high film quality at low growth temperatures, uniformity over large areas, and controllability at nanoscale thicknesses, make these and other materials quite attractive.

Our TaN film is derived from a thermal ALD process using an alkylamide precursor pentakis-dimethylamino-Ta (PDMAT) in the first half reaction that is then reduced with ammonia (NH<sub>3</sub>) in the second half reaction at a deposition temperatures between 225-300°C. The saturation curves illustrate excellent self-limiting behavior with deposition rates between 0.41-0.48A/cycle. We are in the process of developing a plasma based TaN film using PDMAT and a N<sub>2</sub>/H<sub>2</sub> based plasma for the second half reaction. PEALD has been developed to obtain films with higher quality at lower deposition temperature, largely due to the higher reactivity of the ions and radicals.

The Ta<sub>2</sub>O<sub>5</sub> film recently developed is a PEALD process at temperatures from 110-300°C. This utilizes the PDMAT precursor along with an oxygen plasma for the second half reaction. PDMAT dosages and oxygen plasma exposure times were optimized to generate saturation curves with self-limiting behavior with growth per cycle rates between 1.04-1.53A/cycle. The index of refraction was measured using spectroscopic ellipsometry and is between 2.1-2.2 at 630 nm. X-ray photoelectron spectroscopy (XPS) analysis indicates a film stoichiometry close to Ta<sub>2</sub>O<sub>5</sub>, which is targeted.

Carbon was detected at levels < 4 at.% which is excellent considering an organic precursor, while nitrogen was not detected indicating proper reaction and ligand exchange from the amino precursor. Our plan is to develop a thermal based tantalum oxide process in the near future.

Other future ALD film developments include deposition of silicon nitride along with more exotic hafnium silicates and aluminates. Please contact Vince Genova for further information on our ALD processes.

#### **IPE PECVD System Upgrade**

The IPE Plasma Enhanced Chemical Vapor Deposition (PECVD) tool has a new updated control software. In addition to having improved reliability, the new PC based control software will allow easier modifications to recipe parameters, easier setup of multi-step processes and more automation in controlling process parameters. The control software has an advanced data logging feature that will allow tracking current and past run parameters. Contact Phil Infante for more information, infante@cnf.cornell.edu.





Nanoimprint Lithography Update

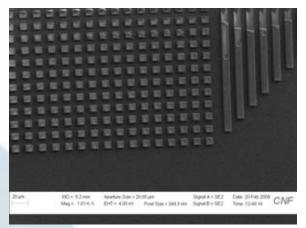


Figure 1: T-NIL into PMMA at 225°C

In the past year, CNF purchased and installed a Nanonex NX-2500 full wafer imprint system with alignment capability. Nanoimprint lithography (NIL) has the advantage of high throughput with sub-10nm resolution. It is so strategically important that NIL was included in the ITRS roadmap for 45nm and below nodes for advanced electronic devices. In addition to electronics, NIL is a benefit to many applications including displays, nanophotonics, nanofluidics, 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 300C 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 100mm diameter. The NX-2500 uses a patented air cushion press, which has the advantage of ultra-uniformity, low lateral stress, and less damage to the mold and substrate.

**Recently CNF staff members Vince Genova, John Treichler,** and Ed Camacho have developed T-NIL and P-NIL process flows including the fabrication of the imprint templates. Imprint templates can be made with a variety of materials such as silicon, fused silica, silicon carbide, sapphire, and others. Considerations for the selection of material include hardness, compatibility with traditional microelectronic fabrication, and thermal expansion coefficient to name just a few. We have developed processes for fabricating T-NIL templates in silicon and P-NIL templates in fused silica. In T-NIL processes, the temperature of the resist is heated above its glass transition temperature Tg, the template is pressed to deform the resist, and a replica of the template is left in the resist upon release. Our silicon template fabrication process starts with a silicon oxide deposition that is patterned lithographically and then pattern transferred into the oxide and silicon substrate using both conventional and ICP based RIE systems. For P-NIL processing, a UV resist layer containing a small percentage of silicon is applied over an organic resist transfer layer. After the template is pressed into the UV resist, UV exposure is used to crosslink the resist and convert it from the liquid to solid state. Since the P-NIL template needs to be transparent, we have developed a template fabrication process using fused silica wafers. The process starts with chrome sputter deposition that is patterned lithographically and then

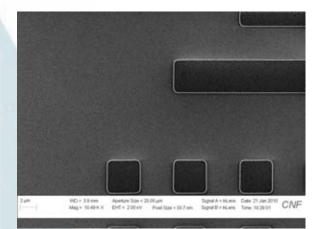


Figure 2: P-NIL sample following residual and transfer layer etches.

pattern transferred into the chrome and fused silica using newly developed etch processes in the Trion Minilock III ICP system. After etching the templates to a prescribed depth, the masking layers are removed, and then the template is coated with a fluorosilane anti-stiction layer (FOTS) in the molecular vapor deposition tool (MVD). The application of FOTS allows the template to be used for a series of wafers, eliminating the immediate need to clean the template.

