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ART AND SCIENCE MELDED IN A RECENT EXHIBIT

An electron micrograph of a glassy metal (left), recorded by CNST scientist John Unguris, was on display at a recent show at John Hopkins University that featured the work of NIST researchers. Photo by Fran Webber
A HEAVENLY MISSION: FILTER FABRICATED AT THE NANOFAB SET TO HELP NASA SURVEY STARBIRTH

If all goes according to plan, an infrared filter meticulously engineered at the CNST will have a starring role next summer. The gold mesh filter, whose fine lines and large size demanded the exquisitely stable fabrication system at the NanoFab, is set to fly aboard NASA balloon-borne experiment that will peer at hidden pockets of starbirth across the sky.

Work on the filter began earlier this year, when astronomers from NASA’s Goddard Space Flight Center in Greenbelt, Md., contacted the NanoFab with an unusual design. In order for their telescope, known as BETTII (Balloon Experimental Twin Telescope for Infrared Interferometry) (http://asd.gsfc.nasa.gov/bettii/), to study starbirth, the instrument had to record certain wavelengths of infrared light while rejecting others. To do so, BETTII would need a filter crisscrossed with lines 100 nanometers wide (one ten-millionth of a meter) and spaced one micrometer (one-millionth of a meter) apart.

The filter “required high precision techniques, and the NanoFab lab at NIST was an ideal partner for building it,” says NASA astronomer Stephen Rinehart, principal investigator for the BETTII mission.

The grid pattern for the filter could be written by a tightly focused beam of electrons, acting like a miniature ballpoint pen, drawing the pattern onto a surface covered with an electron-sensitive film. The NanoFab’s state-of-the-art electron beam lithography tool was just right for the job. But then came an even bigger challenge.

Even with a system as advanced as the one at the NanoFab, electron beam lithography can only write patterns over a field 1 square millimeter in area at a time. The filter, however, has an area about 800 times larger.

To inscribe a grid over such a large region would therefore require that the lithography system move the sample precisely 1 millimeter after completing each inscription, some 800 times in succession. And because the grid pattern has to be seamless, with no discernable difference from one field to the next, the system would have to operate absolutely uniformly during a continuous run of more than 16 hours. Although typical write times for a lithography system are several hours or less, several features of the NanoFab system indicated it could do the job, says NanoFab process engineer Richard Kasica.

The system is enclosed in a temperature-controlled chamber, promoting stability. In addition, it inscribes patterns relatively fast over a large area.

After test runs in April and May, Kasica chose a weekend in June—a time when the lithography system would not likely be needed by researchers—and commanded the electron beam to do its work. The inscribed material consisted of a piece of sapphire coated with a polymer resist and capped with a thin, electrically conducting layer of aluminum. The high-energy electron beam, which readily penetrated the aluminum, inscribed the pattern in the resist material.

The fabrication process is an intricate interplay between the electron beam, various solvents, evaporated gold and the resist material. After penetrating the aluminum conducting layer, the high energy electron beam breaks molecular bonds where it strikes the resist material. Those weakened resist regions wash away when the substrate is immersed in a developer solvent.

A thin layer of gold is then deposited, sticking to the bare sapphire in areas where the solvent had removed resist material and blanketing the resist in areas that had not been weakened by the electron beam. In the final step, the remaining resist is dissolved in a strong solvent, lifting off the gold deposited on those regions as well. Only the gold sticking to the underlying sapphire remains, forming the mesh pattern directly on the material. Increasing the chances for success, the NanoFab staff chose and perfected a resist film that made it easy to inscribe and lift off the dense pattern of lines required by NASA.

A visual inspection of the filter indicated that the NanoFab’s electron beam lithography system had met NASA’s design challenge.

The BETTII mission relies on the detection of infrared light to study nascent stars because the hatchlings form deep within cocoons of dust and gas that are impervious to visible-light radiation. In contrast, infrared light emitted by the newborns can exit the dusty cocoons, allowing astronomers to view the birthing process. In addition, infrared light emitted by the dust itself reveals how the material is reshaped by the energetic radiation that young stars blast into space once they emerge from their birthplace.

