

IN THIS ISSUE

From the Director | 2

Flip Out! Noncommittal Material
Could Make For Hypersensitive
Magnetic Field Direction
Detector | 3

Snooping on Self-Organizing
Molecules | 4-5

Understanding Interactions
Between Light and Motion
in Cavity Optomechanical
Devices | 5

Automated Assembly of
Atomically Perfect Nanostructures
Using a Scanning Tunneling
Microscope | 6

Measuring Absorption Maps and
Spectra of Plasmonic Resonators
with Nanoscale Resolution | 7

A New Technique for Subsurface
Characterization of Thin-Film
Photovoltaic Materials | 8

Graphene Offers X-Ray
Photoelectron Spectroscopy
a Window of Opportunity | 9

CNST NanoFab Opens Soft
Lithography Lab | 9

NIH Researchers and NanoFab
Engineers Create Gratings for
Low-Radiation X-Ray Imaging | 10

Recent Publications by
CNST Staff Researchers | 11

Organizational News:
NanoFab Announces Operating
Status Alert System | 12

EDITOR |
ROBERT RUDNITSKY

ADDITIONAL CONTRIBUTORS |
MARK ESSER

GRAPHIC DESIGN |
KRISTEN DILL

DISCLAIMER | Certain commercial equipment, and software, are identified in this documentation to describe the subject adequately. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the equipment identified is necessarily the best available for the purpose.

FROM THE DIRECTOR

While I find it hard to believe, this May the Center for Nanoscale Science and Technology will celebrate its eighth anniversary.

Since May 2007, the Center has grown from an initial staff of about a dozen to a current roster of about ten times that size. During this period of expansion, which became rather breathless at times, our research participant population grew from a few hundred to over 2,100.

A great deal of planning went into CNST's unique design and I am pleased to say that it has actually worked out just about as we had hoped. Having a large postdoctoral population within the NanoLab has given us the agility to respond to shifting interests within the rapidly growing nanotechnology space. And, our investment in nanofabrication and measurement professionals and equipment within the NanoFab has provided us with a platform that attracts industrial researchers, government scientists, and academics alike.

There were a few, thankfully positive, surprises however. One, which really shouldn't have been a surprise had we taken the time to think about it, was how frequently the interaction between postdocs working on totally different projects would give rise to great ideas for new research directions.

The biggest surprise has been witnessing NanoFab users discover the NanoLab and vice versa. NanoFab users come to access our state-of-the-art tool set to make or measure nanostructures. NanoLab users collaborate with our researchers to develop fabrication and measurement solutions beyond the current commercial state-of-the-art. Each group is attracted to the CNST for a different reason. However, it's rather surprising how often a user of the NanoFab will realize that there is a great deal of skill, knowledge, and experience within the NanoLab staff that he can tap into. Similarly, researchers who come to the NanoLab to participate in an experiment soon learn to appreciate the proximity of the NanoFab and go on to use it for other research they have underway at their home institution. In this way, the center functions a bit like a bar magnet; one may be attracted to one end and then, by proximity, discover the other.

Most recently, we have been noticing how this effect has extended to NIST beyond the CNST. Our users are increasingly discovering the very deep expertise that exists among the scientific and technical staff here at NIST beyond the boundaries of the CNST. The result is typically a multi-institution, multi-disciplinary collaboration that jointly uses the shared toolset of the CNST. I think such serendipitous outcomes are among the best possible.

Robert Celotta

FLIP OUT! NONCOMMITTAL MATERIAL COULD MAKE FOR HYPERSENSITIVE MAGNETIC FIELD DIRECTION DETECTOR

While the mysterious, unseen forces magnets project are now (mostly) well-understood, they can still occasionally surprise us. For instance, thin films of cobalt have been observed to spontaneously switch their poles—something that typically doesn't happen in the absence of an external magnetic field. Researchers from the CNST and the University of Maryland have measured this phenomenon on the largest scale yet.

Most magnets are “permanent,” meaning a magnetic field of some strength must be applied to reverse their north and south poles. This permanence enables the billions of tiny magnets in computer hard drives to reliably store data. It also allows nanomagnetic sensor technology, for example, in the magnetometers which detect the earth's magnetic field in smartphone compasses. Making these devices more energy efficient will require magnets which are increasingly sensitive to external influences, such as small magnetic fields. However, as these magnets become more sensitive they also become more unstable, flipping from north to south and back, even with no magnetic field. The researchers mapped out this instability in a film of cobalt, only a few atoms thick, and determined the conditions under which the instability arises.

