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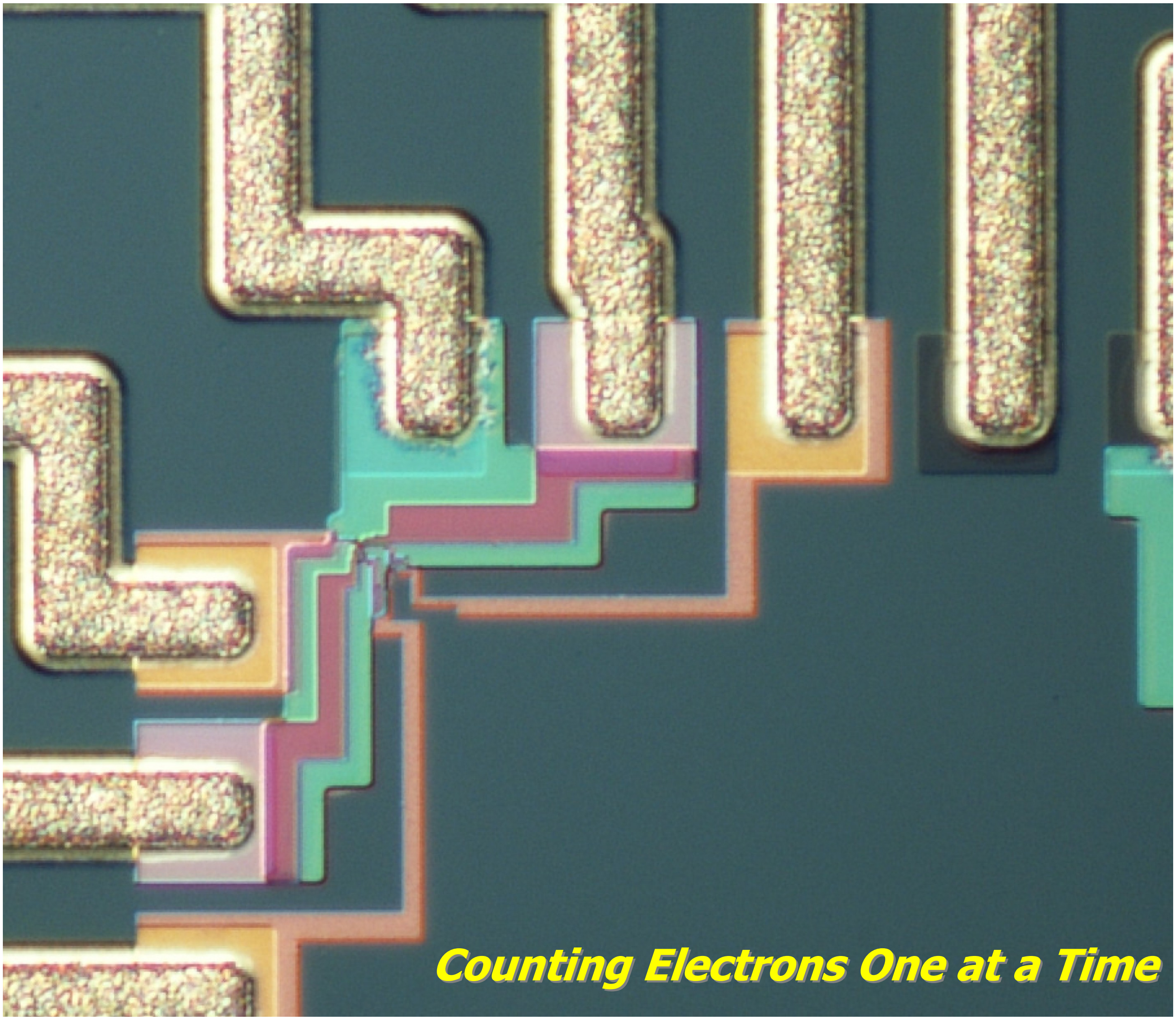
The CNST News



