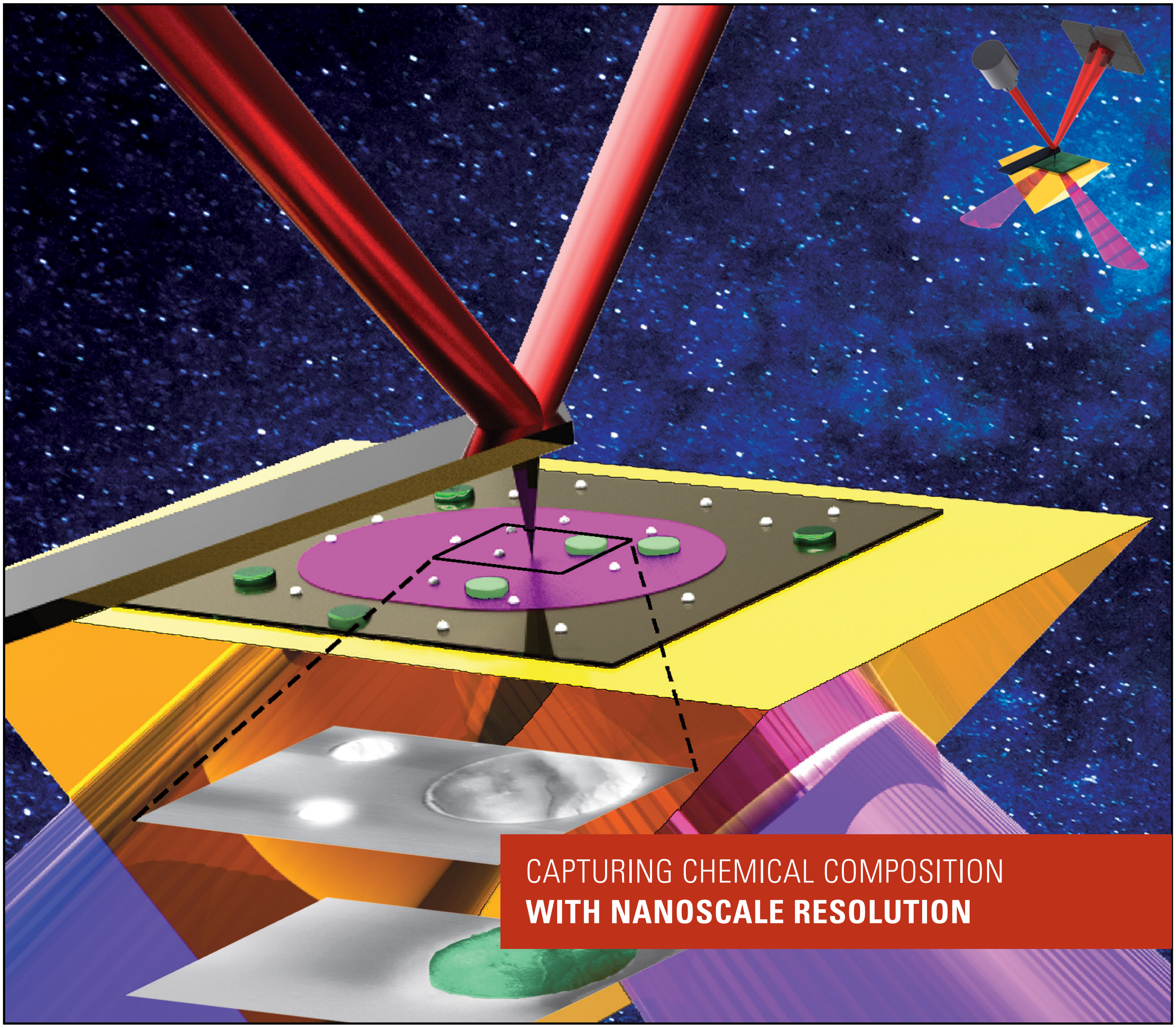
THE CNST NEWS

WINTER 2013



CAPTURING CHEMICAL COMPOSITION
WITH NANOSCALE RESOLUTION

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NIST
National Institute of Standards and Technology
U.S. Department of Commerce

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FROM THE DIRECTOR

In this issue of the *CNST News,* we focus primarily on recent accomplishments in the CNST *NanoLab*.

As NIST's nanotechnology user facility, the CNST enables innovation by providing rapid access to the tools needed to make and measure nanostructures. And we provide these tools to anyone who needs them, both inside and outside NIST, with a particular emphasis on helping industry. We provide access in two ways. In our *NanoFab*, researchers can access a commercial state-of-the-art tool set at economical hourly rates, along with help from our dedicated, full-time technical support staff. In our *NanoLab*, researchers can access the next generation of tools and processes through collaboration with our multidisciplinary research staff, who are developing new measurement and fabrication methods in response to national nanotechnology needs.

Because nanotechnology is evolving very rapidly, we operate the *NanoLab* in a way that ensures maximum agility, with a core cadre of multidisciplinary project leaders and a large cohort of postdoctoral researchers. Currently, our *NanoLab* research is focused in three technical areas – nanomanufacturing and nanofabrication, energy, and future electronics.

The work in the *NanoLab* is at once cutting-edge and highly relevant to current nanotechnology needs. For example, as described on the opposite page, CNST project leader Andrea Centrone has demonstrated that a new infrared spectroscopy technique can simultaneously measure a material's chemical composition and its physical structure with nanometer-scale precision. The approach solves a longstanding goal of many researchers to see chemical composition on this length scale, which is nearly a thousand-fold improvement over standard commercial IR microscopy systems, and can be readily applied to a wide range of material systems of commercial importance.

Pages six and seven highlight recent *NanoLab* accomplishments in nanophotonics, where Kartik Srinivasan, Vladimir Aksyuk, and their collaborators are developing new ways to make and measure nanoscale optical structures for information processing, sensing, and metrology. These novel nanosystems exploit light-matter interactions at the smallest scale for both fundamental and practical applications, including optical frequency shifting for photonic quantum information science; more sensitive and smaller sensors for atomic force microscopes; and highly versatile light and microwave frequency converters made by coupling optical and mechanical systems that can be fabricated on-chip.

We invite you to bring your nanotechnology problems to our *NanoLab*, and we will look for ways to solve the problem together through collaborative development of innovative nanoscale measurement and fabrication solutions.

