

From the Director

As we begin 2011, the NIST Center for Nanoscale Science and Technology enters the “end of the beginning.” We have completed the recruitment of our permanent staff, are in the final stages of equipping their laboratories, and are completing the hiring of a cadre of very talented postdoctoral researchers to begin what we hope will be memorable starts to highly productive careers. Similarly, the NanoFab is adding the last few key items needed to provide the comprehensive tool set requested by our stakeholders, and has brought in the technical experts needed to run and maintain these tools. We will soon be issuing our 2010 Report, which will provide a comprehensive update on the state of the CNST and highlight the 2009-2010 accomplishments of the Center and its research participants. In the interim, we hope you enjoy learning about the latest results in this Winter 2011 newsletter.

—Robert Celotta

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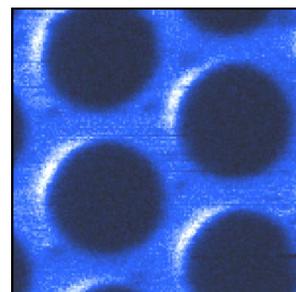
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Chromium Ion Beam Focused to the Nanoscale

Using the NIST-invented magneto-optical trap ion source (MOTIS), CNST researchers recently took a significant step toward the goal of creating a more versatile ion source for use in focused ion beams. Focused ion beams (FIBs) are widely used in nanoscale fabrication and imaging. Ions focused to nanometer dimensions with energies up to 30 kV can be scanned across a surface, removing or adding material through bombardment and beam-induced chemistry, or imaging via the generation of secondary electrons. While commercial FIB tools have been available for several decades, these generally use only gallium ions. Expanding the available ion species beyond gallium would create a suite of focused ion beam tools with a wide range of new nanofabrication and imaging possibilities.

The researchers have recently achieved a focal spot size of 200 nm using a focused chromium ion beam, and were able for the first time to acquire a clear secondary electron image, in this case of a microchannel plate surface containing a regular array of 10 μm -diameter holes. These early results are a significant step in an ongoing research project carried out jointly with FEI Co. to develop new sources of ions for high resolution imaging and fabrication.

Future work will attempt to improve the focused chromium beam system to attain the theoretical limit of a few-nanometer focus, as well as develop sources of both lighter and heavier ions, such as Li, Cs, and Er, based on the MOTIS principle. If these ions can be focused to the nanometer scale, light ions could image with-



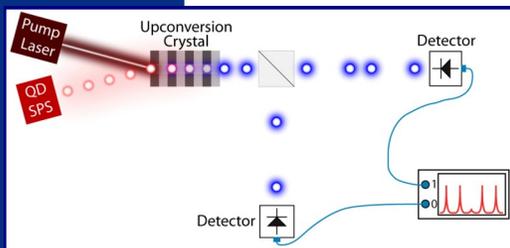
Focused chromium ion beam secondary electron image of a microchannel plate with 10 μm -diameter holes.

out damaging the sample, heavy ions could efficiently remove material by sputtering, and specific ionic species could be exploited for their chemical, material, or conductive properties to realize new ways to perform localized chemistry, deposit high purity nanoscale conductors, or implant dopants.

Focused chromium ion beam, A. V. Steele, B. Knuffman, J. J. McClelland, and J. Orloff, *Journal of Vacuum Science and Technology B* 28, C6F1 (2010).

The tolerances on feature size, shape, and placement for next generation computer chips fabricated with extreme ultraviolet (EUV) lithography will range from a maximum of a few nanometers down to less than 1 nm.

Control over single photon wavelength upconversion from a quantum dot single photon source (QD SPS) opens up possibilities for building so-called “hybrid quantum systems.”



A collaboration between the CNST and NIST's Information Technology Laboratory (ITL) has demonstrated frequency upconversion of single photons from a semiconductor quantum dot from the near-infrared

CNST and LBL Researchers Model EUV Imaging and Exposure

The tolerances on feature size, shape, and placement for next generation computer chips fabricated with extreme ultraviolet (EUV) lithography will range from a maximum of a few nanometers down to less than 1 nm. To achieve these tolerances, the sidewall roughness of the features, traditionally called line edge roughness (LER), is required to be less than 2 nm.

