

# The CNST News

#### FALL 2010

#### From the Director

As part of our ongoing efforts to streamline our communications, we have consolidated the *CNST Quarterly* and *NanoFab News* into an integrated *CNST News*. This quarterly newsletter features project highlights from across the Center, including those conducted by a range of users in the NanoFab. In addition to reporting on the latest developments in nanoscale fabrication and measurement methods, it describes new tools and processes in the NanoFab, and reports other information we hope will be useful to both current and potential research participants. We have also changed our distribution method to GovDelivery, a new government-wide system for communicating electronically with the public. On the last page of the *CNST News* 

you will find instructions for joining or managing your subscription to this and other NIST communications. Please let us know if you have any suggestions that would make this newsletter more useful to you. **—Robert Celotta** 

Center for Nanoscale Science & Technology

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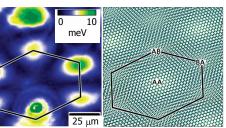
### **CNST-Georgia Tech Team Finds Electron Orbits in Multilayer Graphene Have Unexpected Structure**

The world-leading graphene research team from the Georgia Institute of Technology and the CNST has taken yet another step toward elucidating the unique and often unexpected properties of this two-dimensional carbon material-which, because of its potential applications to sensors and other electronic devices, has received intense international interest and was the subject of the 2010 Nobel Prize in Physics. One of the most important measurement problems related to graphene has been determining how the structure of the material affects the movement of electrons. The team has now measured how magnetic fieldinduced electron orbits in epitaxial graphene are distributed spatially and are affected by rotations between different graphene sheets in the material. The researchers used a custom-built,

low-temperature, high-magnetic field scanning tunneling microscopy system at the CNST to

measure the detailed electronic structure across a multilayer graphene film on a silicon carbide substrate with atomic-scale spatial resolution. They found that the energy states follow contours of constant electric potential, and that there

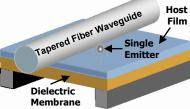
are energy gaps within isolated patches on the surface. These energy gaps create regions where electron transport is not allowed when subjected to high magnetic fields. The researchers proposed that the spatial dependence of the energy states is associated with the "moiré pattern" of atomic alignments that occurs when adjacent layers of graphene are rotated slightly from one to the next. The team's latest study



A map of the measured energy gaps on the surface of graphene (left) appears to be associated with the moiré alignment of two adjacent graphene sheets (modeled on right).

> raises a number of questions for future research, including whether the new phenomenon can be controlled, and how the observed effects may impact proposed carbon-based devices.

Real-Space Mapping of Magnetically Quantized Graphene States, D. L. Miller, K. D. Kubista, G. M. Rutter, M. Ruan, W. A. de Heer, M. Kindermann, P. N. First, and J. A. Stroscio, *Nature Physics* **6**, 811-817 (2010). PAGE 2



#### A tapered waveguide-based probe for deposited emitters on a dielectric membrane surface.

The high concentration of the optical field of these gap modes should enhance light emission rates by a factor of 20, and additional improvements are possible.

CNST theoretical physicists focus on developing models for predicting the behavior of nanoscale materials and structures.

#### An Efficient Optical Fiber Technique to Probe Individual Emitters

Using electromagnetic simulations, CNST researchers have shown that fiber taper waveguides can be very effi-

> cient for optical spectroscopy on individual light emitters, such as single molecules, atoms, or semiconductor quantum dots, deposited on a dielectric membrane. Optical fiber taper

waveguides, sometimes called micro- or nanofiber waveguides, are single mode optical fibers whose diameter is slowly and symmetrically reduced to a wavelength-scale minimum. CNST researchers are developing these structures for a range of light measurement solutions. In this work, the team simulated how, when placed in proximity to single emitters on a dielectric membrane, the fiber probe would form an optical waveguide supporting modes in the low refractive index gap between

the fiber and the membrane. The high concentration of the optical field of these gap modes, when interacting with vertical electric dipoles, should enhance light emission rates by a factor of 20. Additionally, efficient power transfer from the gap modes into the optical fiber should improve collection efficiencies to values of more than 20%. These two factors should significantly improve the signal to noise ratio in light detection from single emitters compared with standard lensbased collection methods.