Robert Celotta

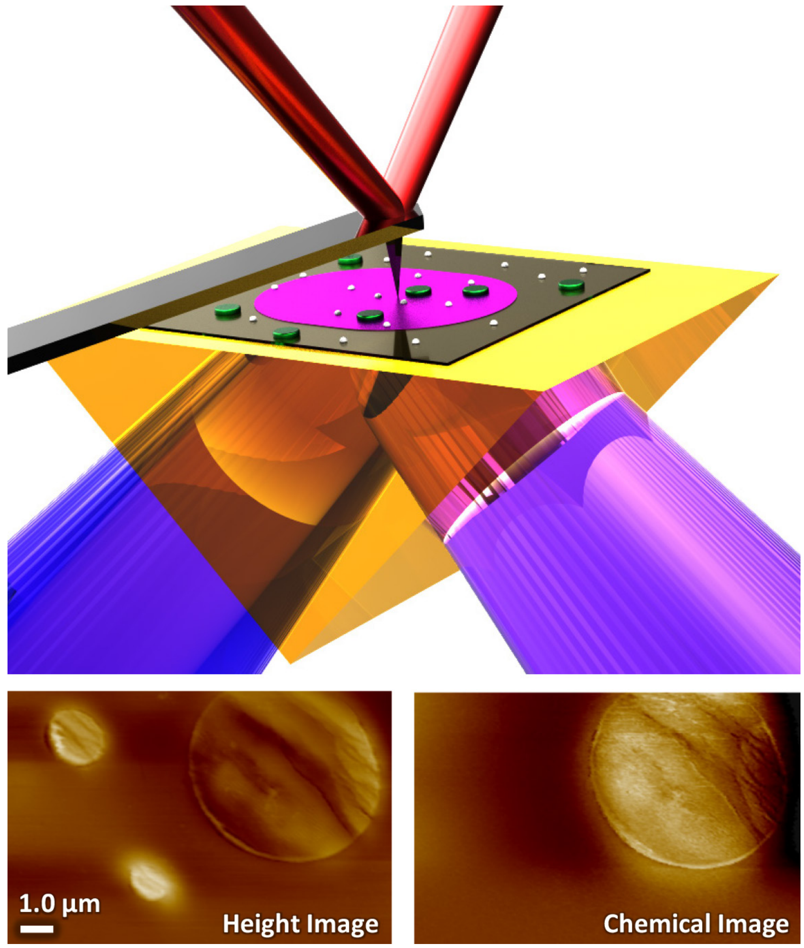
NEW METHOD CAPTURES CHEMICAL COMPOSITION WITH NANOSCALE RESOLUTION

Researchers from the CNST and the University of Maryland have demonstrated that a new spectroscopy technique can simultaneously measure a material's topography and chemical composition with nanometer-scale spatial resolution.

While infrared (IR) microscopy has been used since the 1950s to determine the chemical composition of materials, the spatial resolution of the technique has been limited to tens of micrometers; the new approach overcomes this limitation, improving the spatial resolution by a factor of about a thousand while retaining the high chemical specificity of IR spectroscopy.

The technique, called photothermal induced resonance (PTIR), was demonstrated first at the University of Paris-Sud in France. It uses a tunable infrared laser and an atomic force microscope (AFM) to extract chemical information with nanometer-scale spatial resolution. Every material has a unique infrared spectrum that acts like a chemical fingerprint, and as the laser scans across its surface, the sample at each point absorbs IR light at wavelengths that are determined by the chemical composition at that spot. The absorbed light heats the material, causing it to expand ever so slightly, which can be detected by the AFM. Repeatedly scanning the sample at different wavelengths reveals the sample's underlying chemical composition with a resolution determined by the AFM tip size and the sample's thermo-mechanical properties, resulting in a resolution many times smaller than IR microscopy, which depends on the wavelength of the light. Since the properties of nanomaterials depend on their shape, size and chemical composition, the team's work gives researchers a powerful tool to measure some of the key characteristics of their creations.

For the first time, the CNST researchers have demonstrated that PTIR can be used to generate a signal that is proportional to the energy absorbed by a material, a necessary step for accurately measuring the relative amounts of different chemicals within. The scientists fabricated samples with features having different chemical compositions as narrow as 100 nm wide and as thin as 40 nm. Using PTIR, they were able to distinguish the different chemical components down to the smallest features, suggesting that even better resolution may be achievable.



The sample, made of polystyrene (white) and polymethylmethacrylate (green), absorbs infrared laser light (purple) at wavelengths determined by its chemical composition, causing it to expand, which deflects the AFM cantilever. **Bottom left:** The AFM detects the height of two small polystyrene particles and a large polymethylmethacrylate particle. **Bottom right:** The light is tuned to be absorbed only by PMMA but not by polystyrene. Combining the data and recording chemical images at different wavelengths produces a map of the surface's topography and chemical composition.

"What's extraordinary is that we can see that the chemical map is not necessarily correlated to the height or size of the physical features on the sample surface," says Andrea Centrone, a scientist from the University of Maryland's Institute for Research in Electronics and Applied Physics working in the CNST. "We get independent details about both the surface's physical features and its chemical properties. This result is unmatched by other near-field techniques."

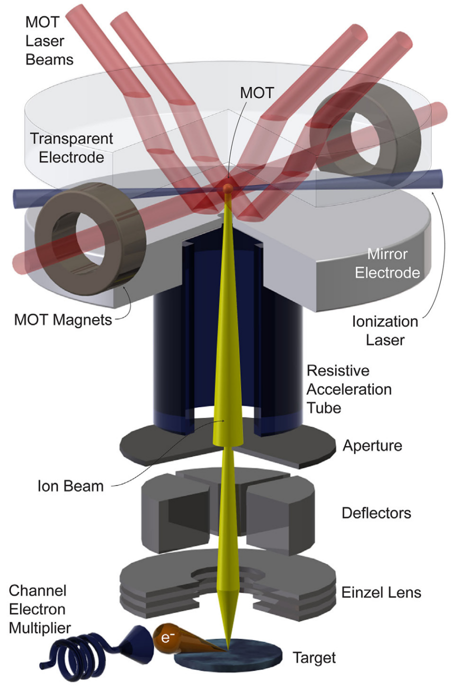
Chemical imaging beyond the diffraction limit: experimental validation of the PTIR technique, B. Lahiri, G. Holland, and A. Centrone, *Small* **9**, 439–445 (2013).

RESEARCHERS PATENT NEW ION ACCELERATION SYSTEM

Researchers from the CNST and FEI Co. have been granted a patent for an innovative method to extract and accelerate ions generated by a spatially distributed ion source such as their previously patented magneto-optical trap ion source (MOTIS). The combination of the two inventions allows a wide range of previously unavailable heavy and light ions to be used by existing focused ion beam (FIB) systems, enabling new applications from nanoscale imaging and defect metrology to ion implantation and material modification.