The CNST's Gregg Gallatin and Patrick Naulleau from the Lawrence Berkley National Laboratory (LBL) have developed a numerical model that accounts for the two dominant

sources of LER: the quantum statistics of exposing and developing the resist; and the roughness of the mask features themselves. Mask LER is about 10 nm, which reduces to 2 nm on the wafer using a 5X EUV imaging system. The model determines the relative contribution each LER source makes to the wafer under various imaging and processing conditions. It predicts wafer LER and also determines to what extent the frequency content of the mask contribution is altered by the imaging, exposure, and development processes.

The researchers have discovered that there are combinations of processes where the mask induced roughness is the dominant contributor to wafer LER, but its frequency signature is virtually indistinguishable from the contributions of exposure and development statistics alone. Therefore, other direct metrology methods in addition to wafer LER frequency content will be required to determine the separate contributions.

Effect of resist on the transfer of line-edge roughness spatial metrics from mask to wafer, G. M. Gallatin and P. P. Naulleau, *Journal of Vacuum Science and Technology B* 28, 1259–1266 (2010).

Changing the Color of Single Photons from a Quantum Dot

Converting single photon emission from one wavelength to another is important for integrating future quantum systems that combine low-loss optical transmission in the near-infrared with the long-lived memories that make use of near-visible light. It is also important in addressing the significant detection challenges present in currently available near-infrared single photon counters.

to the near-visible. The researchers achieved this “quantum transduction” using an on-demand 1300 nm single photon source developed at the CNST combined with an upconversion single photon detector developed in ITL. The single photons were generated using pulsed excitation of an InAs/GaAs quantum dot at a temperature of 8 K, and they were efficiently extracted from the low temperature environment using a novel optical fiber interface. By using a pump at 1550 nm, single photons at 1300 nm were converted to 710 nm in a nonlinear optical sum-frequency device, and then detected by a silicon based avalanche photodiode. The higher sensitivity of silicon avalanche photodiodes at near-visible wavelengths improved the dynamic range of the measurements by 25 times compared to InGaAs detectors, allowing the re-

searchers to confirm that the 710 nm light was still quantum mechanical in nature and still predominantly composed of single photons.

The results open up possibilities for building so-called “hybrid quantum systems,” where light from bright single photon sources, such as quantum dots, can be transmitted through optical fibers with very low loss in the 1300 nm wavelength region and then interfaced with quantum memories commonly operated in the near-visible. The work may also improve near-infrared detector technology, where low light level measurements are difficult due to performance limitations in standard commercial photodiodes.

Quantum transduction of telecommunications-band single photons from a quantum dot by frequency upconversion, M. T. Rakher, L. Ma, O. Slattery, X. Tang, and K. Srinivasan, *Nature Photonics* 4, 786–791 (2010).

Spin Currents Connected to Magnetic and Mechanical Torques

Paul Haney and Mark Stiles of the CNST have developed a theory of current-induced torques that generalizes the relationship between spin transfer torques, total angular momentum current, and mechanical torques, and is applicable to a much wider range of materials than previous theories. Their work describes two types of current-induced torques: a mechanical torque acting on the lattice, and a spin transfer torque acting on the magnetization.

Spin transfer torque is a well-established phenomenon in which the flow of spin current through a magnetic material can change its magnetization direction. This effect underlies a number of potential new technologies, including magnetic random access memory and nanoscale microwave oscillators. The effect has been well understood in materials where total spin is conserved—the magnetization is

equal to the total spin, and if there is a net spin current flux into or out of a volume, then the total spin, and, therefore, the magnetization inside the volume must change.