Counting Electrons One at a Time

Centuries-Old Mathematical Theorems Applied to Nanoscale Measurements

Efficient Piezoelectric MEMS Sensors

On-Chip Resonator Produces Ultra-Short Light Pulses

Nanowire Lithium-Ion Battery

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

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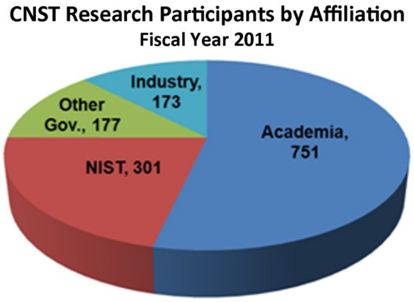
The growth in research participation is beginning to be reflected in publications, with the number of publications increasing by 54 % in 2011.

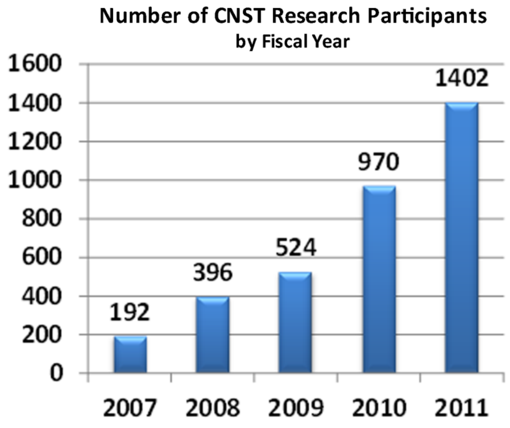
From the Director

The month of May marks the fifth anniversary of the establishment of the NIST Center for Nanoscale Science and Technology (CNST) so it seems appropriate to review how far we have come. In a relatively short time, the CNST has become a widely used and highly productive facility, serving the country as a major national resource for nanoscale measurement and fabrication methods and technology. Growing from an initial staff of about twenty in 2007, this summer we will be bursting at the seams with over 120 people, including over 50 postdoctoral and student researchers—the next generation of nanotechnologists.



The commitment of our staff combined with the growth in our measurement and fabrication capabilities has led to notable year-over-year increases in the CNST's impact. In fiscal year 2011, 1402 researchers participated in CNST projects, a 45 % increase over 2010. These researchers represented 280 academic, government, and industrial institutions from 42 states, the District of Columbia, and 26 different countries. Of these institutions, 79 were private-sector companies, a 49 % increase over 2010.

We have added major new capabilities in both commercial tools and beyond state-of-the art instrumentation. In the NanoFab, recent acquisitions have focused on tools designed to support experimental processes that remain aligned with industrial production methods. The cleanroom's new photolithography suite, opening next month (June 2012), includes a new high-speed ASML stepper, a spin coater, and an automatic developer. This suite will also consolidate in one bay most of the Center's contact lithography equipment, including ovens and two contact mask aligners. Notable new capabilities recently added to our research labs include an environmental scanning transmission electron microscope for *in situ* observations of the structure and chemistry of dynamic processes during gas-solid interactions, as well as a multifunctional instrument capable of simultaneously providing correlated topological, chemical (via infrared spectroscopy), thermal, and mechanical property maps with a spatial resolution below the diffraction limit of infrared radiation.



The rapid annual growth in research participation is reflected in a concomitant growth in technical output, with 145 publications from our research participants appearing in fiscal year 2011, representing a 54 % increase over 2010. These publications included 65 co-authored by CNST staff members, a 55 % increase.

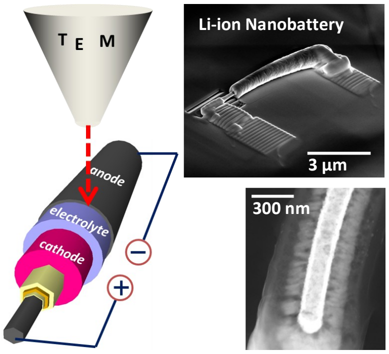
We are also very happy to report that in fiscal year 2011, CNST's own Henri Lezec received the prestigious Julius Springer Prize, which recognizes researchers who have made an outstanding and innovative contribution to the fields of applied physics, for his "pioneering achievements in nanoscale physics and applications." And, Kartik Srinivasan was presented with the Presidential Early Career Awards for Scientists and Engineers for developing measurement methods to probe strong light-matter interactions in semiconductor optical cavities, and for developing processes to fabricate low-loss, on-chip, nanophotonic devices. This award is the highest honor bestowed by our government to science and engineering professionals in the early stages of their independent research careers.