To make the best possible use of the high-intensity collimated ions emitted by the MOTIS, it is desirable to accelerate the ions to a high energy with minimal convergence or divergence. Using a pair of disk-shaped extraction plates spanned by a long resistive tube, the new ion optical system allows ions to be accelerated gradually up to 30 keV or more with minimal lensing. This system preserves the collimation of the beam and avoids the creation of regions with very high current density could that lead to strong ionic interactions that would cause loss of resolution.

The invention has been successfully integrated into a commercial FIB system and has demonstrated imaging performance competitive with the liquid metal ion sources used in most commercial FIBs.



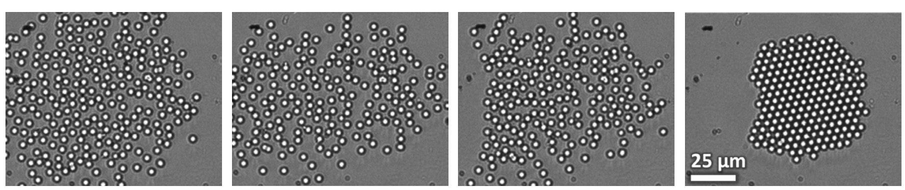
Schematic of a distributed ion source acceleration column and a magneto-optical trap ion source. The ion source acceleration column uses a pair of disk-shaped extraction plates connected to a long resistive tube to accelerate ions gradually up to full beam energy with minimal lensing. The system preserves the collimation of the beam and prevents strong ionic interactions that could reduce resolution.

United States Patent: 8314404 - Distributed ion source acceleration column, J. J. McClelland, B. J. Knuffman, A. V. Steele, and J. H. Orloff, US Patent No. 8314404.

CONTROLLING PARTICLES FOR DIRECTED SELF-ASSEMBLY OF COLLOIDAL CRYSTALS

Researchers from the CNST and the Johns Hopkins University have developed a technique to reliably manipulate hundreds of individual micrometer-sized colloid particles to create crystals with controlled dimensions. The accomplishment is an important milestone for understanding how to direct and control the assembly of microscale and nanoscale objects for nanomanufacturing applications.

The experiment uses four electrodes patterned on a microscope coverslip to move the micrometer-sized particles suspended in liquid by applying a combination of AC and DC electric fields. Using a nonuniform, high-frequency AC field, the dielectrophoretic forces exerted on the dielectric particles are tuned to adjust the strength of their attraction to a collection area in the center of the electrodes. When these forces are low enough, electrophoretic-electroosmotic flows induced by applying a DC field allow the researchers to selectively remove particles from the area and trim the particle assemblies to a chosen size and shape.



Optical micrographs showing the assembly of a 2D colloidal crystal composed of approximately 200 particles. **Left:** The initial configuration is a fluid weakly held by negative dielectrophoresis. **Second and third images:** Particles are then iteratively removed and concentrated by cycling between electrophoretic-electroosmotic actuation and negative dielectrophoretic actuation using feedback control. **Right**: Finally, negative dielectrophoresis is increased to compress particles into quasi-2D colloidal crystals.

By independently varying the AC and DC electrode potentials, the researchers can direct the self-assembly of two-dimensional (2D) rafts made of precise numbers of particles; i.e., 2D colloidal crystals. Once the desired crystal size is reached, the attractive forces holding the particles in the collection area are increased to stabilize the structure. An important component of this work is the application of a computer vision-based, real-time feedback system that dynamically adjusts the AC and DC fields to automate the directed assembly process.

This work shows how the combination of multiple actuators offers extra degrees of freedom that can be used to manipulate ensembles of colloidal components to create desired sizes and shapes. The researchers are now developing measurement methods sensitive enough to track nanometer-scale structures that will allow these methods to be extended to control the assembly of nanoscale materials.

Multiple electrokinetic actuators for feedback control of colloidal crystal size, J. J. Juárez, P. P. Mathai, J. A. Liddle, and M. A. Bevan, *Lab on a Chip* **12**, 4063–4070 (2012).

DNA AND QUANTUM DOTS: ALL THAT GLITTERS IS NOT GOLD

Nanotechnology researchers have long been frustrated trying to control the light emitted from quantum dots, which get brighter or dimmer in proximity to other particles. Now a team of researchers from the CNST and the University of Maryland has shown that by bringing gold nanoparticles close to the dots and using a DNA template to control the distances, the intensity of a quantum dot's fluorescence can be predictably increased or decreased. This breakthrough opens a potential path to using quantum dots as a component in better photodetectors, chemical sensors, and nanoscale lasers.

The team's research consists of two parts: first, developing ways to accurately and precisely place different kinds of nanoparticles near each other and second, measuring the behavior of the resulting constructs. The researchers looked at two types of nanoparticles: quantum dots, which glow with fluorescent light when illuminated; and gold nanoparticles, which have long been known to enhance the intensity of light around them. The two could work together to make nanoscale sensors built using rectangles of woven DNA strands, formed using a technique called "DNA origami," in which DNA is folded to make desired shapes — in this case, rectangles.

These DNA rectangles can be engineered to capture different types of nanoparticles at specific locations, using lock-and-key type chemical interactions, with a precision of about one nanometer. Tiny changes in the distance between a quantum dot and a gold nanoparticle near one another on the rectangle cause the quantum dot to glow more or less brightly as it moves away from or toward the gold. Because these small movements can be easily detected by tracking the changes in the quantum dot's brightness, they can be used to reveal the presence of a particular chemical that is selectively attached to the DNA rectangle, for instance. However, getting it to work properly is complicated.