Following the replication of template features into the resist, a very critical pattern transfer process must occur in the residual resist layer and resist/transfer layers for T-NIL and P-NIL processes respectively. The more complex pattern transfer is that for P-NIL since it involves a bi-layer resist scheme. We have recently developed conventional RIE processes for removal of the residual and transfer layers. The amount of residual UV resist remaining is a function of the applied imprint pressure. Since the UV resist contains silicon, it must be etched in a fluorocarbon chemistry, while the transfer layer is completely organic and must be etched in an oxygen chemistry. Both of these etches must preserve the critical dimension (CD) defined by the imprint and the transfer etch must be highly selective to silicon containing resist. Both of these etch processes have been developed on the Oxford 80 system. Once the resist is complete, the pattern can then be transferred to the substrate or underlayers. We have recently demonstrated NIL pattern transfer into silicon, silicon oxide and silicon nitride.

For further information on NIL, please contact Vince Genova, John Treichler, or Ed Camacho.

#### Suss Microtec MA/BA 6



The MA/BA6 contact aligner from Suss Microtec has arrived and has been qualified for processing. This aligner has a standardized lithography platform optimized for research and development in the various fields of nanotechnology.

Some of the exciting elements that set this system apart from others are its exposure, alignment and bonding compatibility systems. The full field allows for the full exposure of the substrate. The diffraction-reducing exposure optics are made up of a compact modular system. This accommodates various filters for a broad range of applications and resist systems. Three exposure modes (soft, hard, and vacuum) come standard with the tool, along with optimized wafer leveling to improve depth of focus and the critical dimension one is trying to print.

The MA/BA 6 offers four forms of alignment:

- top side
- bottom side
- infrared; and
- enhanced image storage system

<u>Top Side</u> alignment has a tolerance accuracy of  $\pm -0.5 \mu m$ .

<u>Bottom Side</u> alignment is composed of bright-field bottomsided microscopes with the accuracy of 1  $\mu$ m. These work alongside the high resolution CCD cameras that are set up for single and split-field.

The <u>Infrared</u> alignment system is added for transmittance and/or incident illumination via visual detection.

An Enhance Image Storage System (EISS) is used to save alignment marks for future use. The EISS also gives the flexibility of rudimentary image processing such as brightness and contrast between stored and live image.

Finally the MA/BA 6 brings back <u>Bond Alignment</u> (BA) capability to the CNF. The BA module works extremely well with the SB8e fixture and transport systems. This will also improve the level of bonding available, due to the allowed initial vacuum gap that the fixture provides prior to the bonding process. The SB8e and the MA/BA 6 come standard with 150 mm and 100 mm substrate chucks/fixtures.

The MA/BA 6 will be available for training very soon. Contact Edward Camacho for more information.

#### Alternative Method for Depositing the Conductive Polymer *EDOT* is Available

There is new tool available to users for the vapor-phase deposition of the transparent conductive polymer EDOT (ethylenedioxythiophene). This polymer is a transparent polymer with applications in photovoltaics, biosensors and flexible electronics including, touchscreens, organic light-emitting diodes, electronic paper.

The tool has been custom-built by ReynoldsTech, Inc.. to handle 2 ½-inch wafers, and the process is virtually hands-free by the use of a fully automated wafer-handling robot. This allows for better reproducibility and less human error.

Vapor-phase deposition is carried out in a nitrogen-enriched vacuum chamber that contains a heated crucible and a temperaturecontrolled platform. By creating a vacuum inside the chamber along with low heat, the chemistry can be vaporized and deposited onto the wafer in a mode that can be more uniform than traditional spin coating or printing methods. The cluster tool includes a wafer priming step using iron-III-tosylate to enhance the adhesion of the polymer.

The tool is located in the second floor wet lab (Duffield 224).



Contact Beth (rhoades@cnf.cornell.edu) to learn more and set up training.

#### **Plasma Etch Update**

We are pleased to announce several process updates and additions in the reactive ion etch area. These new processes were developed in response to device/application needs, new materials, and new lithographic capabilities with their associated pattern transfer requirements.

A low bias (low damage) silicon nitride (SiN) and low stress SiN etch was developed specifically for group-III nitride devices. The PECVD SiN is used to passivate the channel region and its selective removal must be done without inflicting damage to the active region, which would diminish its carrier transport properties. The etch parameter study was performed on the Oxford 81 using  $CHF_3/O_2$  chemistry. The DC bias values were maintained under -28V and still resulted in etch rates as high as 45 nm/min with resist selectivity of approximately 4:1. These etch conditions could be used for any device sensitive application.