I invite you to participate in research at the CNST that will continue the development of nanotechnology "from discovery to production," either by using our NanoFab or collaborating with one of our instrumentation scientists. To learn more about our research opportunities, our growing capabilities, and our accomplishments, visit us on the web at www.nist.gov/cnst.

–Robert Celotta

Researchers Fabricate Solid-State Nanowire Lithium-Ion Battery

Researchers from the CNST and the University of Maryland have fabricated nanoscale rechargeable lithium-ion batteries (LIBs), enabling them to perform measurements directly correlating electrochemical activity with chemical and morphological changes occurring inside a battery during charge transfer. This experimental platform should overcome the limitations of external electrical measurements and post-mortem analysis that have been slowing the development of new, higher performance battery materials. LIBs are the most efficient electrochemical energy storage technology used in portable consumer electronics and are expected to be the dominant power source for electric vehicles in the near future. However, the high costs, limited range, limited lifespan, and safety concerns associated with LIBs constitute formidable obstacles to increasing the market penetration of electric vehicles. Because the materials and structures that constitute LIBs are extremely complex, neither electrical measurements nor post-mortem analysis using analytical electron microscopy have been sufficient to explain why charge capacity fades rapidly in some battery types or why other batteries fail abruptly. By fabricating batteries measuring less than a micrometer in diameter and approximately seven micrometers in length, the researchers were able to mount complete batteries inside a transmission electron microscope and image changes in the internal battery microstructure during electrochemical cycling. To fabricate these batteries, the researchers developed a technique using silicon nanowires as scaffolding on which the current collectors, cathode, electrolyte, and anode layers were deposited using semiconductor processing methods. This structure allowed them to monitor charge and discharge cycles while observing physical and chemical changes in the battery at the nanoscale, and to make an initial important discovery: LiPON, the solid electrolyte used in most commercial thin film Li-ion batteries, fails when its thickness is reduced below approximately 300 nm. This failure is marked by the rapid (about 2 hour) self-discharge of the battery, accompanied by bubble formation believed to be caused by oxygen gas evolution that was observed *in-situ* with a transmission electron microscope. The researchers believe that their experimental platform will provide insights into battery failure mechanisms, improve solid electrolytes, and advance the development of high energy density Li-ion batteries based on nanowires.



A transmission electron microscope was used to watch individual nanosized batteries with electrolytes of different thicknesses charge and discharge. The researchers discovered that there is likely a lower limit to how thin an electrolyte layer can be made before it causes the battery to fail.

Electrolyte stability determines scaling limits for solid-state 3D Li-ion batteries, D. Ruzmetov, V. P. Oleshko, P. M. Haney, H. J. Lezec, K. Karki, K. H. Baloch, A. K. Agrawal, A. V. Davydov, S. Krylyuk, Y. Liu, J. Huang, M. Tanase, J. Cumings, and A. A. Talin, *Nano Letters* **12**, 505-511 (2011).

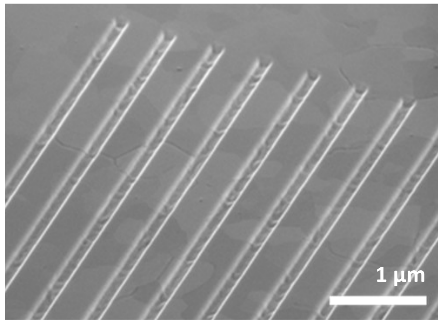
In the few years since its inception, the CNST has become a major national resource for nanoscale science and the development of nanotechnology, and the only national nanocenter with a focus on commerce.