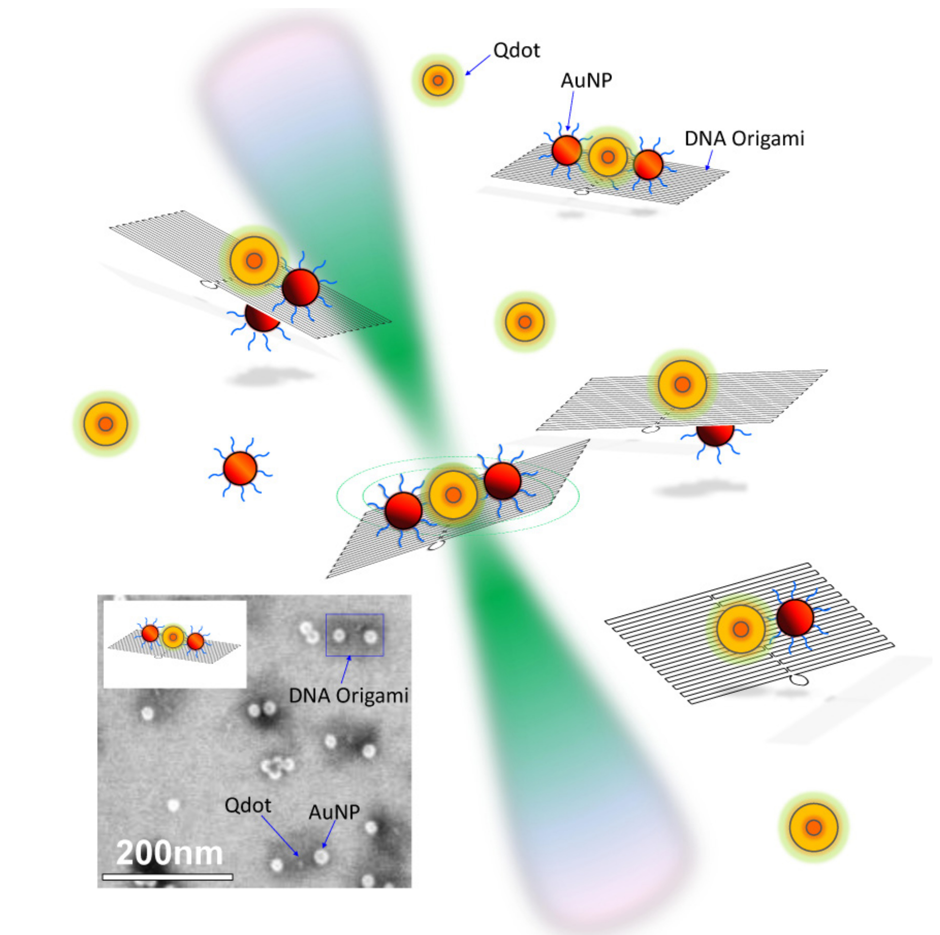
"A quantum dot is highly sensitive to the distance between it and the gold, as well as the size, number and arrangement of the gold particles," says CNST Nanofabrication Research Group Leader Alex Liddle. "These factors can boost its fluorescence, mask it, or change how long its glow lasts. We wanted a way to measure these effects, which had never been done before."

The team made several groups of DNA rectangles, each with a different configuration of quantum dots and gold particles in a solution. Using a laser as a spotlight, they could follow the movement of individual DNA rectangles in the liquid, and also could detect changes in the fluorescent lifetime of the quantum dots when they were close to gold particles of different sizes.

They also showed that using a mathematical model they could exactly predict the lifetime of the fluorescence of the quantum dot depending on the size of the nearby gold nanoparticles.

While their tracking technique was time consuming, Liddle says that the strength of their results will enable them to engineer the dots to have a specific desired lifetime. Moreover, the success of their tracking method could lead to better measurement methods.

"Our main goals for the future," he concludes, "are to build better nanoscale sensors using this approach and to develop the metrology necessary to measure their performance."



The researchers explored the behavior of quantum dots and gold nanoparticles placed in different configurations on small rectangular constructs made of self-assembled DNA (see inset for scanning electron micrograph). Laser light (green) allowed the team to explore changes in the fluorescent lifetime of the quantum dots when close to gold particles of different sizes.

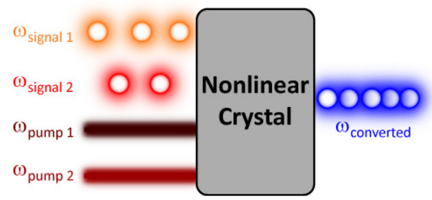
Quantum-dot fluorescence lifetime engineering with DNA origami constructs, S. H. Ko, K. Du, and J. A. Liddle, *Angewandte Chemie International Edition* **52**, 1193–1197 (2013).

PHOTONS FROM QUANTUM DOTS MADE INDISTINGUISHABLE THROUGH QUANTUM FREQUENCY CONVERSION

An international collaboration led by researchers from the CNST has demonstrated the ability to make photons emitted by quantum dots at different frequencies identical to each other by shifting their frequencies to match. This "quantum frequency conversion" is an important step for making solid-state, single photon sources, including quantum dots, more useful light sources for photonic quantum information science. The team included researchers from the CNST, the University of Maryland, the University of Rochester, and Politecnico di Milano, Italy.

Quantum dot sources are desirable due to their high brightness, stability, and amenability to scalable fabrication technology, but frequency variations arising from nonuniform device fabrication have limited their usefulness. Previous research has focused on tuning the sources themselves, for example by inducing strain or by varying the electrical and optical fields surrounding the structures. In their new approach, the CNST-led team uses a fundamentally different approach – manipulating the photons after they are generated, rather than altering the sources.

As reported in the October 5, 2012 issue of *Physical Review Letters* and featured in the November issue of *Physics Today*, the experiment uses photons emitted from a semiconductor quantum dot at two different frequencies which are determined by two of the dot's different energy states. The team demonstrated that the photons can be converted to the same frequency, or color, using quantum frequency conversion, a process in which each a single photon stream is combined with light from a much stronger pump laser in a nonlinear crystal which outputs photons at a frequency that is the sum of the two inputs. Two pump lasers are used, with the frequency difference between them set to match the difference between the two input photon streams, causing the new photons to be at exactly the same frequency. An interference measurement is used to confirm that the frequency-converted photons have become identical. Since the current work uses relatively large nonlinear crystals for frequency conversion, future work will focus on implementing the conversion method in smaller and more scalable device architectures.



The quantum frequency conversion system uses two pump lasers whose frequency difference is matched to the difference between the two input photon streams, causing the new photons from a nonlinear crystal to be emitted at exactly the same frequency.

Two-photon interference using background-free quantum frequency conversion of single photons emitted by an InAs quantum dot, S. Ates, I. Agha, A. Gulinatti, I. Rech, M. T. Rakher, A. Badolato, and K. Srinivasan, *Physical Review Letters* **109**, 147405 (2012).