They hypothesize that the development of magnetic technology will benefit from their continuously flipping cobalt films, which can function as extremely sensitive magnetic test beds. Many proposed devices implement layers of ferromagnetic material which, to be useful, must be controllable by an external influence. According to CNST/UMD Postdoctoral Researcher Andy Balk, however, most magnetic materials are too stable to be influenced at all by small interactions, and researchers have no way of knowing if their proposed devices are even close to working. “As an alternative,” Balk says, “we could make a proposed magnetic device from our unstable film. This way, even if the film were influenced only a very small amount we would see, for example, slightly more north flips than south flips, and we would know we are on the right track.”

Polarized light microscopy, also called Kerr microscopy, can be used to extract different kinds of data about the magnetic state of an ultrathin layer of cobalt. **Bottom:** A map of the instantaneous magnetic state of the sample, where bright areas represent magnetic north poles and dark areas represent magnetic south poles. **Middle:** A map of the coercive field of the same sample. The map shows the sample's magnetic stability with bright colors representing areas with greater stability. **Top:** Spontaneous fluctuations over the same area, where brighter areas represent more fluctuation. Each image is 75 μm x 75 μm .

The measurements were done with video-rate Kerr microscopy, a form of polarized light microscopy that can image the fine-grained details of a material's magnetic state. The scientists found the magnetic fluctuations in the thin cobalt film interact with each other; a fluctuation from north to south will always have a corresponding nearby fluctuation from south to north.

Interestingly, these fluctuations exhibit scale invariance—meaning that their behavior is the same regardless of the length scale on which they are observed—a property they share with otherwise unrelated phenomena such as earthquakes and crumpling paper.

SNOOPING ON SELF-ORGANIZING MOLECULES

A few short years ago, the idea of a practical manufacturing process based on getting molecules to organize themselves in useful nanoscale shapes seemed ... well, cool, sure, but also a little fantastic. Now the day isn't far off when your cell phone may depend on it. Two recent papers emphasize the point by demonstrating complementary approaches to fine-tuning the key step: depositing thin films of a uniquely designed polymer on a template so that it self-assembles into neat, precise, even rows of alternating composition just 10 or so nanometers wide.

The work by researchers at the Massachusetts Institute of Technology, the IBM Almaden Research Center, the NIST Material Measurement Laboratory (MML), and the CNST focuses on *block copolymers*, a special class of polymers that under the proper conditions, will segregate on a microscopic scale into regularly spaced "domains" of different chemical composition. The two groups demonstrated ways to observe and measure the shape and dimensions of the polymer rows in three dimensions. The experimental techniques can prove essential in verifying and tuning the computational models used to guide the fabrication process development.

It's old news that the semiconductor industry is starting to run up against physical limits to the decades-long trend of ever-denser integrated chips with smaller and smaller feature sizes, but it hasn't reached bottom yet. Just recently, Intel Corp. announced that it had in production a new generation of chips with a 14-nanometer minimum feature size. That's a little over five times the width of human DNA.

At those dimensions, the problem is creating the multiple masking layers, sort of tiny stencils, needed to define the microscopic patterns on the production wafer. The optical lithography techniques used to create the masks in a process akin to old-school wet photography are simply not capable of reliably reproducing the extremely small, extremely dense patterns. There are tricks you can use such as creating multiple, overlapping masks, but they are very expensive.

Hence the polymers. "The issue in semiconductor lithography is not really making small features—you can do that—but you can't pack them close together," explains CNST Nanofabrication Research Group Leader Alexander Liddle. "Block copolymers take advantage of the fact that if I make small features relatively far apart, I can put

Transmission electron microscope (TEM) tomography provides a nanoscale, 3D visualization of the structure of a templated block copolymer. The purple features are silica posts fabricated by electron-beam lithography that direct the self-assembly of the copolymer. The material self-assembles to form two orthogonal layers of cylinders (green). Bounding box dimensions: 296 nm × 296 nm × 40 nm.

the block copolymer on those guiding patterns and sort of fill in the small details." The strategy is called "density multiplication" and the technique, "directed self-assembly."

Block copolymers (BCPs) are a class of materials made by connecting two or more different polymers that, as they anneal, will form predictable, repeating shapes and patterns. With the proper lithographed template, the BCPs in question will form a thin film in a pattern of narrow, alternating stripes of the two polymer compositions. Alternatively, they can be designed so one polymer forms a pattern of posts embedded in the other. Remove one polymer, and in theory, you have a near-perfect pattern for lines spaced 10 to 20 nanometers apart to become, perhaps, part of a transistor array.