The mesh filter was set to fly on BETTII this past October, but strong winds scrubbed the flight, which has now been rescheduled for June.
From the printing press to the jet engine, mechanical machines with moving parts have been a mainstay of technology for centuries. As U.S. industry develops smaller mechanical systems, it faces bigger challenges—microscopic parts are more likely to stick together and wear out when they make contact with each other.

To help make microscopic mechanical (micromechanical) systems perform reliably for advanced technologies, researchers at the CNST and their NIST colleagues are getting back to basics, carefully measuring how parts move and interact.

For the first time, the NIST researchers have measured the transfer of motion through the contacting parts of a microelectromechanical system at nanometer and microradian scales. Their test system consisted of a two-part linkage, with the motion of one link driving the other. The team not only measured the motion with record precision but also studied its performance and reliability.

Lessons learned from the study could impact the fabrication and operation of various micromechanical systems, including safety switches, robotic insects and manufacturing platforms.

The motion of micromechanical systems is sometimes too small—displacements of only a few nanometers, or one billionth of a meter, with correspondingly small rotations of a few microradians—for existing measurement methods to resolve. One microradian is the angle corresponding to the length of an arc of about 10 meters along the circumference of the earth.

“There has been a gap between fabrication technology and motion metrology—the processes exist to manufacture complex mechanical systems with microscopic parts, but the performance and reliability of these systems depend on motion that has been difficult to measure. We are closing that gap,” said Samuel Stavis, a project leader at the CNST.

“Despite how simple this system appears, no one had measured how it moves at the length and angle scales that we investigated,” said researcher Craig Copeland of the CNST and the University of Maryland. “Before commercial manufacturers can optimize the design of more complex systems such as microscopic switches or motors, it is helpful to understand how relatively simple systems operate under various conditions.”

The measurements, which the researchers report in *Microsystems & Nanoengineering*, rely on optical microscopy to track surface features on the moving parts. The manufacturer can build in the surface features during the fabrication process so that the system is ready for measurement right out of the foundry. Or, the researchers can apply fluorescent nanoparticles to
the system after fabrication for improved precision. The researchers introduced this measurement method in a previous study and have used related methods to track the motion and interaction of other small systems. Importantly, the ability to simultaneously track the motion of multiple parts in a micromechanical system allowed the researchers to study the details of the interaction.

In their experiment, the researchers studied the transfer of motion through a mechanical linkage, which is a system of parts connected in order to control forces and movement in machines. The test system had two links that connected and disconnected through a joint, which is the point at which the links apply forces to each other. The electrical heating and thermal expansion of one link drove the rotation of the other link around a pivot. The researchers developed a model of how the system should move under ideal operating conditions, and used that model to understand their measurements of how the system moved under practical operating conditions. The team found that play in the joint between the links, which is necessary to allow for fabrication tolerances and prevent the parts from jamming, had a central role in the motion of the system. Specifically, the amount of play was an important factor in determining precisely how the links coupled and uncoupled, and how repeatable this transfer of motion could be.

As long as the electrical input driving the system was relatively free of noise, the system worked surprisingly well, transferring the motion from one part to another very consistently for thousands of operating cycles. “It was perfectly repeatable within measurement uncertainty,” said Copeland, “and reasonably consistent with our ideal model.”

That is important, he notes, because some researchers expected that the friction between small parts would degrade the performance and reliability of such a system. Many engineers have even abandoned the idea of making micromechanical systems out of moving parts that make contact, switching to micromechanical systems with parts that move by flexing to avoid making contact with each other.

The results suggest that micromechanical systems that transfer motion through contacting parts “may have underexplored applications,” said Stavis.