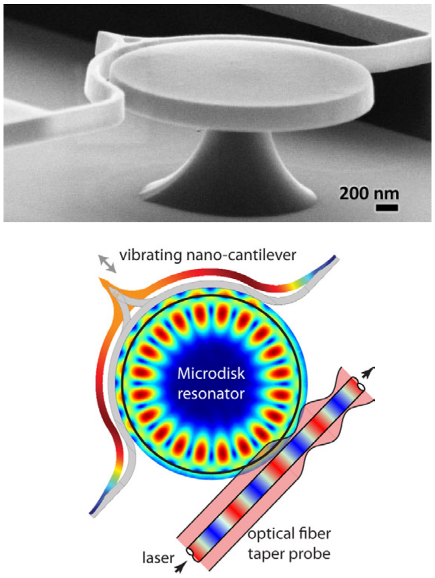
RESEARCHERS DEVELOP VERSATILE OPTOMECHANICAL SENSORS FOR ATOMIC FORCE MICROSCOPY

Researchers at the CNST have developed on-chip optomechanical sensors for atomic force microscopy (AFM) that extend the range of mechanical properties found in commercial AFM cantilevers, potentially enabling the use of this technology to study a wide variety of physical systems. AFM is an important tool for surface metrology that measures local tip-surface interactions by scanning a flexible cantilever probe over a surface. Unfortunately, the bulky free-space optical system commonly used to sense the motion of the probe imposes limits on the tool's sensitivity and versatility.

Previously, the NIST team had demonstrated an alternate, chip-scale sensing platform with a more versatile readout approach in which a nanocantilever probe was integrated with interferometric motion detection provided by a low-loss optical resonator that can be coupled through fiber optics to standard optical sources and detectors. This approach achieved remarkable displacement sensitivity. In the previous work, the cantilever spring constant, or stiffness, was fixed at a moderate value; however, in other applications, the spring constant may need to be much smaller (for studying soft materials or for weak force detection) or much larger (for high-resolution imaging). Ideally, this range of spring constants would be achieved without sacrificing displacement sensitivity or response time.

In the current work, the authors show that geometric scaling of both the cantilever and the optical resonator dimensions can enable a variation in the cantilever spring constant by over four orders of magnitude, ranging from devices that are ten times softer than the original design to ones that are one thousand times stiffer. Importantly, these cantilevers maintain their high displacement sensitivity and achieve measurement response times that are hundreds of times faster than commercial cantilevers with similar spring constants.

Future work will focus on integrating this sensor platform into a commercial AFM system.



Top: Scanning electron micrograph of the chip-based optomechanical sensor, which was built in the CNST NanoFab using electron beam lithography and silicon reactive ion etching. **Bottom:** Schematic of the disk-cantilever sensor geometry with a superimposed simulation of the optical and mechanical modes shown in color.

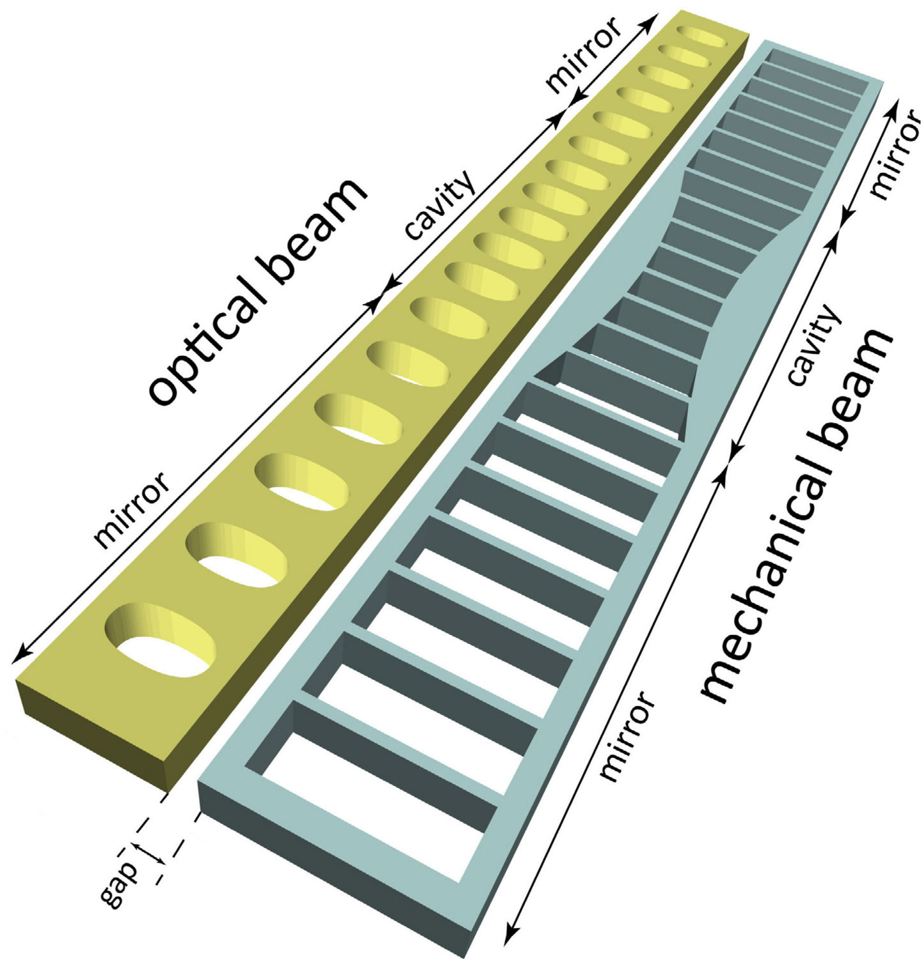
Wide cantilever stiffness range cavity optomechanical sensors for atomic force microscopy, Y. Liu, H. Miao, V. Aksyuk, and K. Srinivasan, *Optics Express* **20**, 18268–18280 (2012).

NEW PLATFORM MEASURES AND EXPLOITS OPTOMECHANICAL INTERACTIONS

Researchers from the CNST and CalTech have developed a new design platform for measuring and exploiting strong interactions between light confined in a nanoscale structure and an adjacent nanomechanical system.* The versatile platform opens new approaches for fabricating sensitive light detectors and for converting wavelengths for use in quantum information science. Previously, the Caltech team used silicon "optomechanical crystals" in which radiation pressure from light drove mechanical vibrations within a single, doubly-clamped silicon nanobeam. In the new work, the CNST-Caltech collaborators developed a design for observing similar effects in silicon nitride, which has a much broader optical transparency window than silicon, but for which radiation pressure interactions within a single nanobeam are expected to be much weaker.

The new approach uses a pair of nanobeams held side-by-side and separated by a nanoscale gap, with one beam supporting a mechanical mode which has a specific vibration pattern and frequency, and the other supporting a confined optical mode which has a specific spatial field distribution and optical frequency. Crucially, while the properties of the optical mode are largely controlled by only one of the two beams, it is concentrated in the small region in-between the beams, ensuring that the radiation pressure interaction with the mechanical mode in the other beam is strong. Electromagnetic simulations show that the optomechanical interaction strength increased by nearly a factor of three relative to the single beam case.