The strong interaction between gap modes and emitters also led the researchers to propose a new technique for spectroscopic measurements based on the ability of a single emitter to strongly modify light transmission through the fiber probe. An individual emitter would be illuminated with light from a narrow bandwidth tun-

able laser whose wavelength is swept with high spectral resolution. This should allow the determination of dipole transitions lines with considerably higher resolution than possible with a grating spectrometer, potentially leading to a better understanding of the interaction between an emitter and its surrounding environment. Calculations performed for two slightly different configurations predicted similar performance at both visible and infrared wavelengths, suggesting the technique will be widely applicable. Finally, light can be efficiently collected with a fiber taper waveguide from a wide variety of structures without complex sample preparation.

Hybrid Gap Modes Induced by Fiber Taper Waveguides: Application in Spectroscopy of Single Solid-state Emitters Deposited on Thin Films, M. Davanco and K. Srinivasan, *Optics Express* **18**, 10995–11007 (2010).

#### Statistical Method Improves Single-Particle Tracking

Andrew Berglund, a Project Leader in the CNST Nanofabrication Research Group, has developed an improved data analysis method for determining the diffusion coefficient of a single nanoparticle using a series of images captured in a microscope.1 In fluidic systems, the diffusion coefficient provides a sensitive measure of nanoparticle size and shape and also contains information about the material properties of the suspending medium, making it an important nanoscale characterization tool. Berglund's new method introduces a maximum likelihood

estimator (MLE), which is computationally simple and accounts for technical difficulties such as finite spatial resolution and motion blur during image acquisition, making it useful in realistic experimental settings. Furthermore, for particles undergoing pure (i.e. not anomalous) diffusion, the MLE is asymptotically optimal, which means it gives an estimate of the diffusion coefficient with the smallest possible variance if the measurement contains enough data points. Analyzing an experiment by collaborators at the University of Maryland, aimed at controlling the position of individual quantum dots in solution with nanometer-scale accuracy, the method was successfully applied to determine the accuracy of the particle tracking and calibrate the diffusion coefficient measurements.<sup>2</sup>

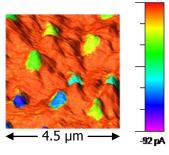
<sup>1</sup>Statistics of Camera-Based Single-Particle Tracking, A. J. Berglund, *Physical Review E* **82**, 011917 (2010).

<sup>2</sup>Manipulating Quantum Dots to Nanometer Precision by Control of Flow, C. Ropp, R. Probst, Z. Cummins, R. Kumar, A. J. Berglund, S. R. Raghavan, E. Waks, and B. Shapiro , *Nano Letters* **10**, 2525–2530 (2010).

#### FALL 2010

#### Imaging Nanoscale Morphology in a Polymer-based Solar Cell

Short-circuit photocurrent of a 300 nm nanodot array under illumination



CNST Researchers have combined scanning probe microscopy with novel nanofabrication and microtomy approaches to measure photocurrent variations with nanoscale spatial resolution in an organic solar cell. The most promising architecture for an organic solar cell consists of a blend of conducting materials that can be selforganized from solution into a film that forms the active device layer. The resulting photogeneration efficiency and the charge transport within this film depend strongly on its nanoscale

morphology. As described in the May 2010 issue of Nano Letters, the researchers used photoconductive atomic force microscopy (PCAFM), in which a conductive tip is raster-scanned over the film while illuminated, to collect photogenerated charges as a function of position while simultaneously measuring the topography of the film surface. By properly engineering the device structure, fabricating nanoscale contacts, and choosing a specific tip material, the CNST researchers revealed the charge transport via electron or hole conducting regions in the active layer as a function of the film's nanoscale morphology. Understanding how such film morphology affects device performance is crucial to designing new materials for highperformance, next-generation solar cells.

The Origin of Nanoscale Variations in Photoresponse of an Organic Solar Cell, B. H. Hamadani, S. Jung, P. Haney, L. Richter, and N. B. Zhitenev, *Nano Letters* **10**, 1611-1617 (2010).