The researchers' theory extends this understanding to materials where total spin is not conserved, including materials with strong spin-orbit coupling, such as the magnetic semiconductor GaMnAs. The analysis of these systems requires the consideration of total angular momentum, which is always conserved. (Since magnetization is proportional to angular momentum, it contributes to the total angular momentum.) Conservation of total angular momentum arguments imply, for example, that a magnetic field-driven reversal of the magnetization is accompanied by a compensating change in the mechanical angular momentum of the sample, known as the Einstein-de

Haas effect. This change in mechanical angular momentum is small, but measurable; in fact, Einstein himself performed the experiment demonstrating this effect. By accounting for the mechanical angular momentum of the material, Haney and Stiles found a general relationship between total angular momentum currents, magnetic torques, and mechanical torques.

The theory is applicable to recent experiments that measure spin-current-induced nanomechanical torques in nonmagnetic materials, and predicts that magnetic torques may become stronger in the presence of spin-orbit coupling, making them more effective for current-induced magnetic switching.

Current-induced torques in the presence of spin-orbit coupling, P. M. Haney and M. D. Stiles, *Physical Review Letters* **105**, 126602 (2010).

CNST research is creating the next generation of nanoscale measurement instruments and methods, which are made available through collaboration.

Unexpected Energy States Revealed in Graphene using Ultra-Low Temperature Scanning Tunneling Microscopy

Measuring and understanding how electrons carry current through a graphene sheet is key to achieving the material's technological promise. Because of the geometry and electromagnetic properties of graphene's structure, an electron in any given energy level populates four possible sublevels in an applied magnetic field, called a "quartet."

As discussed in a highly publicized paper in *Nature*, CNST researchers have measured the energy separations of the quartet electron states for the first time and discovered additional fine details showing that the electron states can split into fractionally filled levels when exposed to extremely low temperatures and high magnetic fields, possibly related to fractional quantum

Hall physics in graphene. The new research raises intriguing questions about the fundamental physics of this exciting material.

Theorists have predicted that this quartet would split into different energies when immersed in a magnetic field, but previously there had not been an instrument sensitive enough to resolve these differences. Using the world's most powerful and stable scanning tunneling microscope (STM), with an unprecedented combination of low temperature (as low as 10 mK), ultrahigh vacuum, and high magnetic field, the researchers resolved fine differences in the electron energies in graphene. Complex quantum behavior was observed that appears to be a many-body effect in which

electrons interact strongly with one another, affecting their energy levels. One hypothesis for this behavior is that the electrons have formed a condensate in which they act as a single coordinated unit. If this theory proves to be correct, it could point the way to the creation of smaller, very low heat producing, highly energy efficient electronic devices based upon graphene.

High resolution tunneling spectroscopy of a graphene quartet, Y. J. Song, A. F. Otte, Y. Kuk, Y. Hu, D. B. Torrance, P. N. First, W. A. de Heer, H. Min, S. Adam, M. D. Stiles, A. H. MacDonald, and J. A. Stroscio, *Nature* **467**, 185–189 (2010).

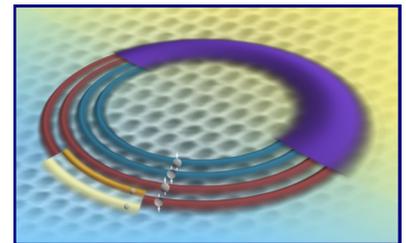


Illustration of the electron energy levels in graphene as revealed by CNST's new ultra-low temperature STM. An electron in any given energy level (the wide, purple band) comprises four quantum states (the four rings), called a "quartet."



A Titan 80-300 TEM has been installed in the NanoFab.

NanoFab Adding New TEM and FIB Capabilities

The NanoFab received an FEI Titan 80-300 transmission electron microscope (TEM) in January, and expects to make it available to researchers in the spring. The Titan is capable of atomic scale imaging and analysis of a wide range of materials and nanostructures, including electron tomography at high spatial resolution, allowing 3D imaging of nanostructures.

The microscope has a variable operating voltage of 80 kV to 300 kV, and accepts samples of 3 mm maximum diameter up to 100 nm thick. It can be operated in full-field imaging mode for conventional and

high-resolution TEM imaging, or in high resolution scanning transmission microscopy mode (STEM). TEM and STEM spatial resolutions are approximately 0.2 nm and 0.14 nm, respectively. Multiple electron diffraction modes are also available, including selected area electron diffraction and convergent-beam electron diffraction. STEM mode coupled with spectroscopy allows for chemical and elemental analysis from the very small volumes and is useful for studying the chemistry of interfaces.