An important aspect of the new platform is that it enables near-independent design of the optics and the mechanics, so a wide range of new functionalities may be possible. In particular, the team is working on geometries in which two optical beams operating at widely different wavelengths are coupled to a mechanical beam held in-between them. Such devices are expected to enable wideband optical-to-optical and even microwave-to-optical frequency conversion, which would be significant advances in this field.



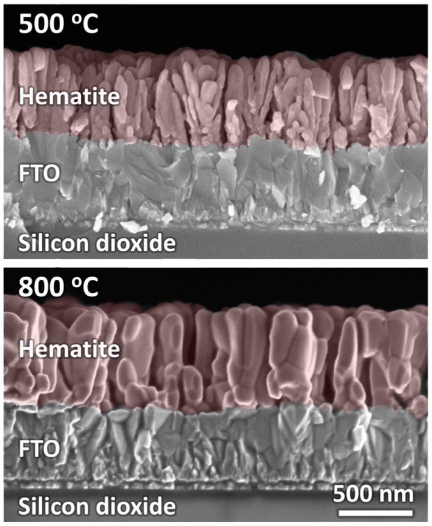
A pair of nanobeams is held side-by-side and separated by a nanoscale gap, with one beam supporting a mechanical mode, and the other supporting a confined optical mode. The properties of the optical mode of the beam on the left are concentrated in the nanoscale gap in-between the beams, ensuring a strong radiation pressure interaction with the mechanical mode in the beam on the right. The platform enables nearly independent design of the optics and the mechanics.

*Slot-mode-coupled optomechanical crystals, M. Davanço, J. Chan, A. H. Safavi-Naeini, O. Painter, and K. Srinivasan, *Optics Express* **20**, 24394–24410 (2012).

CRITICAL FACTORS DETERMINED FOR IMPROVING THE PERFORMANCE OF A SOLAR FUEL CATALYST

Hydrogen gas that is created using solar energy to split water into hydrogen and oxygen has the potential to be a cost-effective fuel source if the efficiency of the catalysts used in the watersplitting process can be improved. By controlling the placement of key additives (dopant atoms) in an iron oxide catalyst, researchers in a CNST-led collaboration have found that the final location of the dopants and the temperature at which they are incorporated into the catalyst crystal lattice determine overall catalytic performance in splitting water. The collaboration included researchers from the CNST, the NIST Material Measurement Laboratory, the University of Maryland, and Syracuse University.

The iron oxide hematite is a promising catalyst for water splitting because it is stable in water and absorbs a large portion of the solar spectrum. It is also abundant in the earth's crust, making it inexpensive. Unfortunately, pure hematite has only modest catalytic activity, falling well short of its predicted theoretical maximum efficiency.



False-color scanning electron micrographs of cross-sectioned hematite films grown by sputter deposition and then annealed at two different temperatures. The physical structure and the tin dopant atom distributions in the hematite films differ depending on the annealing temperature. Hematite annealed at higher temperatures has better catalytic performance for splitting water.

Incorporating dopants such as tin atoms into hematite's lattice improves performance, but it is a challenge to accurately measure the dopant concentration, making it difficult to understand and optimize their effects on catalyst performance.

Using thin films of hematite doped with tin, the researchers produced highly active samples that enabled them to measure and characterize the spatial distribution of dopants in the material and their role in catalysis. The researchers determined that as a result of the sample preparation protocol they followed, a dopant gradient extends from the interface with the dopant source to the catalyst surface, where the measured concentration is low compared with previous estimates from similarly prepared samples. Contrary to prior results, they found that only a small dopant concentration is needed to improve catalytic activity.

The researchers believe this study creates a path for improving the rational design of inexpensive catalysts for splitting water using solar energy.

Effect of tin doping on α-Fe_2O_3 photoanodes for water splitting, C. D. Bohn, A. K. Agrawal, E. C. Walter, M. D. Vaudin, A. A. Herzing, P. M. Haney, A. A. Talin, and V. A. Szalai, *The Journal of Physical Chemistry C* **116**, 15290–15296 (2012).

KEY PROPERTY OF GRAPHENE SUSTAINED OVER WIDE RANGES OF DENSITY AND ENERGY

A collaboration led by researchers from the CNST has shown for the first time that charge carriers in graphene continue to behave as massless particles, like photons, over wider ranges of both density and energy than previously measured or modeled. The collaboration included researchers from the CNST, the University of Maryland, Columbia University, Korea Research Institute of Standards and Science, and the National Institute for Materials Science in Japan.

Graphene, a single layer of carbon atoms, is a material of great scientific and technological interest in part because it conducts electrons at high speed. However, in order for graphene to achieve its promise as a component of future electronic devices, it is important to understand at a fundamental level how charge carriers in the material interact with each other. The researchers used scanning tunneling spectroscopy measurements of the magnetic quantum energy levels of the graphene charge carriers to determine the changes in velocity of the charge carriers. Using a CNST-developed technique called "gate mapping scanning tunneling spectroscopy," the researchers measured the energy levels as they changed the density of the carriers in the graphene by applying different potentials between a conducting gate and the two-dimensional graphene sheet. They established that the graphene carriers retain a proportional relationship between energy and momentum—a "linear dispersion" characteristic of massless particles—across an unexpectedly broad range of energies and densities, from electrons to holes. They were also able to show that when the density of carriers in graphene is lowered, the effect of each electron on other electrons increases, resulting in higher velocities than expected.

These surprising results are important both for understanding the physics of future graphene devices and because they will help guide the development of more accurate theoretical models of the interactions between electrons in two-dimensional systems.

Renormalization of the graphene dispersion velocity determined from scanning tunneling spectroscopy, J. Chae, S. Jung, A. F. Young, C. R. Dean, L. Wang, Y. Gao, K. Watanabe, T. Taniguchi, J. Hone, K. L. Shepard, P. Kim, N. B. Zhitenev, and J. A. Stroscio, *Physical Review Letters* **109**, 116802 (2012).

NEW TOOLS IN THE NANOFAB

Photoresist Stabilization System

The CNST has installed a new B&A Associates LS-200FSX deep ultraviolet (UV) flood exposure system which is now available to users for curing photoresists on substrates up to 200 mm in diameter. Located in cleanroom bay A105, the system facilitates easier resist stripping after processing, improves resist stability, and increases etch selectivity. The device uses a hotplate combined with UV flood exposure to cause polymer molecules in the photoresist to cross-link, enabling the resist to hold up better to processes such as ion beam etching, reactive ion beam etching, and ion implantation. For more information, contact Liya Yu, 301-975-4590, liya.yu@nist.gov.