If it works. "The biggest problem for the industry is the patterning has to be perfect. There can't be any defects," says MML researcher Joseph Kline. "In both of our projects we're trying to measure the full structure of the pattern. Normally, it's only

easy to see the top surface, and what the industry is worried about is that they make a pattern, and it looks okay on the top, but down inside the film, it isn't."

Kline's group, working with IBM, demonstrated a new measurement technique, called "resonant critical dimension small angle X-ray scattering" (res-CDSAXS), that uses low-energy or "soft" X rays produced by the Advanced Light Source at Lawrence Berkeley National Labs to probe the structure of the BCP film from multiple angles. Because the film has a regular, repeating structure, the scattering pattern can be interpreted, much as crystallographers do, to reveal the average shapes of the stripes in the film. If a poor match between the materials causes one set of stripes to broaden out at the base, for example, it will show up in the scattering pattern. Their major innovation was to note that although the basic technique was developed using short-wavelength "hard" X-rays that have difficulty distinguishing

SNOOPING ON SELF-ORGANIZING MOLECULES (CONT.)

two closely related polymers, much better results can be obtained using longer wavelength X-rays that are more sensitive to differences in the molecular structure.

While X-ray scattering can measure average properties of the films, Liddle's group, working with MIT, developed a method to look, in detail, at individual sections of a film by doing three-dimensional tomography with a transmission electron microscope (TEM). Unlike the scattering

technique, the TEM tomography can actually image defects in the polymer structure—but only for a small area. The technique can image an area about 500 nanometers across.

Between them, the two techniques can yield detailed data on the performance of a given BCP patterning system. The data, the researchers say, are most valuable for testing and refining computer models. "Our measurements are both fairly time-consuming, so they're not something industry

can use on the fab floor," says Kline. "But as they're developing the process, they can use our measurements to get the models right, then they can do a lot of simulations and let the computers figure it out."

"It's just so expensive and time-consuming to test out a new process," agrees Liddle. "But if my model is well validated and I know the model is going to give me accurate results, then I can crank through the simulations quickly. That's a huge factor in the electronics industry."

Determination of the internal morphology of nanostructures patterned by directed self assembly, D. F. Sunday, M. R. Hammond, C. Wang, W. Wu, D. M. Delongchamp, M. Tjio, J. Y. Cheng, J. W. Pitera, and R. J. Kline, *ACS Nano* **8**, 8426–8437 (2014).

3D TEM tomography of templated bilayer films of block copolymers, K. W. Gotrik, T. Lam, W. Bai, A. F. Hannon, Y. Ding, J. Winterstein, A. Alexander-Katz, J. A. Liddle, and C. A. Ross, *Advanced Functional Materials* **24**, 7689–7697 (2014).

UNDERSTANDING INTERACTIONS BETWEEN LIGHT AND MOTION IN CAVITY OPTOMECHANICAL DEVICES

Researchers from the CNST have combined simulation, fabrication, and measurements to develop a quantitative understanding of how light couples to motion in nanoscale cavity optomechanical devices made of gallium arsenide (GaAs). They have used this understanding to develop devices in which the optomechanical interaction is large, enabling the demonstration of low-power, optically-driven GHz frequency mechanical oscillators. This work is significant because understanding the strength of the optomechanical interaction in similar devices is necessary for cavity optomechanical systems to be used effectively in chip-scale sensors and in quantum information applications.

In cavity optomechanics, mechanical vibrations influence the light confined within an optical resonator. Conversely, the light within the resonator also influences the resonator's mechanical motion. The mechanical vibrations induce a time-varying change in the number of optical wavelengths that can fit within the cavity. In solid materials this change is mainly determined by two factors: the change in the physical size of the resonator (the "moving boundary" effect) and the change in the wavelength of light inside the material comprising the resonator due to changes in the material's refractive index (the "photoelastic" effect). The authors' work explained how these effects contribute to the optomechanical interaction in structures that confine near-infrared light and GHz frequency mechanical vibrations to micrometer-scale volumes.

Left: Scanning electron micrograph of an array of GaAs nanobeam optomechanical crystals with varying in-plane orientations. A pronounced change in the optomechanical interaction strength is observed depending on the device's orientation. This dependence is a direct result of the different photoelastic properties of GaAs along different crystalline planes. **Right:** One device within the array that has a [110] crystal orientation.