However, the researchers found that when they added a normal amount of electrical noise to the driving mechanism, the system became less reliable and did not always succeed in transferring motion from one link to the other. Further, exposure of the system to atmospheric humidity for several weeks caused the parts to stick together, although the researchers could break them loose and get them moving again.

These findings indicate that while micromechanical systems have the potential to transfer motion between contacting parts with unexpectedly precise performance, the driving signal and operating environment are critical to the reliable output of motion.

The researchers now plan to improve their measurements and extend their work to more complex systems with many moving parts.

“Micromechanical systems have many potential commercial applications,” said Stavis. “We think that innovative measurements will help to realize that potential.”

This project was a collaboration between researchers at the CNST, NIST's Physical Measurement Laboratory and the University of Maryland as part of the NIST Innovations in Measurement Science program.

For more than a quarter century, the nanotechnology revolution has spurred the development of novel devices, materials and instruments in the laboratory—including carbon nanotubes, atomic-force microscopes and DNA-based self-assembling systems.

Increasingly, the challenge is in making the leap from the laboratory to the factory. Successful mass production of nanomaterials and devices requires careful consideration of processing methods along with cost, volume and time from factory to market. The manufacturing process, for instance, can’t cost more than the devices that are being produced.

But rather than limiting the kinds of nanodevices that are mass produced, these constraints can lead to greater innovation in how the devices are built, what they look like and even how they perform.

Nanomanufacturing requirements “can force researchers to think outside the box,” says Alex Liddle, group leader of the CNST’s Nanofabrication Research Group. “Instead of simply scaling an existing structure down to the nanolevel, making production expensive, or even impossible, an entirely new design may need to be invented and the device and process may no longer resemble those useful at large scales,” he says. “At the nanoscale, the combination of function and manufacturability should drive form.” Liddle and Gregg Gallatin, formerly of CNST, reviewed the successes and challenges of nanomanufacturing in the March 22, 2016 ACS Nano.

The scientists surveyed the two main approaches to nanomanufacturing—top-down and bottom-up. The top-down strategy starts by imposing structure on the system from the outside.

The top-down approach can be costly and is often best suited for manufacturing complex objects such as integrated circuits. (In photolithography, the poster child of a top-down process, light shining on a mask transfers a geometric pattern onto a silicon wafer in order to fabricate a circuit.)

The bottom-up approach, in contrast, begins with molecular-scale components that already contain the information needed to guide their assembly. In this strategy, the orderly structure of a nanosystem is created as the tiny components come together, rather than imposed by external tools. One example is DNA self-assembly, in which strands of DNA come together on their own to create a pattern that is complex in both structure and function.

To gauge progress in nanomanufacturing, the researchers defined a new term, manufacturing complexity. Complexity includes such factors as the amount of information required to build a device, the minimum size of components, the distance over which these components must be aligned or correlated in order for the device to function, and the maximum allowed fraction of components that can be defective in a particular nanomanufacturing process.

The numerical value of nanomanufacturing complexity can vary widely. For instance, only a few kilobytes of information are needed to specify a titanium oxide nanoparticle, defining its composition, size and acceptable size distribution. The permissible fraction of defective particles may be as high as a few percent.

In contrast, the computer-aided design data required to make an integrated circuit—an example of a top-down manufacturing strategy—demands many billions of bits of information and no more than one component in a trillion can be defective.

Typically, as complexity increases, so does the price the product commands and its ability to perform sophisticated tasks. In comparing nanomanufacturing complexity with the price-per-unit area of a product, Liddle and Gallatin found, as expected, that sophisticated information, storage and transmission products command a high price...
while products involving bulk materials and relatively simple structures were cheap. Of course, there are outliers – a gold bar is simple to specify, but intrinsically expensive, while a secret may be worth far more than the paper and ink used to write it down. To their surprise, the researchers also found that few nanosystems occupy the middle ground, with an intermediate degree of complexity and function.