#### NIST Team Measures Temperature-Dependent Current Spin Polarization in NiFe Alloy

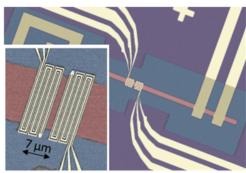
NIST researchers have measured the current spin polarization of Ni<sub>80</sub>Fe<sub>20</sub> (Permalloy) over a range of temperatures using an innovative spin-wave Doppler technique. Electrical currents in ferromagnetic metals tend to be magnetically polarized, meaning that a majority of the current is carried by either spin -up or spin-down electrons. Current spin polarization, which represents the imbalance of spin-up and spin-down electrons in the current, is a key parameter in the spintransfer torgue effect, which describes the torque exerted by polarized conducting electrons on local magnetic moments. The ability to switch the magnetization using electrical current though spintransfer torgue is the basis for number of new technologies, such as magnetic random access memory (MRAM) and nanoscale microwave

oscillators. Conventional methods to measure spin polarization are limited to low temperatures, but measurement of current spin polarization over a temperature range including room temperature is important to successful implementation of these new technologies. CNST and Material Measurement Laboratory researchers excited spin waves in ferromagnetic Ni<sub>80</sub>Fe<sub>20</sub> wires using nanoscale microwave antennas and measured the frequency shift of the spin wave when passing currents through the wires. Similar to the Doppler Effect in a moving medium, the currentdriven drift velocity of magnetization is obtained from the frequency shift and the wave vector of the spin wave. Then the current spin polarization is deduced from the drift velocity. This technique has the advantages of being immune to

interfacial effects and offering the freedom to change the temperature. Results including increasing current spin polarization in  $Ni_{80}Fe_{20}$  with decreasing temperature were reported in a Rapid Communication in the April 2010 issue of Physical Review B.

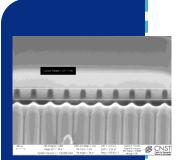
Temperature Dependence of Magnetization Drift Velocity and Current Polarization in  $Ni_{80}Fe_{20}$  by Spin-Wave Doppler Measurements, M. Zhu, C. L. Dennis, and R. D. McMichael, *Physical Review B* **81**, 140407 (2010).

While the NanoFab provides a **comprehensive** suite of commercial tools, the **CNST's** scientists and engineers are creating the next generation of nanoscale measurement instruments and fabrication methods, which are accessible through collaboration.



False-color SEM image of a spin wave Doppler device. The inset shows the antennas that launch and detect spin waves in a Ni<sub>80</sub>Fe<sub>20</sub> magnetic metal wire (in red).

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PAD process gives conformal TiO<sub>2</sub> coatings for gratings.

# New High Index Conformal Dielectric Coating Process

Conformal coating of nanofabricated structures with high index materials is a challenge for photonic nanostructures and other nanoscale devices. Recently, researchers from Senspex, working in the Nanofab with assistance from the NanoFab staff, developed a low cost, polymer-assisted deposition (PAD) process for coating titanium dioxide  $(TiO_2)$  into nanostructures. In the PAD process, a thermally curable, hybrid, high refractive index polymer solution is spin-coated

on to a substrate. The viscous polymer binds the metal ions, which are in a homogeneous distribution in the precursor solution. When cured at elevated temperature, the polymer decomposes to form a metal oxide-rich film with high refractive index that conformally fills the voids in nanofabricated structures. This new process can be applied inexpensively to make a diverse set of integrated photonic and plasmonic devices such as planar waveguides, on-chip spectrometers, gratings, flat panel displays, optical sensors, and integrated optical devices. For more information about the process, contact Lei Chen, 301 -975- 2908.

Polymer-Assisted Conformal Coating of  $TiO_2$  Thin Films, E. S. Gillman, D. Costello, M. Moreno, A. Raspopin, R. Kasica, and L. Chen, *Journal of Applied Physics* **108**, 044310 (2010).

The NanoFab is accessible to industry, academia, NIST, and other government agencies on a costreimbursable basis.

### **IBM Uses NanoFab for Supercomputer Optimization**

IBM researchers are investigating advanced supercomputer design at the CNST NanoFab. IBM's Blue Gene family of supercomputers delivers ultrascale performance within a standard programming environment enhancing efficiencies in power, cooling and component density.