The researchers used detailed electromagnetic and structural mechanics simulations of microdisk and nanobeam optomechanical crystals to understand their optical and mechanical properties, and to predict the strength of the optomechanical interaction. The calculations described how the interaction was distributed between the moving boundary and photoelastic effects, and how these effects were expected to change as a function of microdisk diameter, nanobeam orientation, and other parameters. Then, using a custom-built near-field optical system developed in their laboratory for probing nanophotonic devices, they optically characterized devices fabricated in the CNST NanoFab in order to determine the strength of the optomechanical interaction and its behavior as a function of the parameters used in their calculations.

The experimental results strongly corroborated the theoretical calculations, allowing the researchers to use the theory to develop a nanobeam optomechanical crystal geometry in which the optomechanical interaction was especially strong. In these devices, the light field injects energy into the mechanical resonator, overcoming the resonator's intrinsic energy dissipation and resulting in self-sustained oscillations. The researchers believe that such optically-driven oscillators may be useful as low-noise radio frequency sources for a range of navigation and timing applications.

The team included researchers from the CNST, The Korea Institute of Science and Technology, and the University of Maryland.

Moving boundary and photoelastic coupling in GaAs optomechanical resonators, K. C. Balram, M. Davanço, J. Y. Lim, J. D. Song, and K. Srinivasan, *Optica* **1**, 414–420 (2014).

AUTOMATED ASSEMBLY OF ATOMICALLY PERFECT NANOSTRUCTURES USING A SCANNING TUNNELING MICROSCOPE

NIST researchers have demonstrated the autonomous computer-controlled assembly of atoms into perfect nanostructures using a low temperature scanning tunneling microscope. The results, published in an invited article in the *Review of Scientific Instruments*, show the construction without human intervention of quantum confined two-dimensional nanostructures using single atoms or single molecules on an atomically flat copper surface.

A major goal of nanotechnology is to develop so-called “bottom up” technologies to arrange matter at will by placing atoms exactly where one wants them in order to build nanostructures with specific properties or function. The researchers, led by Robert Celotta and Joseph Stroscio from the CNST, have demonstrated the first steps towards achieving that capability using the atom manipulation mode of a scanning tunneling microscope (STM) in combination with autonomous motion algorithms.

The team, which includes Stephen Balakirsky (previously in EL and now at Georgia Tech), Aaron Fein (PML), Frank Hess (previously in the CNST), and Gregory Rutter (previously in the CNST and now at Intel), used autonomous algorithms to manipulate single atoms and molecules, much like the algorithms for “hands-free” car driving. The system works by first scanning the locations of available atoms on the surface. It then specifies the desired coordinates of atoms of a nanostructure, and autonomously calculates and directs the trajectories for the STM probe tip to move all the atoms to their desired locations.

Automated assembly of individual cobalt atoms on an atomically flat copper surface into simple geometric shapes: a square, a triangle, and a circle. From left to right, each figure shows the configuration after each atom move. Image size 15 nm × 15 nm. **Center:** Perfect assembly of the NIST logo after four steps of automated assembly. Image size 40 nm × 17 nm. All images are shown in colored 3D top view with light shadowing with a height range of ~100 pm.

The team was able to demonstrate that it could autonomously construct cobalt atoms into nanostructures that confine the quantum properties of the copper’s surface electrons. It then used the STM to measure those properties. In addition to demonstrating the construction of nanostructures made out of atoms, they demonstrated that it was possible to construct nanoscale lattices made of carbon monoxide molecules and to tailor-make interacting quantum dots formed from vacancies in the carbon monoxide lattices.

The researchers believe that an approach based on autonomous construction of atoms and molecules using this technique could be the foundation for an easily accessed toolkit for producing tailored quantum states with applications in quantum information processing and nanophotonics.

MEASURING ABSORPTION MAPS AND SPECTRA OF PLASMONIC RESONATORS WITH NANOSCALE RESOLUTION

Researchers from the CNST and the University of Maryland have for the first time used photothermal induced resonance (PTIR) to characterize individual plasmonic nanomaterials in order to obtain absorption maps and spectra with nanometer-scale resolution. Nanostructuring of plasmonic materials enables engineering of their resonant optical response and creates new opportunities for applications that benefit from enhanced light-matter interactions, including sensing, photovoltaics, photocatalysis, and therapeutics.