Combining Centuries-Old Mathematical Theorems to Efficiently Characterize the Shape of Nanoparticles

CNST researcher Gregg Gallatin has shown that a mathematical technique combining Gauss's Law with Fourier transforms can be used as a starting point to solve a wide variety of standard problems in mathematics and physics. Because of the ubiquity of digital data derived from Fourier transforms, this technique is likely to find broad application to a range of physical science and engineering measurements. Using the technique, Gallatin demonstrated how Porod's law, which describes how x-rays scatter from small spherically-shaped particles, can be re-derived and extended to the broader case of particles that are nonspherical, thereby providing a powerful and useful approach for determining the shape of nanoparticles using x-ray scattering. He then demonstrated that this approach can be further extended to visible light scattering, which depends on the moments of the nanoparticle shape and therefore provides a more general method for measuring nanoparticle shape from scattering data. The technique of combining Gauss's Law with Fourier transforms can also be applied to the classical physics problem of Fraunhofer diffraction, providing an explicit formula for the diffraction pattern of arbitrary polygonal-shaped openings in an opaque screen in terms of the vertices of the polygon. It is also applicable to a variety of mathematics problems, including the Hopf Umlaufsatz, which states that the angle of the tangent along a simple smooth closed curve turns by 360 degrees when making a complete circuit around the curve; Stokes' Law, which relates integrals over an area in two dimensions to the one dimensional curve bounding the area; and the isoperimetric inequality, which states that a circle is the shape that encloses the largest area for a given circumference. Given the simplicity and generality of this mathematical technique, Gallatin believes that it can be applied to many other problems as well.

Fourier, Gauss, Fraunhofer, Porod and the shape from moments problem, G. M. Gallatin, *Journal of Mathematical Physics* **53,** 013509 (2012).

Efficiently Coupling Light from a Plane Wave into a Surface Plasmon Mode

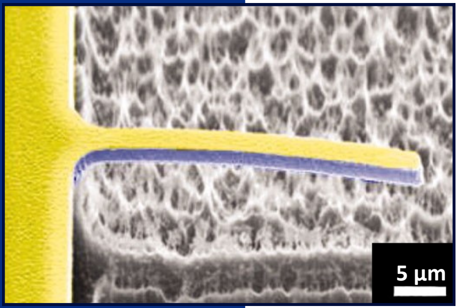


Scanning electron micrograph of a grating coupler. The gold film on mica is atomically smooth except at the grain boundaries.

CNST researchers have made a grating coupler that transmits over 45 % of the incident optical energy from a plane wave into a single surface plasmon polariton (SPP) mode propagating on a flat gold surface, an order-of-magnitude increase over any SPP grating coupler reported to date. Surface plasmons are propagating waves of light tightly confined to a metal surface via coupling with oscillating electrons in the metal. SPPs have been used to route signals for optical interconnects and to concentrate light for molecular sensors. The researchers' simple integrated coupler may improve performance and lower packaging costs for such devices and may also enable high-frequency optical connections between devices over longer distances. The researchers developed an analytical model of the coupling process to optimize the depth, width, and period of the identical rectangular grating grooves that they nanofabricated on a gold surface. Optical measurements on different sets of gratings confirmed the model's prediction that the highest efficiency would occur with "critical coupling," when the scattering by the grating grooves is matched to the intrinsic losses of the SPP propagating on the grating. Because these couplers can be used to excite surface plasmonic devices more efficiently, the researchers expect that they will enable the development of a variety of future on-chip devices.

An efficient large-area grating coupler for surface plasmon polaritons, S. T. Koev, A. Agrawal, H. J. Lezec, and V. A. Aksyuk, *Plasmonics*, **published online November 2011**, 1-9 (2011).

Multi-Institutional Collaboration Integrates New Efficient Piezoelectric Material into MEMS Sensors



False color scanning electron micrograph of a lead magnesium niobate-lead titanate

