CENTER FOR NANOSCALE SCIENCE & TECHNOLOGY

CNST

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

2010

FROM THE DIRECTOR: THE END OF THE BEGINNING

The publication of this 2010 Report marks the "end of the beginning" of the Center for Nanoscale Science and Technology (CNST). As we conclude our third full year of operation, 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. This Report updates the state of the CNST and highlights the 2009-2010 accomplishments of the Center and its research participants.

As one of NIST's two User Facilities, the CNST strives to make the measurement and fabrication tools needed to advance nanotechnology readily accessible to our stakeholders—industry, academia, NIST, and other government agencies. These tools include both those that are commercially available, available through the shared NanoFab resource, and the next generation being developed by our research project leaders.

As a result of CNST's growth in resources and capabilities over the past two years, we have seen a dramatic growth in our impact. During fiscal year 2010, the CNST had 970 researchers participating in projects using CNST resources, representing a 145 % growth over fiscal year 2008. Participants represented 53 companies, 133 universities, 24 government institutions, and 39 states and the District of Columbia. Corporate researchers working in the NanoFab ranged from a small company, WCH technologies, started by an entrepreneurial inventor who needed the tools and expertise to turn his invention into a working prototype, to IBM, a Fortune 20 company utilizing our resources to reduce the development cycle time of future supercomputer technologies. NanoFab tool use increased by 90 % during the last year alone. The growth in research participation is beginning to be reflected in publications, with the number of participant publications growing by 40 % in the last year.

The CNST research staff is demonstrating major innovations in nanoscale measurement, including break-through advances characterizing the electronic structure of graphene, enabling sensitive spectroscopy on nanophotonic devices, and rapidly tracking and manipulating individual nanoparticles. The quality of their work is demonstrated by the substantial fraction of CNST staff publications that have appeared in high-impact journals, as well as by the professional honors and awards bestowed on the staff.

We appreciate your interest in the CNST and invite you to participate with us in the development of nanoscale science and technology by using our NanoFab or collaborating with one of our researcher staff members. You can keep abreast of such opportunities, our growing capabilities, and our most recent accomplishments by visiting us on the web at www.nist.gov/cnst.

Robert Celotta
December 31, 2010

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The Center for Nanoscale Science and Technology in Brief

The Center for Nanoscale Science and Technology (CNST) supports the U.S. nanotechnology enterprise from discovery to production by providing industry, academia, NIST, and other government agencies with access to world-class nanoscale measurement and fabrication methods and technology. The CNST is the only national nanocenter with a focus on commerce.

The CNST's shared NanoFab resource gives researchers economical access to and training on a state-of-the-art, commercial tool set required for cutting-edge nanotechnology development. The simple application process is designed to get projects started in a few weeks.

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

As the Department of Commerce nanocenter, the CNST provides:

- A unique user facility offering access to the instrumentation, methods, and technical expertise required to make and measure components at the nanometer scale;
- A world-class, shared resource for nanoscale measurement and fabrication widely accessible to researchers from both inside and outside NIST;
- Expertise in a wide variety of disciplines, including physics, chemistry, materials science, molecular biology, computer science, and electrical, mechanical, chemical, and aeronautical engineering; and
- A hub linking the international nanotechnology community to the comprehensive related measurement expertise throughout NIST.

FY2010 RESOURCES

$23 million annual budget

97 staff (87 technical)

NANOFAB CAPABILITIES

- Over 5600 m^2 (60,000 square feet) of laboratory space, including a 1,800 m^2 (19,000 square foot) cleanroom, with 750 m^2 (8,000 square feet) at class 100
- Over 65 major commercial measurement and processing tools in the cleanroom, including electron beam-, photo- and nanoimprint-lithography, laser writing and mask generation, field emission scanning electron microscopy, metal deposition, plasma etching, chemical vapor deposition, atomic layer deposition, and silicon nano/micromachining.
- Additional tools outside the cleanroom, including a transmission electron microscope, multiple focused ion beam systems, and an atomic force microscope.

NEXT GENERATION MEASUREMENT RESEARCH FOCUS AREAS

- Future Electronics
- Nanomanufacturing
- Energy Transport, Storage, and Conversion

CNST ORGANIZATIONAL GROUPS

- Electron Physics
- Energy
- NanoFab Operations
- Nanofabrication Research

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Mission and Metrics

CNST OVERVIEW

The CNST was established in May of 2007 as a unique national 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.