Progress in nanotechnology is often enabled by the availability of measurement methods for characterizing materials at appropriately small length scales. By measuring infrared absorption at the nanoscale, PTIR provides information that is not otherwise available for characterizing and engineering plasmonic materials. PTIR measures light absorption in a material using a wavelength-tunable laser and a sharp tip in contact with the sample as a local detector. Unlike many other methods that use nanoscale tips for probing materials, in PTIR the tip is passive and does not interfere with measurement. Consequently, light absorption in the sample can be measured directly without requiring either a model of the tip or prior knowledge of the sample.

The researchers collected nanoscale absorption information in two ways: first, by mapping infrared absorption while scanning a tip on a sample under constant wavelength illumination; and second, by measuring location-specific absorption spectra while sweeping a laser across a range of infrared wavelengths. Using tunable lasers that give CNST facility users the ability to vary the wavelengths from 1.55 μm to 16.00 μm , the researchers acquired the nanoscale infrared absorption spectra of gold resonators, the first such measurement of any plasmonic nanomaterial. Although absorption images allow immediate visualization and can be measured with other techniques, the PTIR spectra provide needed information to interpret the images and guide experiments.

Schematic showing the photothermal induced resonance (PTIR) technique, which combines the lateral resolution of atomic force microscopy (AFM) with the chemical specificity of IR spectroscopy. A wavelength-tunable, pulsed IR laser (purple) illuminates a sample consisting of plasmonic gold resonators from the below. The resulting thermal expansion of the sample is detected locally by the AFM cantilever tip, which is monitored by reflecting a laser (blue) off the back of the cantilever.

Plasmonic materials like gold, which have large thermal conductivity and relatively small thermal expansion coefficients, were previously thought to be challenging to measure using PTIR because the technique relies on the sample's thermal expansion for measuring light absorption. According to Andrea Centrone, a Project Leader in

the Energy Research Group, "we showed that PTIR characterization is not just applicable to insulators and semiconductors, as demonstrated previously, but that metals are also amenable to it. This is an important step forward for applying the PTIR technique to a wider variety of functional devices."

A NEW TECHNIQUE FOR SUBSURFACE CHARACTERIZATION OF THIN-FILM PHOTOVOLTAIC MATERIALS

Although the performance of many materials used in devices is determined by microscale and nanoscale structures and buried interfaces hidden up to a few micrometers below the material surface, most commonly used nanoscale characterization tools are only sensitive to phenomena either at the surface or just a few nanometers below it. As a step towards addressing the increasing need for subsurface characterization with nanoscale spatial resolution, researchers from the CNST and the University of Maryland have demonstrated a new approach for probing the interior of photovoltaic devices.

The technique uses a scanning probe microscope to control a tapered optical fiber with a nanoscale aperture that transmits laser light in order to locally excite a subsurface volume of a photovoltaic device. The photocurrent generated by the cell is then measured using a low-noise amplifier. By varying the wavelength of the laser light in the optical fiber, the penetration depth of the light can be readily changed from approximately 100 nm to

more than 3 μm , allowing different volumes of the device to be excited. However, deeper penetration results in lower spatial resolution. Therefore, for comparison, the researchers used a focused ion beam available to users in the CNST NanoFab to prepare a wedge-shaped device sample by carefully thinning down the absorber thickness at a small angle while ensuring that the device still functioned as a solar cell. This shape effectively brings some of the buried interfaces closer to the exposed surface so that these interfaces can be interrogated with higher spatial resolution using light with shallower penetration.

The characterization technique was demonstrated on cadmium telluride (CdTe) solar cells, a common photovoltaic technology. These solar cells are based on 1 μm to 3 μm thick multilayer polycrystalline films with the size of the crystal grains comparable to or smaller than the device thickness. The cells have both intentional and unintentional variation

in composition throughout the absorber layer and device interfaces, making them ideal for testing this technique. Currently, solar cells of this type operate at efficiencies well below the theoretical limit, and their inefficiency is believed to be due to the uncontrolled and often unknown effects of the microscale structure buried beneath the surfaces of the devices.

According to CNST researcher Nikolai Zhitenev, the new technique is just an early step towards the development of a full set of quantitative nanoscale sub-surface measurement tools. While patterning the photovoltaic device into a wedge shape inevitably modifies its structure and performance, the measurements are highly reproducible for a variety of processing conditions. Through the development of new theoretical models explicitly incorporating the effects of the device modification, the new measurement approach can be developed into a powerful quantitative technique.