Two major issues being addressed at the CNST are device size mismatch and assembly. Component size variability is large enough to significantly limit the number of usable connections. A tunable lithography is required to overcome this mismatch. Secondly, increased device density demands an expensive toolset upgrade. The CNST NanoFab's Heidelberg laser direct write tool is providing the crucial pattern recognition and overlay capability needed to obtain the required interconnection density and handling requirements for future systems. IBM's research and toolset acquisition will leverage learning from the current work.

NanoFab Project Leader: Roy Yu (IBM), 914-945-1343

The NanoFab adds ASML's Model PAS 5500/275D i-line stepper to its toolset.



#### NanoFab Acquires a Stepper

The NanoFab is expanding its lithographic capability with the recent purchase of a stepper, Model PAS 5500/275D Aerial i-Line from ASML, with resolution down to 280 nanometers and overlay accuracy down to 40 nanometers. The tool will be capable of handling 200 mm wafers up to 1 mm thick as well as small pieces, and will provide front and backside alignment. It should be available for users in early 2011. For more information, contact Rich Kasica, 301-975-2693.

# **University-Army Collaboration Investigating SiC MOSFET Treatments**

Researchers from the University of Maryland, Auburn University, and the Army Research Laboratory (ARL) are working in the NanoFab to investigate various treatments for SiC devices to extend performance and reliability for communications applications. Silicon carbide (SiC) is a wide bandgap semiconductor used to fabricate devices that operate at high temperature, frequency, and power. Its high electron mobility makes it attractive for MOSFETs. However, SiO<sub>2</sub>/ SiC MOSFETs show reduced channel mobility related to interface

Researchers prepared their TEM samples using the NanoFab's Zeiss NVision Focused Ion Beam tool

traps believed to be caused by carbon diffusion into the SiO<sub>2</sub> layer. The research team improved both interfacial sharpness and electron mobility by using a nitrogen plasma treatment. Chemical analysis of the  $SiO_2/$ SiC interfaces

by electron energy loss spectroscopy (EELS) shows less carbon and oxygen diffusion for the nitrogen plasma treatment compared with the untreated sample, with channel mobility in the plasma-treated devices of 30-40 cm<sup>2</sup>/Vs compared to 10 cm<sup>2</sup>/Vs for the untreated devices. The desired electron mobil-



Lamella prepared using FIB after mounting on a TEM grid (left) and high resolution lattice image from the SiC/SiO<sub>2</sub> interface (right).

ity is an order of magnitude higher than the present result.

TEM samples were prepared in the NanoFab using the Zeiss NVision 40 CrossBeam<sup>™</sup> focused ion beam (FIB) tool. A layer of carbon was deposited on the surface to protect the sample from gallium damage during milling. A thin slice (lamella) was removed from the sample after gallium ion milling. It was then mounted on a grid for TEM analysis that produced a high resolution lattice image revealing the interfacial structure.

The EELS data from the TEM lamella show a slight increase in the C/Si ratio within 23 nm from the interface on the untreated sample, less than the 20% that has been previously reported. The second sample was treated with nitrogen plasma at 1,175°C for 20 hours, with the lamella extracted in the same way, but shows no increase in the C/Si ratio at the interface. The change in C/Si and O/Si across the interface of this sample is very sharp except for some possible

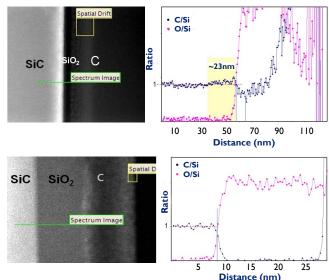
# Sputtered ITO Film Process Developed

Indium tin oxide (ITO) is widely used as a conductive transparent contact layer. It is amorphous and non-conductive as deposited, and must be annealed to crystallize. A new ITO process now available in the NanoFab achieves both optimum conductivity and transmissivity using our Sputter#2 tool and Rapid Thermal Annealer. The steps at the interface and along the direction of the beam.

The team is planning two additional treatments, annealing and ion implantation, to obtain additional target characteristics.

This work is sponsored by ARL and is a collaboration between John Williams at Auburn University, Aivars Lelis at ARL, and Lourdes Salamanca-Riba at the University of Maryland.