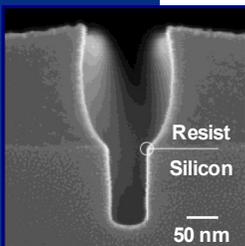
Elemental and chemical analysis

can be simultaneously acquired with an electron energy loss spectrometer (EELS) and an EDAX Si(Li) x-ray energy dispersive spectrometer. Energy-filtered TEM (EFTEM) elemental maps can also be acquired.

To complement the TEM, two FEI Helios NanoLab 650 Dual Beam focused ion beam systems are expected in the NanoFab in March 2011, which will provide additional capabilities for TEM sample preparation this summer.

For more information, contact Alline Myers, 301-975-3775; or Mike Hernandez, 301-975-4590.

Argon Pulsing in Etch Process Improves Etch Rate and Selectivity



A Si trench with smooth side walls patterned by electron beam lithography and alternating Ar and SF₆/C₄F₈ plasma etch.

Small and high aspect ratio structures can be easily fabricated in the NanoFab using fluorinated plasma chemistry that is inherently isotropic. To obtain the desired etch characteristics, NanoFab process engineers have optimized the addition of argon to the process flow.

Although direct addition primarily caused dilution, reducing the etch rate, by alternating the etch step with an argon-only step both high selectivity and high etch rates were ob-

tained while maintaining anisotropic etching. SF₆ and C₄F₈ were the active gases, with C₄F₈ used to protect the Si sidewalls and SF₆ used to etch. For a typical process with these gases, the etch rate is about 8.0 nm/min. Mixing argon with the etchant gases in concentrations from 0.0 % to 34.8 % gives very limited or no improvement on Si etch rate due to dilution. However, alternating argon surface bombardment steps with the chemical etch steps results in a four-fold increase in silicon etch rate while maintaining vertical sidewalls.

It is postulated that argon surface bombardment amorphizes the silicon, and the fluorine then reacts with and removes the silicon in the gas phase. The silicon etch rate increases with the argon step time and is independent of SF₆ step time. The argon bombardment step is the rate-determining step, and influences both etch rate and selectivity. By alternating argon bombardment and SF₆ etch steps, etch rate and selectivity are both improved.

For more information about using this process, contact Lei Chen, 301-975-2908.

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

New Capability for Depositing Ferromagnetic Materials

A special sputtering gun for ferromagnetic materials has been installed in the NanoFab's Sputter 1 tool (DC gun #1). It is similar to the DC gun #1 within Sputter 2, and enables the deposition of iron, cobalt, nickel, their alloys, and other ferromagnetic materials. It can also be used with other conductive materials. Recipes for use of this gun are posted on the tool.

For more information, contact Gerard Henein, 301-975-5645.

Dual Zone Plate Improvement Enables Large Radius-of-Curvature Metrology

Researchers from the Engineering Laboratory (EL) and process engineers for the NanoFab have collaborated on the fabrication of a dual-focus zone plate that improves the calibration service NIST can offer for measuring very large radius, spherical surfaces. The zone plate is used to calibrate the radius of curvature (ROC) of spherical surfaces using an interferometric test bench. The optimized dual zone plate made in the NanoFab extends the measurable ROC to 10 m, allowing very large radius spheres to be measured on the existing test bench. This calibration service is provided by NIST to a variety of companies, such as manufacturers of interferometers.

Two fabrication challenges were solved in the NanoFab. The first was to etch lines of widely varying pitch to the same depth. The second was to accommodate the very thick (5 mm) fused silica substrate in the RIE etch tool.



A new dual-focus zone plate.