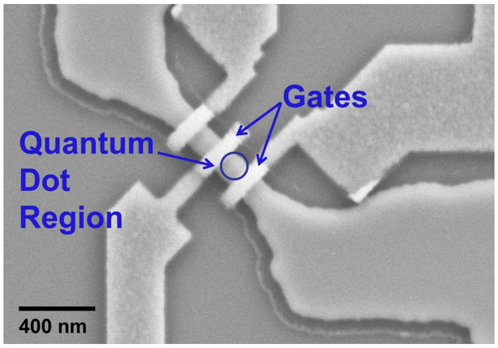
A team of researchers from the University of Wisconsin-Madison, Penn State, University of California, Berkeley, University of Michigan, Cornell, and Argonne National Laboratory, working with the CNST's Vladimir Aksyuk, have succeeded in integrating a new, highly efficient piezoelectric material, lead magnesium niobate-lead titanate (PMN-PT), into a silicon microelectromechanical system (MEMS). This development could lead to significant advances in sensing, imaging, and energy harvesting. Piezoelectric materials, such as quartz, expand slightly when fed electricity and, conversely, generate an electric charge when squeezed. Quartz watches take advantage of this property to keep time: electricity from the watch's battery causes a piece of quartz to expand and contract inside a small chamber at a specific frequency, and circuitry converts that motion into the displayed time. Piezoelectric materials are also found in sensors in sonar and in ultrasound systems, which use the same principle in reverse to translate sound waves into images. Although conventional piezoelectric materials work fairly well for many applications, researchers have long sought to develop new ones that expand more and produce higher voltages. More reactive materials would make for better sensors and could enable new technologies such as "energy harvesting" to transform the energy of walking and other mechanical motions into electrical power. The researchers developed a way to incorporate PMN-PT into tiny cantilevers on a silicon base, and demonstrated that PMN-PT could deliver two to four times more movement with greater force—while using only 3 volts—than most rival materials studied to date. CNST's Aksyuk conducted resonance frequency measurements and developed engineering models of the cantilevers to determine how much they would bend at different voltages, confirming that the experimental observations were due to the piezoelectric's performance. He also modeled silicon systems that achieve similar effects using electrostatic attraction to compare and highlight the benefits of piezoelectric actuation. "Our work shows definitively that the addition of PMN-PT to MEMS designed for sensing or as energy harvesters will provide a tremendous boost to their sensitivity and efficiency," says Aksyuk, "a much bigger 'bend for your buck,' you could say."

Giant piezoelectricity on Si for hyperactive MEMS, S. H. Baek, J. Park, D. M. Kim, V. A. Aksyuk, R. R. Das, S. D. Bu, D. A. Felker, J. Lettieri, V. Vaithyanathan, S. S. N. Bharadwaja, N. Bassiri-Gharb, Y. B. Chen, H. P. Sun, C. M. Folkman, H. W. Jang, D. J. Kreft, S. K. Streiffer, R. Ramesh, X. Q. Pan, S. Trolier-McKinstry, D. G. Schlom, M. S. Rzchowski, R. H. Blick, and C. B. Eom, *Science* **334**, 958 - 961 (2011).

Fabrication of CMOS-Quality Single-Electron Nano-Transistors

NIST researchers have fabricated multi-level silicon nano-MOSFET's, which at low temperatures can measure or control the motion of single electrons. This advances their ongoing efforts to develop both electrical standards based on the charge of the electron and single electron devices (SEDs) for quantum computing applications.

SEDs have the ability to move electrons around one-by-one in solid-state devices; for comparison, a typical household current of 1 Ampere corresponds to 10^{19} electrons passing per second. While researchers have been fabricating and measuring SEDs made from aluminum and aluminum oxide for a number of years, recently interest has developed in making these devices with silicon using standard CMOS processing techniques in order to improve device stability and speed. Physicist Neil Zimmerman and a team of researchers from the NIST Physical Measurement Laboratory have been trying to make such devices elsewhere for several years. However, the devices require a process flow with over 80 individual processing steps, all of which must work reliably to make functioning devices. By working with a process engineering team in the CNST NanoFab, they have now succeeded in making robust SEDs that show good single electron characteristics at low temperatures. These devices have only small inhomogeneity in their threshold voltages, high robustness with respect to application of large voltages, and small charge offset drift. According to Zimmerman, the multi-step process was “a tour-de-force in nanofabrication at a research user facility. The reliable processes we developed with NanoFab staff allowed the project to go forward, both in terms of the integration ability to make multi-level complex nano-transistors, and the ability to reproducibly grow high quality insulating films.”



Scanning electron micrograph of a silicon single electron device. Electrons are trapped in a quantum dot, defined by the field from two conducting gates.