NanoFab Project Leader: Lourdes Salamanca-Riba (University of Maryland), 301-405-5220



Dark field Scanning TEM images of the lamellae and corresponding C/Si and O/Si ratios as measured along the green line on the images for untreated SiC/SiO<sub>2</sub> (top) and nitrogen-plasma treated SiC/SiO<sub>2</sub> (bottom).

process was developed in collaboration with Edward Flagg of the Atomic Physics Division at NIST. For more information, contact Gerard Henein, 301-975-5645.

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NanoFab Process Engineer gives oneon-one training for the Zeiss FESEM Ultra60.

> With a simple application process, the NanoFab provides rapid access to a comprehensive suite of tools and processes for nanofabrication and measurement.

The NanoFab is staffed Monday through Friday, 7 am until 12 midnight. After hours access is possible with the buddy system, and advance approval, subject to NIST campus access restrictions.

#### **Improved Nanoimprint Lithography Mold Fabrication Process**

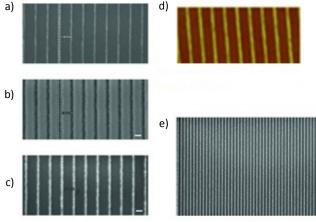
Nanoimprint lithography (NIL) is a direct-write technology that may replace expensive serial patterning methods such as high resolution electronbeam lithography. Researchers from NIST, the University of Michigan, and industry have demonstrated a fast. low-cost mold fabrication process based on a readily available bifunctional silsesquioxane (SSQ) monomer. The cubic SSQ monomer is directly patterned by NIL and then thermally converted into high-modulus structures with very little shrinkage, excellent thermal stability, quartz-like transparency in the UV, and an intrinsically low surface energy.

This high-performance sol-gel combines the ease of processing typical of organic materials with the thermal resistance, structural rigidity, and patterning resolution of hard template materials. It exhibits superior performance as a mold replication material, creating robust high-modulus replicas of a master. These daughter molds can directly imprint resists such as polystyrene or poly (methylmethacrylate) (PMMA). The CNST NanoFab NIL tool supports either ultraviolet or thermal imprinting processes.

This work is described in the Aug. 16, 2010 online issue of Advanced Materials. To learn more about using this process in the NanoFab, contact Lei Chen, 301-975- 2908.

Cubic Silsesquioxanes as a Green, High-Performance Mold Material for Nanoimprint Lithography, H. W. Ro, V. Popova, L. Chen, A. M. Forster, Y. Ding, K. J. Alvine, D. J. Krug, R. M. Laine, and C. L. Soles, Advanced Materials. First published online: 16 AUG 2010, DOL: 10.1002/adma.201001761.

a) SEM of the master SiO<sub>2</sub> grating pattern with ~30 nm line width and 20 nm pattern height. b) SEM of replica of the master SiO<sub>2</sub> mold. c) SEM of imprint from the replica. d) AFM height image for the imprint. e) SEM of the imprint from replica over a large scale.



### NanoFab Schedule for the Holidays

Day	Date	Event	Staff Support	NanoFab Access
Fri	29 Oct	Site wide outage	7 am – 12 pm	No Access after 12 noon
Sat	30 Oct	Site wide outage	None	No Access
Sun	l Nov	Site wide outage	None	No Access
Thu	11 Nov	Veterans Day	None	Buddy system only
Thu	25 Nov	Thanksgiving	None	Buddy system only
Fri	26 Nov	Day after Thanksgiving	7 am – 5 pm	Buddy system after 5 pm
Thu	23 Dec	Early closing	7 am – 5 pm	Buddy system after 5 pm
Fri	24 Dec	Federal holiday	None	Buddy system only
Fri	31 Dec	Federal holiday	None	Buddy system only

# The CNST Welcomes New Staff Members and Visiting Fellow

Sam Bowden, NRC Postdoctoral Research Associate, Electron Physics Group

#### Andrea Centrone,

CNST Visiting Fellow in the **Energy Research Group** and a Visiting Assistant Research Scientist at the Institute for Research Electronics and Applied Physics of the University of Maryland