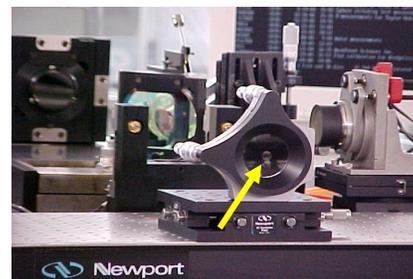
The zone plate contains two zones with different focal lengths, one confocal and one in the “cat’s eye” position. Each zone contains rings etched into the fused-silica substrate material where the ring spacing varies continuously. These rings

are created using optical photolithography, then etched by reactive ion etching.

Etch non-uniformity was improved from about 20 % to 2 % by careful optimization of power and chamber

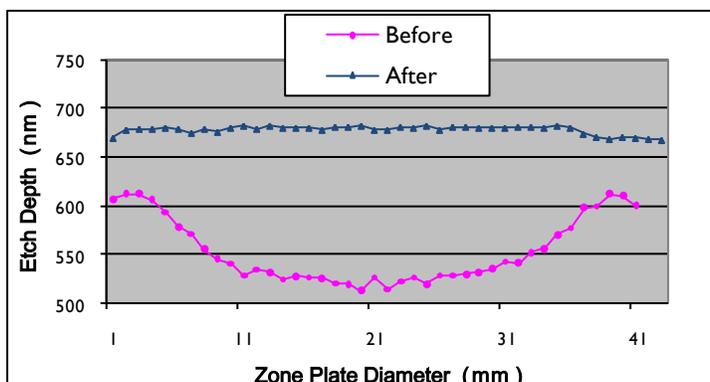
pressure, among other parameters.

The optimal conditions were identified as 300 watts at 30 mT pressure, with both parameters yielding linear responses for etch rate during optimization trials. A Teflon ring adapter of 5 mm thickness and 100 mm diameter with a 50 mm central void was designed and used in the etch tool to compensate for the thickness of the fused silica zone plate.



New dual zone plate incorporated into calibration test bench.

For more information, contact Lei Chen (NanoFab), 301-975-2908; or Quandou Wang (EL), 301-975-6364.



Zone plate etch uniformity, before and after optimization.

Critical Point Dryer Capability Expanded with Additional Small Chamber

The NanoFab has expanded its processing capability by adding a second Critical Point Dryer (CPD), which uses liquid carbon dioxide as a transitional fluid to dry fragile MEMS, suspended, and floating structures. Used after wet etching, CPD reduces stiction and increases yield of delicate MEMS structures. The Tousimis Autosamdri®-815 Series B is made for small samples,

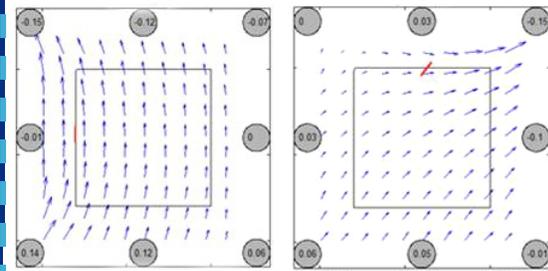
with a chamber size diameter of 32 mm and a volume of 25 ml. The small chamber dries faster and more efficiently, cooling from room temperature to 0 °C in less than 90 seconds, compared to 10 minutes on the larger CPD. The new unit also consumes half as much liquid CO₂ as the larger tool.

For more information, contact Marc Cangemi, 301-975-5993.

The NanoFab is staffed and open from 7 am to midnight, Monday through Friday.

Translational and Rotational Control of Nano-objects

CNST and University of Maryland researchers have developed a new technique for simultaneously controlling the position and orientation of a single nano-object in a fluid by precise manipulation of the



Two frames of a simulation of a 10 μm -long, 200 nm-wide nanowire (red) being translated along a square path and simultaneously rotated along each side of a 100 μm -wide square.

flow around the object. Vision based feedback control in a microfluidic device with 2D planar flow that is actuated by electro-osmosis is used to

move the object along the desired trajectory while compensating for translational and rotational Brownian motion of the rod. The position and orientation of the object is estimated at regular intervals using a camera and a robust vision algorithm. A controller then uses this estimate to calculate the deviation of the rod from a user-specified desired trajectory, and computes the required voltages to be applied at the electrodes located at the periphery of the device. The applied voltages generate an electro-osmotic flow (EOF) in the device that imparts a velocity to the object that corrects this deviation and steers the rod along the desired trajectory in a 2D plane.