LEFT: Schematic of the experimental apparatus. A tapered optical fiber probe with a nanoscale aperture transmits laser light and locally excites a subsurface volume of a photovoltaic device. The resulting photocurrent can then be measured using a low-noise amplifier. **RIGHT:** By varying the wavelength of light, nanoscale spatial variations in external quantum efficiency (EQE) are mapped.

GRAPHENE OFFERS X-RAY PHOTOELECTRON SPECTROSCOPY A WINDOW OF OPPORTUNITY

X-ray photoelectron spectroscopy (XPS) is among the most sensitive and informative surface analysis techniques. However, it requires high vacuum equipment for operation because electrons can only penetrate a short distance in dense media such as air. An international team led by CNST researchers working with collaborators from Elettra Sincrotrone Trieste, Italy and the Technical University of Munich, Germany have overcome this limitation by employing the fact that graphene is transparent to electrons (electrons can pass through it largely unimpeded) but is impermeable to gas or liquids.

The researchers used graphene covering a small opening to separate a liquid sample cell from the high vacuum conditions of the electron spectrometer. They were able to demonstrate that good quality XPS data can be recorded from liquid using the approach. They evaluated the electron transparency of graphene membranes quantitatively and compared it with theoretical predictions.

Additionally, the researchers were also able to spectroscopically measure *in situ* the chemistry of

Schematic of an X-ray photoelectron spectroscopy instrument incorporating suspended, electron-transparent graphene membranes—or windows—that separate the sample from the high-vacuum detection system.

bubble formation due to radiation-induced splitting of water into oxygen and hydrogen. Because the bubble formation is a frequent unwanted process, the measurement of the onset of bubble formation sets an upper limit for the intensities of the X-rays (or electrons) which can be used in this approach.

The researchers' work potentially fills a much needed gap. Assessing the chemical status of

surfaces and interfaces immersed in liquids or atmospheric pressure gas is very much needed for many applications such as biomedical research, electrochemical energy devices, and catalysis. The current state of the art of high pressure XPS is to use sophisticated, expensive and bulky differentially pumped stages in front of the electron energy analyzer. Only a few instruments of this kind are currently available worldwide but there is great need for these experimental capabilities. The researchers' design is far simpler and has the potential to reduce costs to the level that this type of measurement could be afforded by many more labs.

As often happens with new technologies, there remain some challenges and limitations. The graphene adhesion to the surface of the apertures needs to be improved, as do the radiation and electrochemical stability of the atomically thin graphene. The researchers plan to work on these challenges in order to develop better techniques for clean and non-disruptive transfer of graphene windows to the supporting openings.

Photoelectron spectroscopy of wet and gaseous samples through graphene membranes, J. Kraus, R. Reichelt, S. Gunther, L. Gregoratti, M. Amati, M. Kiskinova, A. Yulaev, I. Vlassiuk, and A. Kolmakov, *Nanoscale* 6, 14394–14403 (2014).

CNST NANOFAB OPENS SOFT LITHOGRAPHY LAB

The CNST NanoFab has established a new soft lithography laboratory. This lab provides facility users with rapid access to tools and processes for fabricating devices in soft materials, with applications ranging from microfluidics and nanofluidics, to flexible electronics and optics, to devices with patterned surface chemistries.

Located in Building 216, room F102, the 25 m² lab is equipped with a casting station for polydimethylsiloxane (PDMS) coating, a plasma cleaner for surface preparation, a convection oven for PDMS baking, and a laminar clean hood for final assembly. The casting station is housed in a fume hood, which also includes a spin coater, a scale, a hot plate, and a desiccator for PDMS mixing, degassing, coating, and baking. The casting station and oven support substrates ranging from 150 mm diameter wafers down to small pieces and the plasma cleaner supports wafers as large as 75 mm in diameter. Over the next 15 months, the

CNST plans to double the soft lithography lab space to 50 m² and add tools to improve process reliability and repeatability. The new tools will include a silane vapor deposition tool, a PDMS mixing tool, and a PDMS punch.

To arrange lab access and training, or for more information, please contact Robert Newby, 301-975-6070, robert.newby@nist.gov.