The researchers are currently fabricating single-electron turnstiles in order to measure the error rate of passing electrons one-by-one and fabricating double quantum dots with charge sensors for use in quantum coherence experiments. They believe these devices will enable fundamental current standards at higher values of current, and development of Si-based qubits for use in quantum computing .

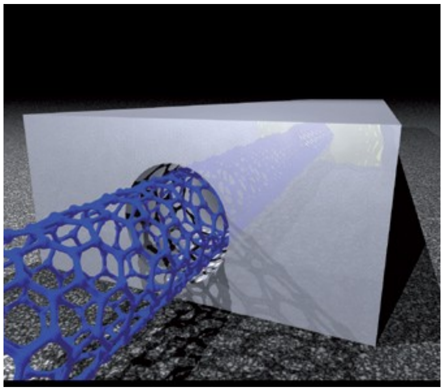
For more information, contact neil.zimmerman@nist.gov.

Existing models of electrical contacts in bulk semiconductor devices are inapplicable at the nanoscale. In order for nanosystems to progress to practical use, it is critical to control charge at the electrical contacts.

Researchers Publish a Detailed Review of Electrical Contacts in One and Two Dimensional Nanomaterials

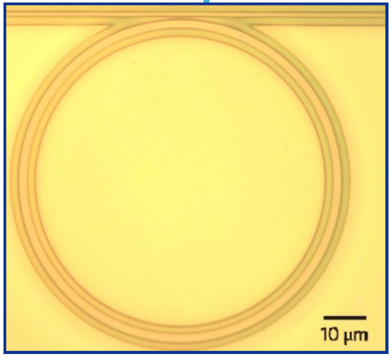
Researchers from the CNST and Sandia National Laboratories have published a detailed review of recent experimental and theoretical work highlighting the unusual physics and material science of electrical contacts to nanostructures. In the *Nature Nanotechnology* article, the researchers explain that existing models of electrical contacts in bulk semiconductor devices are inapplicable at the nanoscale, and argue that in order for nanosystems to progress to practical use, it is critical to control charge at the electrical contacts. New models are required to understand contact formation and charge transport. In conventional contacts, the interface between a metal and a semiconductor is planar, but nanocontacts have multiple possible geometries, each with unique properties. The kinetics and thermodynamics of metal/nanostructure interfaces also differ from those of the bulk materials due to their small lateral dimensions and to the greater ability of nanostructures to accommodate strain. Three examples illustrate the range of contacts that are possible with different nanomaterials. First, abrupt epitaxial silicide/silicon nanowire junctions with novel orientations can be formed at temperatures well below those required for thin metal films, providing new opportunities for emerging devices such as metal source-drain MOSFETs and SpinFETs. Second, for metal contacts to carbon nanotubes, catalytically driven carbonization of the interface results in an electrically transparent graphene-CNT contact. Finally, making low resistance ohmic contacts to semiconductor nanowires has proven challenging and requires new understanding of doping at the nanometer scale. The researchers conclude that better understanding of the basic science of nanoscale contacts is necessary to allow nanoscale materials to be incorporated into useful new device designs.

Electrical contacts to one- and two-dimensional nanomaterials, F. Leonard and A. A. Talin, *Nature Nanotechnology* **6**, 773-783 (2011).



An illustration of a single carbon nanotube encapsulated by an electrode.

On-Chip Resonator Produces Ultra-Short Light Pulses



Microscope image of a 40 µm-radius microring with a coupling region.