Manipulating the fluid by EOF offers significant advantages as compared to other approaches. It allows for ma-

nipulation of a wider class of objects (the object does not have to be magnetic or have a significant dipole moment), it can be controlled over a long range ($>100 \mu\text{m}$) with sub-micrometer accuracy, the control algorithm can be tailored to the shape of the object, and the device is easy to make (requiring only the molding of an inexpensive polymer). Such a control technique holds promise for applications in nanofabrication such as building waveguides, force microscopy, and in biophysical applications where a sensor can measure properties of cell walls.

Simultaneous positioning and orientation of a single nano-object by flow control: theory and simulations, P. P. Mathai, A. J. Berglund, J. Alexander Liddle, and B. A. Shapiro, *New Journal of Physics* **13**, 013027 (2011).



Henri Lezec
CNST Nanofabrication
Research Group

Henri Lezec Wins Julius Springer Prize for Applied Physics

CNST Project Leader Henri Lezec was recently honored with the 2010 Julius Springer Prize for Applied Physics. Lezec shared this prestigious award with Federico Capasso, Robert L. Wallace, Professor of Applied Physics, and Vinton Hayes, Senior Research Fellow in Electrical Engineering at Harvard. The Springer prize recognizes researchers who have made an outstanding and innovative contribution to the field of applied physics, and has been awarded annually since 1998. The award recognized Lezec for his “pioneering achievements in nanoscale physics and applications,” and was presented in October at

the Julius Springer Forum on Applied Physics at Stanford University. Attendees heard talks from both prize winners, as well as a series of special lectures given by luminaries in the field of nanoscale physics.

Lezec, who joined NIST in 2007, received his Ph.D. from MIT in 1992 and has held research positions at NEC Fundamental Research Laboratories in Japan, Micrion Corp., the Centre national de la Recherche Scientifique in France, and at the California Institute of Technology. He has investigated a broad range of topics associated with the interaction of light with nanoscale struc-

tures. He is widely known for his research observing and explaining how plasmons can control the propagation of light through nanoscale apertures, and for creating and measuring metamaterials (materials that have a negative refractive index).

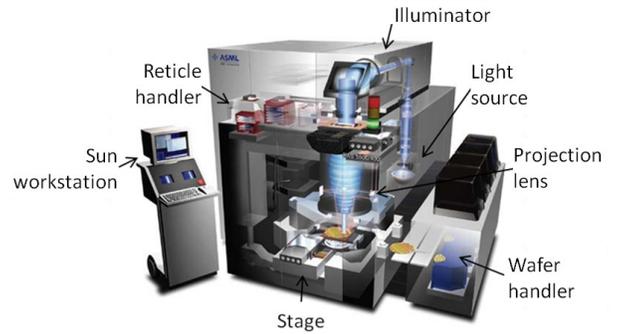
Lezec’s current work is centered on the use of nanoplasmonics, optical metamaterials, and nearfield optics to develop novel nanoscale measurement methods. He pioneered the now widely applied use of the focused ion beam in fabricating plasmonic structures, and it continues to be central to his research.

New Stepper Coming to the NanoFab in 2011

Later this year a new lithography tool will be installed in the NanoFab filling an important gap in our lithography suite. The ASML PAS 5500/275D i-line stepper will be capable of printing 280 nm-sized features across substrates as large as 200 mm in diameter. This model is one of the most advanced i-line steppers available and will provide researchers in the NanoFab with a much-needed option for patterning sub-micrometer features onto their substrates. The stepper will complement the suite of lithography tools already available to users of the NanoFab.