Jungseok Chae, CNST/ UMD Postdoctoral Researcher, **Electron Physics Group** 

Kan Du, CNST/UMD Postdoctoral Research Associate, Nanofabrication Research Group

Teresa Figgs, Administrative Support Staff, Electron Physics Group

Veronika Szalai leads

fabricating biomimetic

nanostructures and on

measurements of nano-

biomaterials for artifi-

cial photosynthesis ap-

plications.

projects focused on

Niv Levy, NRC Postdoctoral Research Associate, Electron Physics Group

> Luanne Mehelich, Administrative Support Assistant, Center Office

Alline Myers, Senior Process Engineer, Nan-

oFab Operations Group Veronika Szalai, Project Leader, Energy Research Group

Tong Zhang, CNST/ UMD Postdoctoral Researcher, Electron Physics Group



The NanoFab offers researchers state-of-the-art tools.

# Summer Research Fellows and Interns Learn About NIST with Hands-On Experience

Yeehing Lam, Administrative

Research Group

Support (WAE), Nanofabrication

The CNST welcomed its 2010 class of summer student researchers. Nine Summer Undergraduate Research Fellowship (SURF) students and one Summer High School Intern Program (SHIP) student participated in a wide variety

of research projects ranging from nano-plasmonics to spin-

tronics, and learned about the latest in nanotechnology fabrication and measurement methods. The SURF program is sponsored by the National Institute of Standards and Technology and the National Science Foundation.

#### Summer Student Researchers

Mathew Swisher (Carnegie Mel-Ion U.), Nanofabrication Research Group

Olivia Hoff (U. of Southern Mississippi), Electron Physics Group

Chad Webb (U. Of Texas, Austin), Nanofabrication Research Group

Joshua Leibowitz (U. of Connecticut), NanoFab Operations Group

Ramsey Noah (U. of California, Long Beach), Nanofabrication Re-

#### search Group

Philip Cambell (U. of Texas), Nanofabrication Research Group

**Robert Hoyt** (Harvey Mudd College), Nanofabrication Research Group

Brian Soe (Harvey Mudd College), Electron Physics Group

Martin Vilarino (U. of Maryland), Energy Research Group



The CNST SURF students were among 130 at NIST attending seminars, going on lab tours, and presenting their work at an end of summer colloquium.

SHIP Student

Parakh Jain, Electron Physics Group





# Center for Nanoscale Science and Technology

National Institute of Standards and Technology 100 Bureau Drive, MS 6200 Gaithersburg, MD 20899-6200 Phone: 301-975-8001 E-mail: cnst@nist.gov Supporting the development of nanotechnology from discovery to production. The NIST Center for Nanoscale Science and Technology (CNST) was established in May of 2007 to accelerate innovation in nanotechnology-based commerce. Located in NIST's Advanced Measurement Laboratory Complex on the Gaithersburg, MD campus, the CNST is the only national nanocenter with a focus on commerce. It supports the development of nanotechnology through research on measurement and fabrication methods and technology. The CNST has a unique design which supports the U.S. nanotechnology enterprise through the readily available, shared use NanoFab, as well as providing opportunities for collaboration in multidisciplinary research on new nanoscale measurement instruments and methods.

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# **Online Reservation System Debuts in the NanoFab**

www.nist.gov/cnst

The new NanoFab Lab Reservation System (NLRS) went online on September 1, 2010, giving external users access to the Coral scheduling application from any computer. External customers will receive their NLRS account information in the next few weeks. The system gives users without NIST domain access the ability to make and delete tool reservation requests with fast response, improving scheduling convenience and flexibility.



Announcing the next NanoFab Users Meeting Friday, December 10th , 2 pm – 4 pm Building 215/C103

Current and potential NanoFab users 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, luciani@nist.gov.

# **Upcoming CNST Seminars**

Seminars are held in Bldg. 217, Rm. H107, unless otherwise noted. Designing Nanostructures for Energy, Gary Rubloff, Director, Maryland NanoCenter, University of Maryland, College Park.

Tuesday, November 16, 2010, 11 am

#### Visit the CNST Booth at these Upcoming Meetings

October 19 – 21	AVS International Symposium	Albuquerque, NM
Nov. 30 – Dec. 2	MRS Fall Meeting	Boston, MA
December 8 – 9	Nanotechnology Summit	Washington, DC