Researchers from the CNST and Purdue University have designed and fabricated an on-chip microresonator that converts continuous laser light into ultrashort pulses consisting of a mix of well-defined frequencies, a technology with applications in advanced sensors, communications systems, and metrology. In the new approach, infrared light from a continuous laser enters a chip through a single optical fiber and is directed into an 80 µm-diameter silicon nitride ring. The microscale ring acts as a nonlinear optical resonator with a defined set of resonances that reemit the light in a set of evenly spaced frequencies. These are called "comb lines" because they resemble teeth on a comb when represented on a frequency graph. The light is then collected through another optical fiber and sent to a pulse shaper to control the phase of each individual frequency line. The research team demonstrated that an optical frequency comb generated on-chip in this way can be highly coherent, meaning that individual comb lines remain synchronized with each other for long periods of time. Moreover, the phases of the comb lines can be adjusted to compress the light into a train of ultrashort pulses. The high repetition rate of the pulses produced by the on-chip microresonators may enable their use for improving the performance of high speed electron microscopes. In addition, they may be competitive with mode-locked lasers for some laboratory measurement applications: such lasers also produce a train of short pulses with well-defined frequencies, but are typically much larger than the chip-based devices and are limited by longer time delays between pulses.

Spectral line-by-line pulse shaping of on-chip microresonator frequency combs, F. Ferdous, H. Miao, D. E. Leaird, K. Srinivasan, J. Wang, L. Chen, L. T. Varghese, and A. M. Weiner, *Nature Photonics* **5**, 770-776 (2011).

Researchers Resolve Century-Long Debate Over How to Describe Electromagnetic Momentum Density in Matter

The interaction between a light pulse and a light-absorbing object can be calculated for any positive index of refraction using a few, well-established physical principles combined with a new model for mass transfer from light to matter.

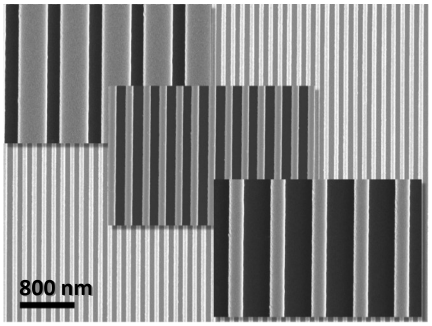
Researchers from the CNST and the University of British Columbia have shown that the interaction between a light pulse and a light-absorbing object, including the momentum transfer and resulting movement of the object, can be calculated for any positive index of refraction using a few, well-established physical principles combined with a new model for mass transfer from light to matter. This work creates a foundation for understanding light absorption in metamaterials, artificially tailored materials of intense interest in nanophotonics and microwave engineering that can have negative indices of refraction, and have potential applications in high resolution imaging, lithography, optical sensing, high gain antennas, and stealth radar coatings.

Light carries momentum and can transfer momentum to matter via radiation pressure. However, for the past century, there has been an ongoing debate over the correct form of the electromagnetic momentum density in matter. In the "Minkowski formulation," the momentum density is proportional to the index of refraction; in direct contrast, the "Abraham formulation" finds it to be inversely proportional. While light is known to carry mass, a detailed model for mass transfer from light to a medium that absorbs light had not been formulated to date. The researchers propose a set of postulates for light-matter interaction that encompass: a) the Maxwell equations, which govern classical electromagnetic behavior; b) a generalized Lorentz force law, which describes the force felt by matter in the presence of an electromagnetic field; c) a model for electromagnetic mass density transfer to an absorbing medium; and d) the Abraham formulation of momentum density. Using both closed-form calculations and numerical simulations of the interaction between an electromagnetic pulse and a test slab, the researchers demonstrated that their postulates yield results that are consistent with conservation of energy, mass, momentum, and center-of-mass velocity at all times. They further showed that satisfaction of the last two conservation laws unambiguously identifies the Abraham form as the true form of momentum density in a positive-index medium. In addition to the theoretical significance of these results and the implications for metamaterials, the results will enable more accurate modeling of light-matter interaction at the nanoscale and open new routes to optical control of nano-mechanical systems incorporating light absorbing materials.

Revisiting the Balazs thought experiment in the presence of loss: electromagnetic-pulse-induced displacement of a positive-index slab having arbitrary complex permittivity and permeability, K. J. Chau and H. J. Lezec, *Applied Physics A* **105**, 267-281 (2011).