NIH RESEARCHERS AND NANOFAB ENGINEERS CREATE GRATINGS FOR LOW-RADIATION X-RAY IMAGING

Scientists at the National Heart, Lung, and Blood Institute at the National Institutes of Health (NIH), working with engineers in the CNST NanoFab have developed a cost-effective process to fabricate large area gratings with high-aspect-ratio (HAR) lines and small pitch (spacing between the grating lines). These gratings have been successfully used as a component in an experimental low dose and compact (desktop) X-ray imaging system, pointing a path to phase-contrast X-ray systems that could expose patients to lower radiation than conventional systems while providing high resolution images.

Traditional medical X-rays rely on high-energy X-ray photons passing through the human body, where they are partially absorbed, with the emerging radiation forming a shadow image on a detector plate. Computed tomography (CT) scans have become increasingly popular in recent years and use multiple X-ray images to provide three dimensional data, exposing patients to between 150 and 1,100 times the radiation of a single conventional x-ray. Inherently, these techniques

lead to the absorption of harmful ionizing radiation, which is a growing public concern.

Grating based X-ray imaging technology may provide a solution to the problem of high dose X-ray absorption in current CT scans. Phase contrast measurements detect the deflection of the X-rays instead of their absorption and allow lower dosing to be used. Detectors sense the distortion of the wavefront after it passes through a sample.

However, a key requirement of the far-field phase contrast detection mechanism needed for medical applications is that the period of grating lines used in these devices must be comparable to or smaller than the coherent area of the X-rays (the area in which the photons maintain a fixed phase relationship). Unfortunately, the fabrication of phase imaging gratings that meet that requirement has not been easy. Large area gratings with small pitch have been produced using e-beam lithography but at costs that are not affordable by industry.

Using equipment available to users of the NanoFab, the NIH/CNST team was able to address

these challenges. The researchers made molds out of single crystal silicon, due to its superior mechanical properties. They were able to show that nanoimprint lithography could be used to pattern large area gratings with the required small pitch (100 nm half pitch). They also developed new plasma etching and atomic layer deposition processes to make and coat the HAR (>32) nanometer-scale grating trenches. Using cryogenic reactive ion plasma etching, they created deep trenches which were then conformal coated using atomic layer deposition of a thin (≈ 10 nm) layer of platinum. With the platinum coating as a seed layer, gold was filled between the trench walls using electroplating.

These processes are replicable and scalable, and can lower the patterning cost. The researchers believe that in addition to finding use in medical X-ray devices, the newly developed HAR nano-grating fabrication process may also be useful for making small but high surface area sensors and catalysts.

Cross-sectional scanning electron micrographs of etched silicon grating molds with a half-pitch of 100 nm. **Left:** This mold has 3.2 μm trenches, giving a depth-to-wall thickness aspect ratio of 32. **Right:** This mold has a depth of 4.0 μm and an aspect ratio of 40.

RECENT PUBLICATIONS BY CNST STAFF RESEARCHERS

Below is a list of recent publications by CNST staff researchers. Visit the CNST Publications Portal (nist.gov/cnst/pubs/index.cfm) to view full text versions of these papers and a complete list of publications from research supported by the CNST, including work performed in the NanoFab and by CNST grant recipients.

3D TEM tomography of templated bilayer films of block copolymers, K. W. Gotrik, T. Lam, W. Bai, A. F. Hannon, Y. Ding, J. Winterstein, A. Alexander-Katz, J. A. Liddle, and C. A. Ross, *Advanced Functional Materials* **24**, 7689–7697 (2014).

Moving boundary and photoelastic coupling in GaAs optomechanical resonators, K. C. Balram, M. Davango, J. Y. Lim, J. D. Song, and K. Srinivasan, *Optica* **1**, 414–420 (2014).

Dissipation due to pure spin-current generated by spin pumping, T. Taniguchi and W. M. Saslow, *Physical Review B* **90**, 214407 (2014).

Flat lens criterion by small-angle phase, P. Ott, M. H. Al Shakhs, H. J. Lezec, and K. J. Chau, *Optics Express* **22**, 29340 (2014).

Invited Article: Autonomous assembly of atomically perfect nanostructures using a scanning tunneling microscope, R. J. Celotta, S. B. Balakirsky, A. P. Fein, F. M. Hess, G. M. Rutter, and J. A. Stroscio, *Review of Scientific Instruments* **85**, 121301 (2014).

Energy transfer between eigenmodes in multimodal atomic force microscopy, S. An, S. D. Solares, S. Santos, and D. Ebeling, *Nanotechnology* **25**, 475701 (2014).