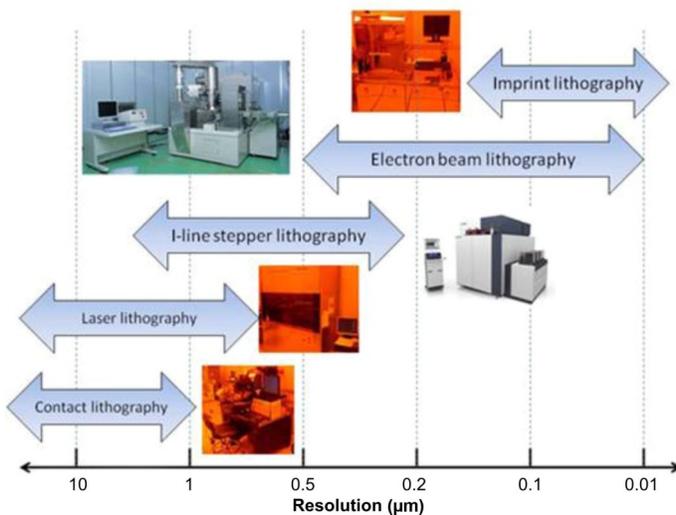
The process by which a photosensitive substrate is exposed in the

stepper involves the projection of features found on a reticle or mask onto the wafer or substrate plane. This projection is achieved with a 5x reduction lens system with variable numerical aperture for high resolution printing of reticle features. The maximum field size of uniform illumination is 22.0 mm x 27.4 mm. Coupled to the projection lens is ASML's AERIAL™ illumination system with variable coherence optics that allow optimum dimension control when printing features down to 280 nm. The light source used in the system is a high intensity 3.5 kW mercury arc lamp filtered to provide 365 nm wavelength light.



The wafer stage is driven by high-speed, linear electromagnetic motors and is interferometrically controlled for precise movement. Wafers are secured to the stage with an electrostatic chuck and the system can be set up to accommodate a variety of wafer sizes as well as small pieces. Global alignment through the projection lens is directly referenced to alignment marks on the reticle, providing overlay accuracy on the order of 40 nm using two global alignment marks. In addition to the conventional top or frontside alignment, the system will also have the option to perform front-to-backside alignment for fabricating structures such as MEMs devices. Control of the exposure system and the wafer and reticle handling systems is through a Sun workstation. Both the wafer and reticle handling is fully automated, allowing exposure job files to be setup and run unattended.

For more information, contact Rich Kasica, 301-975-2693.



John Unguris Recognized as APS Outstanding Referee

John Unguris, a Project Leader in the Electron Physics Group, has been designated a 2011 Outstanding Referee by the American Physical Society (APS). This lifetime award, initiated in 2008, is conferred annually to fewer than one percent of the 42,000-strong referee pool, and is awarded to show appreciation for the essential work that anonymous peer reviewers do

for the journals. It recognizes how the reports and advice of these referees have helped to advance and diffuse the knowledge of physics, while creating a resource that is invaluable to authors, researchers, librarians, students, and readers. Unguris leads multiple projects at the CNST, developing techniques to measure the properties of magnetic nanostructures, with a focus

on spin sensitive electron microscopy. He is an author of over 80 publications, a fellow of the APS, and a recipient of the Bronze Medal from the Department of Commerce. He will be recognized as an Outstanding Referee on March 21, 2011, during the prize and award session at the 2011 APS March Meeting in Dallas, Texas.



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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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News from the NanoFab Office

We are establishing a new NanoFab Office that will house both of our user coordinators, Wade Hall and Jeff Pasternak, thus providing more seamless support to NanoFab users. We invite you to stop by and check the progress of the remodeling. The new office will be located in Building 216/A155, and will share the same contact information:

1-877-NANO-USI (1-877-626-6871)
NanoFabuseroffice@nist.gov



Visit our Booth at these Upcoming Meetings

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|----------------|---------------------------|-------------------|
| March 1 – 2 | SPIE Advanced Lithography | San Jose, CA |
| March 14 – 17 | Pittcon | Atlanta, GA |
| June 14 – 15 | Nanotech | Boston, MA |
| June 28 – 30 | BIO International | Washington, DC |
| July 12 – 14 | SEMICON West | San Francisco, CA |
| August 22 – 24 | SPIE Optics + Photonics | San Diego, CA |

Announcing the next NanoFab Users Meeting

Friday, March 11, 2–4 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.



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