Automated Spectroscopic Ellipsometer

The recently installed Woollam M-2000 spectroscopic ellipsometer in cleanroom bay B103 provides fast and accurate thin film characterization over a wide spectroscopic range. The system's high speed CCD collects data automatically in a fraction of a second at hundreds of wavelengths, ranging from infrared to deep ultraviolet, and at multiple angles. This information can be used to determine film thickness, index of refraction, and extinction coefficient on single or multilayer film stacks using the system's user-friendly modeling software. For more information, contact Marc Cangemi, 301-975-5993,marc.cangemi@nist.gov.

Laser Pattern Generator Upgrade

The CNST is installing a new Heidelberg Instruments DWL 2000 laser pattern generator in cleanroom bay A103 which is expected to be available to users by the end of March. The new system is faster and more flexible than the laser pattern generator it replaces and can expose substrates up to 200 mm x 200 mm, with feature resolution down to 700 nm. This laser pattern generator can expose photomasks for both contact and stepper lithography at speeds up to 110 mm^2 per minute, and has the ability to write patterns directly on substrates. For more information, contact Marc Cangemi, 301-975-5993, marc.cangemi@nist.gov.

The new Heidelberg Instruments DWL 2000 laser pattern generator can expose substrates up to 200 mm x 200 mm for use in the stepper or for contact lithography, and has the ability to write patterns directly on substrates. It can expose photomasks with feature resolution down to 700 nm at speeds up to 110 mm^2 per minute.

Electron Backscatter Diffraction System

A new Oxford Instruments Electron Backscatter Diffraction (EBSD) system has been integrated into the NanoFab's Helios 650 Dual Beam FIB/SEM System, located in room 216/G113. This addition increases the capability of the FIB system to provide material crystallographic information, such as crystal orientation mapping, phase identification, grain boundaries, and localized strain. For additional information, please contact Joshua Schumacher, 301-975-8065, joshua.schumacher@nist.gov.

Secondary Ion Mass Spectrometer for Ion Milling System

A new Hiden Analytical IMP 301 secondary ion mass spectrometer (SIMS) has been integrated into the NanoFab's 4Wave Ion Milling System, located in cleanroom bay A106. The module can reliably determine the etching endpoints at or near the interface of almost any two dissimilar materials, allowing users to terminate etches with better than 0.2 nm accuracy. The SIMS can be programmed for multi-step processes, and detects the composition of ion mill by-products for up to four materials at one time. For more information, contact Gerard Henein, 301-975-5645, gerard.henein@nist.gov.

Flip Chip Bonder

The CNST has purchased a Tresky T-3000-FC3-HF die bonder which will be available to users in Spring 2013. The bonder is a versatile manual tool that allows users to permanently attach chips onto semiconductor, opto-electronic, and other packages; it is a capable of eutectic, ultrasonic, thermo-compression, and epoxy bonding with 1 µm placement accuracy. For additional information, please contact Gerard Henein, 301-975-5645, gerard.henein@nist.gov.

Solvent Lift-Off Tool

Located in cleanroom bay A102, the new Modular Process Avenger heated spray solvent metal lift-off tool provides users with automatic recipe control and reduces lift-off time from hours to minutes. The tool removes unwanted target material on samples ranging from 150 mm-diameter wafers down to 25 mm-on-a-side squares. Its dry wafer in and dry wafer out processing provides cleaner sample finishing than manual lift-off processes, while reducing user exposure to solvents. For more information, contact Jerry Bowser, 301-975-8187, jerry.bowser@nist.gov.

Parametric Test Station

The CNST's new Keithley 4200 parametric test system provides users with in-line electrical characterization capabilities, including the ability to measure capacitance at multiple frequencies, and to measure both DC and ultra-fast pulsed currents and voltages. The system features a dark box for light sensitive measurements and a heated chuck to allow measurements up to 300 °C. For more information, contact Jerry Bowser, 301-975-8187, jerry.bowser@nist.gov.

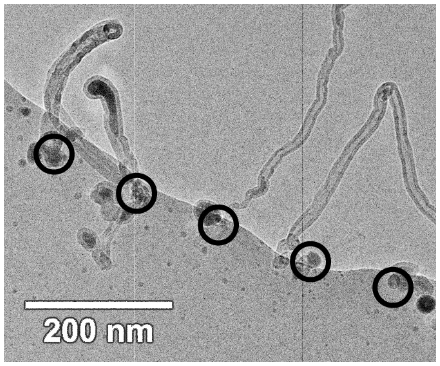
RESEARCHERS OBSERVE, CONTROL, AND OPTIMIZE THE GROWTH OF INDIVIDUAL CARBON NANOTUBES

Researchers from the CNST and Arizona State University have used an environmental scanning transmission electron microscope (ESTEM) to control the size and placement of iron nanoparticles in order to catalyze the growth of carbon nanotubes on a silicon oxide substrate. Large scale synthesis of carbon nanotubes for applications such as low-cost field emission displays (FEDs) require stringent control on nanotube length, diameter, and surface density.

Using the ESTEM, the researchers were able to visualize the placement of the catalyst nanoparticles and the growth of the nanotubes in real-time. They tested the hypothesis that the diameter of carbon nanotubes is dependent on the size of the catalyst particles by depositing iron catalyst nanoparticles of different sizes and densities on a substrate using the microscope's electron beam to induce dissociation of the iron-containing catalyst precursors.

The researchers found that a number of factors control the size and catalytic activity of nanoparticles for nanotube growth, including the choice of the precursor (ferrocene or diiron nonacarbonyl), the substrate temperature, the residence time of the precursor on the substrate, and the electron beam energy. They were able to use the deposition time to control the particle size and the position of the electron beam to control the location of the catalyst particles on the surface of the substrate. They also found that the catalytic activity of the iron particles for tube growth depends upon the amount of carbon co-deposited with the iron during the electron beam-induced deposition process, because co-deposited carbon forms graphitic shells around the iron particles. These shells made the particles chemically inactive for inducing carbon nanotube growth. This problem was solved for the diiron nonacarbonyl precursor by increasing the substrate temperature to 100 °C, which reduced the amount of co-deposited carbon.

Since heating the substrate did not affect co-deposited carbon levels in the ferrocene samples, diiron nonacarbonyl appears to be better suited as a catalytst precursor for controlled carbon nanotube growth. The researchers believe these results will help in creating substrates with carbon nanotubes at appropriate sizes and surface densities for use in FEDs and other applications.