A successful nanomanufacturer must balance technical constraints such as energy consumption and production rate with cost-related factors such as profit margin and equipment depreciation, but often these factors are poorly estimated, Liddle and Gallatin note. In addition to such challenges, a breakthrough in basic understanding that leads to a new design that is easy to make can suddenly transform a laboratory curiosity into a potential product.

But the most exciting prospect, the researchers maintain, is the ability to create dynamical nanoscale systems, which can react and adapt to a changing environment. Whether scientists ultimately achieve this by learning how to control and engineer biological systems directly (by harnessing DNA and cellular activity, for example) or by building systems based on biological examples, it “will undoubtedly be disruptive and quite probably revolutionary,” the researchers concluded.

ON DISPLAY: RESEARCHERS CONTRIBUTE TO THE ART OF SCIENCE

Art and science melded in a recent exhibit, “MICRO/MACRO: Big Images of Small Things from NIST Labs” (http://mcc.jhu.edu/news/the-art-of-science-on-display-at-nist-exhibit-at-jhu) at the 9605 Gallery, part of the Johns Hopkins University Montgomery County Campus. Two researchers from the CNST had their science images on display at the show, which ran from September 6 to November 11.

CNST project leader John Unguris says he didn’t intend to create an “arty” image. In this instance, nature just provided one. His kaleidoscope-like image portrays a glassy metal typically used in electrical transformers that reduce voltages to household levels. Unguris recorded the image using Scanning Electron Microscopy with Polarization Analysis (SEMPA), a technique he and his colleagues developed that can see magnetic features as small as 10 nanometers—about the width of 20 silicon atoms. SEMPA reveals the underlying magnetic structure of a material by measuring the direction of its electrons’ spins, with different colors signifying the different directions of spin. The larger bands of color represent desirable magnetic structures, while the “noise” in the upper right corner indicates defects that waste energy. Researchers made these measurements so that they could identify and find ways to reduce the sources of these defects.

Bharath Natarajan, formerly of the CNST, believes creativity and aesthetics aren’t just for artists—that scientists can use aesthetics to understand their own work better, too. His submission to “MACRO/MICRO,” which was also the cover of the spring CNST News, is a 3-D reconstruction of carbon nanotubes suspended in an epoxy, obtained by electron tomography. Credit: CNST

Left: Image shows a glassy metal typically used in electrical transformers that reduce voltages to household levels. The image was recorded using scanning electron microscopy with polarization analysis. Right: 3-D reconstruction of carbon nanotubes suspended in an epoxy, obtained by electron tomography. Credit: CNST

Clumped together. Natarajan and his colleagues’ research revealed that the more clumped the nanotubes are, the better they conduct heat and electricity. Moreover, the physical contact causes the nanotubes to conform to one another, which straightens them and makes them stronger. Knowing these properties and how and why they change is important when incorporating carbon nanotubes into new materials. Often, Natarajan says, visualizing data can convey more information than simply listing numbers, and, if the images are also aesthetically pleasing, they’re more likely to have an impact.
The rolled-up sheets of carbon atoms known as carbon nanotubes bestow a dazzling array of electrical, thermal and optical properties when they are inserted into polymer nanocomposites. But in order for the nanotubes to weave their magic, they must be spread out in the composite according to a precisely prescribed arrangement.

In a factory setting, engineers have neither the time nor the money to halt production while they directly investigate the nanoscale distribution of carbon nanotubes. Instead, researchers have been searching for an indirect, noncontact method that could rapidly assess—without interrupting production—whether or not a carbon nanotube nano-composite has the desired internal structure.

Researchers at the CNST have now demonstrated that a simple macroscopic measurement, using a microwave cavity, can accurately assess the distribution of carbon nanotubes within a polymer nanocomposite. Importantly, the technique can be incorporated into roll-to-roll production (http://www.nature.com/articles/srep17019) and quality control without slowing the manufacturing process of carbon nanotube composites.