Critical behavior of zero-field magnetic fluctuations in perpendicularly magnetized thin films, A. L. Balk, M. D. Stiles, and J. Unguris, *Physical Review B* **90**, 184404 (2014).

Direct evidence of active and inactive phases of Fe catalyst nanoparticles for carbon nanotube formation, S. Mazzucco, Y. Wang, M. Tanase, M. Picher, K. Li, Z. Wu, S. Irlle, and R. Sharma, *Journal of Catalysis* **319**, 54–60 (2014).

Nucleation of graphene and its conversion to single-walled carbon nanotubes, M. Picher, P. A. Lin, J. L. Gomez-Ballesteros, P. B. Balbuena, and R. Sharma, *Nano Letters* **14**, 6104–6108 (2014).

Nanoscale imaging of photocurrent and efficiency in CdTe solar cells, M. S. Leite, M. Abashin, H. J. Lezec, A. Gianfrancesco, A. A. Talin, and N. B. Zhitenev, *ACS Nano* **8**, 11883–11890 (2014).

Insights into carbon nanotube nucleation: cap formation governed by catalyst interfacial step flow, R. Rao, R. Sharma, F. Abild-Pedersen, J. K. Nørskov, and A. R. Harutyunyan, *Scientific Reports* **4**, (2014).

Faceting control in core-shell GaN micropillars using selective epitaxy, S. Krylyuk, R. Debnath, H. P. Yoon, M. R. King, J.-Y. Ha, B. Wen, A. Motayed, and A. V. Davydov, *APL Materials* **2**, 106104 (2014).

Spectral broadening and shaping of nanosecond pulses: toward shaping of single photons from quantum emitters, I. Agha, S. Ates, L. Sapienza, and K. Srinivasan, *Optics Letters* **39**, 5677–5680 (2014).

CENTER FOR NANOSCALE SCIENCE AND TECHNOLOGY

The CNST is a national user facility purposely designed to accelerate innovation in nanotechnology-based commerce. Its mission is to operate a national, shared resource for nanoscale fabrication and measurement and develop innovative nanoscale measurement and fabrication capabilities to support researchers from industry, academia, NIST, and other government agencies in advancing nanoscale technology from discovery to production. The Center, located in the Advanced Measurement Laboratory Complex on NIST's Gaithersburg, MD campus, disseminates new nanoscale measurement methods by incorporating them into facility operations, collaborating and partnering with others, and providing international leadership in nanotechnology.

If you would like to subscribe to this newsletter, go to <http://www.nist.gov/cnst/newsletters.cfm>, enter your e-mail address, and follow the on-screen instructions. Alternately, you may request a subscription via email or phone.

SUPPORTING THE DEVELOPMENT OF NANOTECHNOLOGY FROM DISCOVERY TO PRODUCTION

Center for Nanoscale Science and Technology
National Institute of Standards and Technology
100 Bureau Drive, MS 6200
Gaithersburg, MD 20899-6200
Phone: 301-975-8001
E-mail: cnst@nist.gov

NANOFAB ANNOUNCES OPERATING STATUS ALERT SYSTEM

According to NanoFab Manager Vincent Luciani, the CNST has the goal of keeping the NanoFab open year-round, with staff support available from 7 am to midnight and after-hours access possible 24 hours a day, 7 days a week. But, according to Luciani, "mother nature sometimes has other ideas."

On days when weather conditions make it unsafe to come to NIST, the decision to close or delay opening the NanoFab is typically made by 6 AM.

This information is communicated to users in the following ways:

By email: By 6:30 AM, all NanoFab users and PI's (NIST and non-NIST) will receive a *NanoFab Alert* advising them of the closure or delayed opening.

On our web page: The *Operating Status* box on the NanoFab home page will be updated by 6:30 AM.

By calling NIST: Users can call 301-975-8000, the NIST status phone number, for information about NIST's operating status.

When NIST is closed due to weather conditions, the NanoFab will also be closed. It also will not be accessible through the "buddy system."

The Operating Status box at the top of the right column on the NanoFab home page (<http://www.nist.gov/cnst/nanofab/>) will be updated by 6:30 AM to indicate any closures or delayed openings.

According to Luciani, users should not base their decisions to come solely on TV or radio announcements about "Federal Government" status. Occasionally, local conditions on the Gaithersburg, MD campus will force NIST to close even though the Federal Government is open.

If you have questions about the NanoFab's weather related closing policy, please contact: nanofabuseroffice@nist.gov