Bright field transmission electron micrograph showing carbon nanotubes grown from an array of equally-sized iron catalyst particles (circled) created by electron beam-induced decomposition of a diiron nonacarbonyl precursor.

Controlling the size and the activity of Fe particles for synthesis of carbon nanotubes, S. W. Chee and R. Sharma, *Micron* **43**, 1181–1187 (2012).

LLOYD WHITMAN SELECTED TO CO-CHAIR THE NANOSCALE SCIENCE, ENGINEERING, AND TECHNOLOGY (NSET) SUBCOMMITTEE

CNST Deputy Director Lloyd Whitman was selected by the White House Office of Science and Technology Policy (OSTP) to serve as Agency Co-Chair of the Nanoscale Science, Engineering, and Technology (NSET) Subcommittee for a two year term effective December 1, 2012.

The NSET, which is a subcommittee of the National Science and Technology Council (NSTC) Committee on Technology in the Executive Office of the President, coordinates the planning, budgeting, program implementation, and review of the National Nanotechnology Initiative (NNI). The subcommittee is composed of representatives from the federal departments and agencies that participate in the NNI, and is co-chaired by an agency representative and a representative from OSTP. Lloyd has been an active participant in the NSET subcommittee since 2007 and has represented NIST on the NSET since 2008. As noted in his appointment letter from the OSTP, the NNI continues to be a key administration priority, with a broad range of departments and agencies participating. Twelve years into this science and technology initiative, there continues to be much exploration to be done via fundamental research, and a growing emphasis on and need for manufacturing advancements, technology transfer, and commercialization—all key NIST priorities.

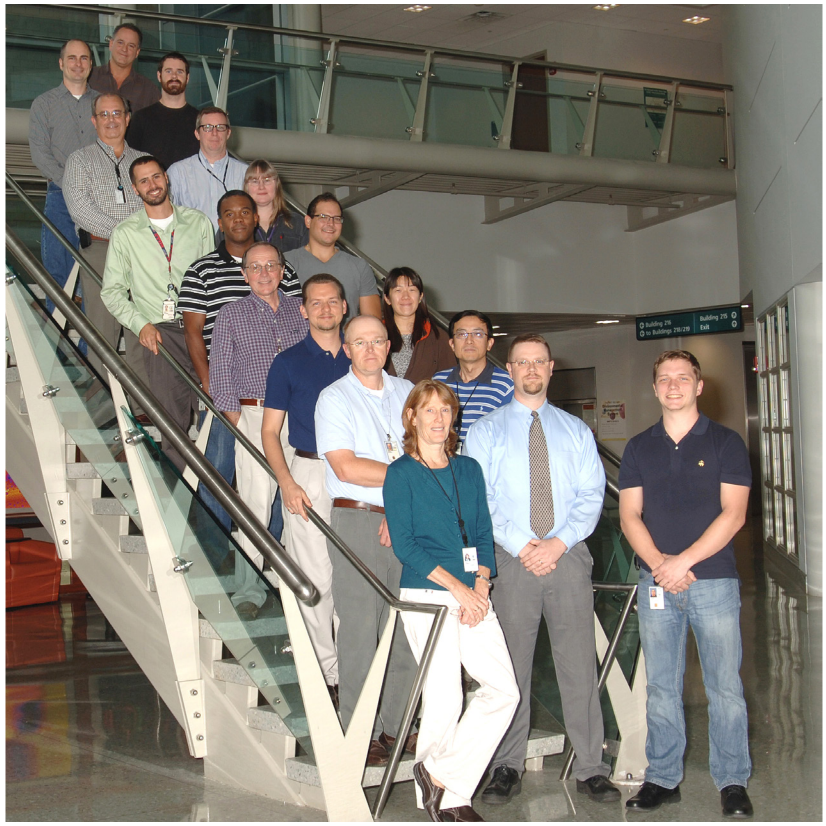
As NSET Co-Chair, Lloyd plans to work closely with OSTP and the other NSET subcommittee members to foster interagency coordination of and collaboration in nanotechnology, while advancing the goals of NIST's nanotechnology programs.

NANOFAB WINS 2012 NIST SAFETY AWARD

The CNST NanoFab has won the 2012 NIST Safety Award "for establishing and implementing an outstanding safety program in the NanoFab that protects the safety of researchers from industry, academia, NIST, and other government agencies, who range from novices to experts in nanofabrication." First established in 1979, the NIST Safety Award is granted by the NIST Safety Review Committee to recognize NIST employees and organizations for substantial contributions to improving safety at NIST.

The award was presented to the NanoFab staff, along with CNST Safety Officer Russell Hajdaj, at the 40th Annual NIST Awards Ceremony on December 5th, 2012. As recognized at the award ceremony, "there has not been a single reported injury associated with the operation or use of the NanoFab in the past three years."

During that time, NanoFab use has more than doubled, and over 400 researchers were trained and approved to access NanoFab laboratories. According to the awards ceremony booklet, "This organization has created and manages what is arguably the most diverse, complex, and challenging safety program at NIST, starting from a much simpler operation with only dozens of users five years ago."



JOSEPH STROSCIO WINS NIST STRATTON AWARD FOR DEVELOPING NEW ATOMIC SCALE MEASUREMENT METHODS

Joseph Stroscio, a Project Leader and NIST Fellow in the CNST Electron Physics Group has been awarded the 2012 Samuel Wesley Stratton Award "for scientific achievements in developing new atomic scale measurement methods involving low and ultralow temperature scanning tunneling microscopy." The Stratton Award, first presented in 1962, is granted for outstanding scientific or engineering achievements in support of NIST objectives.

The award was presented at the 40th Annual NIST Awards Ceremony on December 5th, 2012. In selecting Dr. Stroscio for the award, the The NIST Named Awards Review Panel noted that his work exemplified the mission of the CNST and NIST "to develop new measurement methods that support the U.S. nanotechnology enterprise" and that he "has used this beyond-state of-the-art equipment to conduct a series of groundbreaking experiments on graphene, a system of great interest to the nation's electronics industry as a pathway to the continued scaling of electronic devices." This work has resulted in a series of outstanding publications in *Science*, *Nature*, and *Nature Physics* that have garnered wide acclaim in the scientific community and beyond.

CNST Center for Nanoscale Science & Technology

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.

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SUPPORTING THE DEVELOPMENT OF NANOTECHNOLOGY FROM DISCOVERY TO PRODUCTION

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