To provide some process duplication from the PT72, hydrogen (H<sub>2</sub>) was added to the Oxford 80 systems and processes were developed using CF<sub>4</sub>/H<sub>2</sub>. The goal of this chemistry is to etch silicon oxide (SiO<sub>2</sub>) and SiN selectively to PMMA. Polymethylmethacrylate (PMMA) has notoriously poor selectivity in most fluorocarbon chemistry, however, when  $CF_4$  is combined with  $H_2$ , the fluorine/carbon ratio is reduced, enhancing the polymerization effect. The chemistry must be carefully balanced to allow polymerization to enhance selectivity but not inhibit the etch. Processes were developed for SiO<sub>2</sub>, and both LPCVD and PECVD SiN of low stress and stoichiometric forms. Selectivity as high as 8:1 is demonstrated with respect to PMMA. This allows us to lithographically define nanoscale features with a very thin layer of PMMA and yet effectively pattern transfer through a correspondingly thicker film, producing a higher aspect ratio.

We continue to develop etches for ALD deposited materials including TaN and  $Al_2O_3$ . A TaN etch study was done using BCl<sub>3</sub>/Cl<sub>2</sub> chemistry in the PT740, while an aluminum oxide etch was refined using BCl<sub>3</sub>/Ar also in the PT740. TaN etch rates ranged from 15-70 nm/min with selectivity to resist as high as 1.4:1. Aluminum oxide is difficult to dry etch due to its high bond strength, but the BCl<sub>3</sub>/Ar at a DC bias of -500V results in a controllable etch rate of 14 nm/min and a resist selectivity of 0.6:1.

As part of CNF's ongoing cooperative development agreement with Oxford Instruments, we continue our investigation into fused silica etching in the Oxford 100-380 ICP system. In this study, we compare three fluorocarbon chemistries (CHF<sub>3</sub>, C<sub>4</sub>F<sub>8</sub>, and C<sub>2</sub>F<sub>6</sub>) along with the role of specific additives oxygen, argon, and a novel CO<sub>2</sub>. Our latest results illustrate the effect of electrode temperature on the profile evolution. Because these are all heavily polymerizing gases, the etch temperature can be used to either mitigate polymer formation on the sidewalls at higher temperature, or enhance polymer formation at lower temperatures. Truly anisotropic etches have been demonstrated with C<sub>4</sub>F<sub>8</sub>/CO<sub>2</sub> and C<sub>2</sub>F<sub>6</sub>/Ar at 30°C. Our development activity has resulted in a paper "Characterization and comparison of fused silica etch processes in fluorocarbon based ICP chemistries" by Colin Welch (Oxford) and Vince Genova (CNF) to be presented at the PESM conference in Genoble, France, in March 2010.



Figure 1, left: Cr masked fused silica etched in  $C_4F_8/CO_2$  at 30°C in Oxford 100.

Figure 2, right: Cr masked fused silica etched in  $C_2F_6/Ar$  at 30°C in Oxford 100.

Our recent characterization of the ASML 248 nm DUV stepper included development of new etch processes for pattern transfer into  $SiO_2$  and SiN films. The bottom antireflective coating (BARC) etch was developed on the Oxford 80, while high resolution and high aspect ratio etches were developed on the Oxford 100 ICP system. Both the BARC etch and the film etches retain the critical dimension (< 150 nm) that was defined on the ASML along with a highly anisotropic etch profile.

A rather novel  $CHF_3/CO_2$  was used to etch into thermal oxide, and a  $CHF_3/O_2$  mix was used for the SiN etch. Selectivity to the DUV resist is 2.5:1.

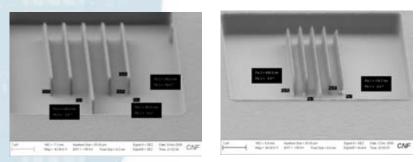


Figure 3, left: ASML defined SiN etched with CHF<sub>3</sub>/O<sub>2</sub> in Oxford 100.

Figure 4, right: ASML defined SiO, etched with CHF, CO, in Oxford 100.

As part of our Nanoimprint Lithography (NIL) effort, we have developed template fabrication processes for photocurable imprint lithography using fused silica substrates and quartz mask blanks. This includes the etching of the BARC in the Oxford 80 followed by a chrome and glass etch in the Trion Minilock III ICP system. The quality of the template etch is vital to the preservation of the critical dimensions and the replication of these features in the NIL process.

Contact Vince with any questions, genova@cnf.cornell.edu.



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