NanoFab Develops Process for Fabricating Gratings for Optical Devices

Engineers in the CNST NanoFab have developed a set of techniques to manufacture optical gratings tailored to the needs of photonics, medical imaging, and telecommunications researchers. While optical gratings are typically manufactured using interferometric techniques that expose a large surface area, gratings produced using nanofabrication techniques can be localized anywhere on a wafer surface, allowing them to be integrated into the fabrication of a wide variety of devices, including lasers, spectrometers, and wavelength division multiplexing devices. The engineers use direct-write e-beam lithography (EBL) to define the grating pattern, and reactive ion etching (RIE) to anistropically etch the pattern into the substrate material. By using direct write EBL, the engineers can tailor the spacing as well as the location of the gratings to produce diffracted light of any desired optical wavelength. Their fabricated gratings have linewidths and pitches ranging from 100 nm to 400 nm. Unlike gratings produced using interferometry, which have cross-sections which are sinusoidal, this technique produces gratings with rectangular cross-sections. Given the widespread use of optical gratings in a variety of devices, the engineers believe that this process will be a valuable nonproprietary tool for laser and optics manufacturers who want to incorporate optical gratings into their device designs.



Scanning electron micrographs of a set of nanofabricated optical gratings. The grating widths and pitches can be varied to meet the

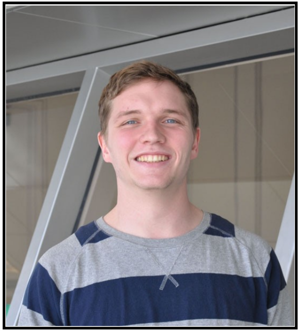
New Staff in the NanoFab

Joshua Schumacher is a Senior Process Engineer in the NanoFab. He received a B.S. and a Ph.D. in Electrical Engineering from the University of South Florida (USF), where he also received a certificate in Materials Science. Prior to joining the CNST, Joshua worked on materials characterization using scanning electron microscopy, transmission electron microscopy, and focused ion beam systems at the Nanotechnology Research and Education Center at USF. Joshua is responsible for training NanoFab users and for operating the NanoFab's focused ion beam systems.

Liya Yu is a Process Engineer in the NanoFab. She received a B.S. in Material Science from National Cheng Kung University, Taiwan, and a Ph.D. in Materials Science and Engineering from North Carolina State University. Prior to joining the CNST, she worked on process optimization for deep ultraviolet lithography at the North Carolina State University Nanofabrication Facility. Liya is responsible for developing and maintaining lithography processes in the NanoFab and for training and assisting users.

Kerry Siebein is a Process Engineer in the NanoFab. She received a B.S. in Mechanical Engineering from the Worcester Polytechnic Institute and a Ph.D. in Materials Science and Engineering from the University of Florida. Prior to joining the CNST, she managed the transmission electron microscopy and field emission scanning electron microscopy laboratories at the Major Analytical Instrumentation Center at the University of Florida, and taught, trained, and assisted students and faculty. Kerry is responsible for training NanoFab users and for operating the focused ion beam systems, scanning electron microscopes, and the transmission electron microscope in the NanoFab.

James Bittner is an Engineering Technician in the NanoFab. He has a B.S. in Physics from Longwood University, and has extensive experience repairing and troubleshooting computer-controlled electrical and mechanical systems. In the NanoFab, James assists researchers, maintains laboratory equipment, and services several advanced tools.



Center for Nanoscale Science and Technology

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Supporting the development of nanotechnology from discovery to production.

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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Announcing the Next
NanoFab Users Meeting

Thursday, June 7, 2012, 2 pm to 4 pm

Building 215/C103

Current and potential NanoFab researchers and others interested in NanoFab operations are invited to the quarterly NanoFab Users meeting. Topics typically include safety, policy changes, new equipment purchases or upgrades, research highlights, and new standard processes. Every meeting also includes an open discussion to allow users to bring ideas and suggestions to our attention. Anyone wishing to have a specific item added to the agenda should contact Vincent Luciani at 301-975-2886, vincent.luciani@nist.gov.

NanoFab Purchases Automated Lift-Off Tool

The new Avenger Spray Solvent System heated metal liftoff tool will be delivered in June. The device will provide automatic recipe control and reduce liftoff time from hours to minutes. It will process samples ranging from 150 mm-diameter wafers down to 25 mm-on-a-side squares. For more information, contact Jerry Bowser at 301-975-8187, jerry.bowser@nist.gov.

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