“We are one step closer to overcoming a major hurdle in the mass production of these materials,” says Alex Liddle, leader of the CNST Nanofabrication Research Group. Liddle and his colleagues, including Bharath Natarajan of NIST’s Material Measurement Laboratory, describe their findings in the November issue of Carbon.

In their experimental setup, the researchers employed a tube-shaped cavity whose dimensions permit a particular set of microwave frequencies to bounce back and forth without dissipation. At these so-called resonant frequencies, a standing wave—a pattern shaped like a sine wave—fills the cavity (Orloff, Nathan D., et al. IEEE Transactions on Microwave Theory and Techniques 62.9 (2014): 2149-2159).

When a nanocomposite sample is inserted into the cavity, it disrupts the standing wave pattern, shifting or perturbing the frequency of the radiation, much as a finger on a guitar string changes note. The shift in resonant frequency, along with the damping ratio of the cavity (a measure of how quickly oscillations decay after a disturbance), is used to calculate the sample’s electrical conductivity.

But what can electrical conductivity reveal about the composition and microstructure of a nanocomposite? Earlier studies had indicated that conductivity was correlated with the amount and spatial arrangement of carbon nanotubes but a comprehensive study had never been conducted.

To do so, the researchers directly analyzed the detailed dispersion and distribution of the carbon nanotubes at length scales spanning seven orders of magnitude (a tenth of a nanometer to a millimeter), using three standard laboratory methods that would be too disruptive and costly to employ in a factory: transmission electron microscopy, scanning gallium-ion microscopy and small-angle neutron scattering. After compiling these ground-truth measurements, the scientists compared the findings to the electrical conductivity measurements.

The team found that the conductivity technique, formally known as the resonant microwave cavity perturbation (RCP) method, is indeed an accurate indicator of the distribution of carbon nanotubes in a nanocomposite—good enough that the method can stand alone, without the need for microscopy or other measurement methods that can’t be used in a manufacturing setting.

“We’ve shown here that this microwave cavity measurement is viable for online process monitoring and quality control of CNT composites,” says Natarajan. “We believe that this technique may be extended to many other hybrid materials whose ultimate functionality is determined by the spatial arrangement of their constituents,” he adds. “This will, however, require a detailed understanding of the relationships between properties, structure and processing.
Laboratory researchers have all experienced the frustration of trying to replicate an exciting result only to find that some essential point is missing from a process recipe. Part of the problem is that the gory details are often too lengthy to print in full in the relatively short manuscripts published in journals, notes Alex Liddle, leader of the CNST’s Nanofabrication Research Group. Yet the details are crucial, he adds, for anyone trying to build a complex device or work with a new processing technique.

The CNST has now published online the first in a series of articles describing the step-by-step procedures for building devices fabricated at the CNST’s NanoFab. And, since scientists want to know why they should follow a specific set of instructions, these papers also take the time to describe the reasons behind the choice of design, materials and chemistries used. The first of these comprehensive recipes, which detail such techniques as the process for rapid prototyping of nanofluidic slits in a silicone bilayer (https://www.ncbi.nlm.nih.gov/pmc/articles/PMC4730671/), appears in the Journal of Research of the National Institute of Standards and Technology.

The published recipes will enable NanoFab users and other researchers to easily access the extensive knowledge base assembled at the CNST.

“These articles provide an invaluable resource for external users at NIST because the recipes are clear, detailed, and tool-specific,” says senior engineer Juraj Topolancik of Roche Sequencing Solutions in Pleasanton, Calif. More recipes will follow soon, so watch this space.

From photomask to finished fluidic device: Top: A photomask with patterned arrays of channels, inlets, and outlets, visible as transparent glass features on an opaque chromium field for exposing a negative-tone photoresist. Bottom: Final assembled device, consisting of a pair of channels. For this particular device, each inlet hole and outlet hole punches through the terminus of a linear microfluidic channel, which intersects with a circular microfluidic reservoir. Credit: CNST


CNST WELCOMES NEW DEPUTY DIRECTOR

For James Kushmerick, who became the deputy director of CNST on October 3, joining the Center “feels like I’m getting back to my roots.”

In 1994, Kushmerick was finishing up a bachelor’s degree in chemistry at the University of Delaware when he heard a talk that changed his career path. A researcher visiting the chemistry department, Paul Weiss of Penn State, extolled the wonders of using scanning tunneling microscopy to study individual atoms and molecules on surfaces.

“It was the first time that I had been exposed to those ideas, and it was just really kind of amazing,” recalls Kushmerick. “I had to play in that field, it was just so cool,” he says. Cool enough that instead of pursuing a course of study in analytical chemistry, he chose to earn his Ph.D. with Weiss, combining chemistry with his newfound interest in nanotechnology.

By the time Kushmerick joined NIST’s Surface and Microanalysis Science Division in 2005 as a chemist studying how groups of molecules might be used as nanoscale electronic components, he had conducted research in nanoscience at both Sandia National Laboratory and the Naval Research Laboratory. In 2013, he became deputy chief of NIST’s Materials Measurement Science Division, which covers a wide breadth of research but has only a small program in nanoscience.

With the new appointment at CNST, “I’m excited to be focusing on nanotechnology again,” he says. “It’s not only going to challenge me to be working on nanoscience and nanotechnology in this bigger arena, it will be wonderful to work with all the great people at CNST.”

When he’s not pondering the nanoscale, Kushmerick scales the rocks at nearby Sugarloaf Mountain and Seneca Rocks in West Virginia. This summer he went rock climbing and hiking at Yosemite and Glacier National Park with his girlfriend and his three children, ages 16 to 19.

A NEW (ROOM-TEMPERATURE) WAY TO DEPOSIT ITO

Electrically conducting, optically transparent and easy to apply in a thin layer: These properties have made indium tin oxide (ITO) an ideal coating for liquid crystal, flat panel and plasma displays, as well as organic LEDs, solar cells and electromagnetic shielding. ITO films also help defrost airplane windshields.

The standard techniques of depositing the films, such as magnetron sputtering, work well but require the application of heat. Before heating, the ITO coating has an amorphous structure and is mostly opaque and electrically insulating. Only after the temperature is raised to 350 °C does the coating become polycrystalline, transparent and conducting.

The CNST NanoFab has now developed a new deposition process, using an ion beam, which creates transparent and conducting films without the need for heating. A clear advantage when ITO has to be deposited on heat-sensitive materials such as any plastic or organic substance, the process uses a 500-volt neutralized argon ion beam that bombards an ITO target. The energetic argon atoms eject indium, tin and oxygen atoms, which travel in a direct line of sight to the substrate. At room temperature, the process creates a polycrystalline ITO film, transparent and conductive. The deposition geometry, coupled with a lower deposition pressure, also improves the uniformity and roughness of the ITO film when compared to magnetron sputtering. The technique is now available to users in the NanoFab.
When someone asks high school student Shraeya Madhu what she did on her summer vacation, she can tell them she was at the CNST, calibrating a state-of-the-art spectrometer in order to explore new ways to study the eye pigment melanopsin. College student Kimi Bourland used the CNST’s focused ion beam microscope to better understand the dynamics of lithium-ion batteries.

These young researchers were among the six Summer Undergraduate Research Fellowship (SURF) students and two high school students who joined the CNST this July and August to gain hands-on experience with the latest nanofabrication and nanomeasurement techniques. Other summer projects included the exploration of a defect in diamonds to detect magnetic fields near nanostructures and a study of new ways to fabricate nanostructures that are tall and skinny.

NIST and the National Science Foundation jointly sponsor the SURF program.
**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. Alternatively, you may request a subscription via email or phone.

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**CNST NANOFAB HOLIDAY HOURS**

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*SUPPORTING THE DEVELOPMENT OF NANOTECHNOLOGY FROM DISCOVERY TO PRODUCTION*

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