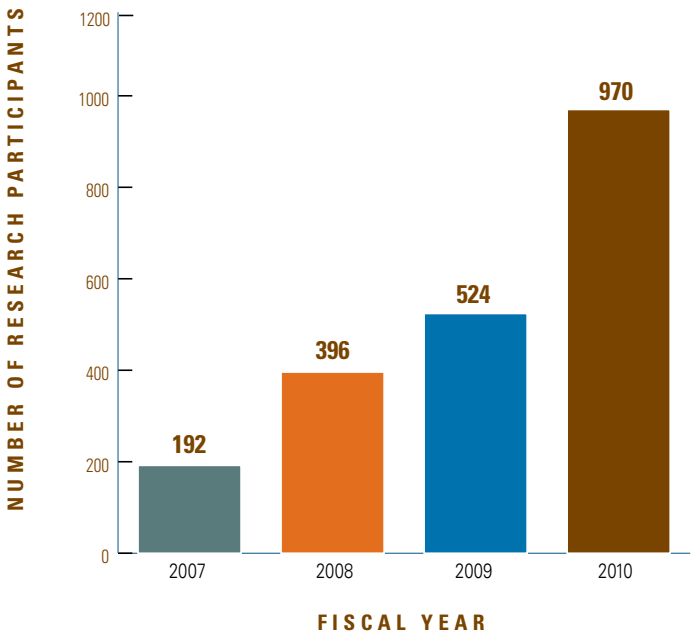
In the few years since its inception, the CNST has become a major national resource for nanoscale science and the development of nanotechnology, and the only national nanocenter with a focus on commerce. Unique in its mission to provide the measurement infrastructure that underlies all progress in this critically important 21st century technology, the CNST serves the U.S. industrial and scientific research communities by providing a venue for highly collaborative, multidisciplinary research, supported by direct access to the required state-of-the-art tools. The continued development of nanotechnology is crucial to firmly establishing U.S. leadership in such diverse fields as energy, manufacturing, information technology, electronics, health, and biotechnology. In the case of energy, nanoscale phenomena lie at the heart of a great many energy production, storage, and transmission processes. Research aimed at optimizing the nanoscale structure of photovoltaic or solar-thermal devices can, for example, have a profound impact by enhancing the conversion of the sun's energy to electricity. Such research demands a multidisciplinary approach and the development and ready availability of advanced tools capable of manipulating and measuring the properties of structures at sizes that can be counted in atoms.

The CNST has been purposely built to uniquely satisfy these demands. Offering many unique measurement capabilities, it provides a collaborative, multidisciplinary research environment focused on national nanoscale measurement needs in such areas as next-generation energy systems, future electronics, nanofabrication, and nanomanufacturing. In this environment, innovative research is advancing the state-of-the-art of nanoscale measurement and fabrication. A critical component of the CNST, the NanoFab, offers open, convenient, and economical access to a comprehensive suite of state-of-the-commercial-art fabrication tools, measurement tools, and processes. The NanoFab is uniquely designed to support both new ventures that require hands-on assistance and training, and experienced practitioners needing access to a reliable and professionally-run research "fab" with a broad selection of advanced,

well maintained tools. Quick access (a few weeks) is available through a simple, merit-based application process. Proprietary research can also be performed.

Having now completed its initial ramp up in staff, equipment, facilities, and processes, the CNST is continuing to expand on its strategic relationships and collaborations with industrial and academic partners. As reported in more detail below, in FY2010 nearly 1000 researchers participated in CNST-supported projects, affiliated with 53 companies, 24 government institutions, 133 universities, and coming from 39 states and the District of Columbia.

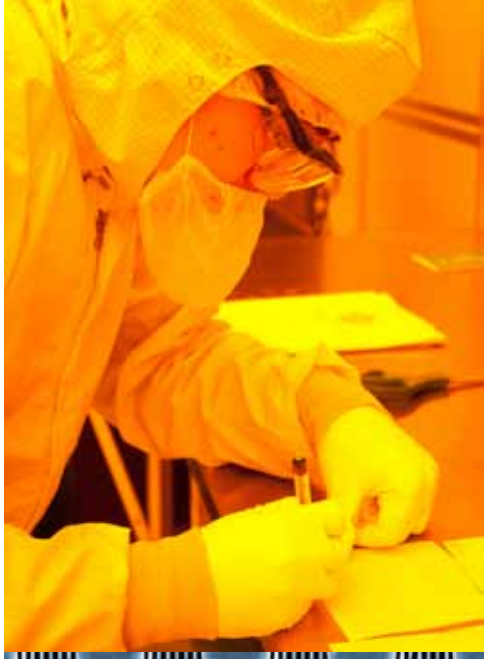
RESEARCH PARTICIPANT GROWTH

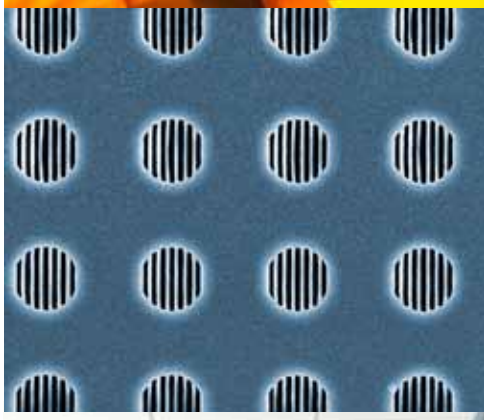


RESEARCH PARTICIPANT AFFILIATIONS BY FISCAL YEAR

Affiliation	2008	2009	2010
Academia	169 (43 %)	216 (41 %)	439 (45 %)
NIST	163 (41 %)	198 (38 %)	311 (32 %)
Other Government	46 (12 %)	55 (10 %)	105 (11 %)
Industry	18 (5 %)	55 (10 %)	115 (12 %)
Total	**396 (100 %)**	**524 (100 %)**	**970 (100 %)**

The number of Research Participants is considered the principal metric for the NIST User Facilities, as it is the most direct measure of the extent that our stakeholders are benefiting from these facilities.





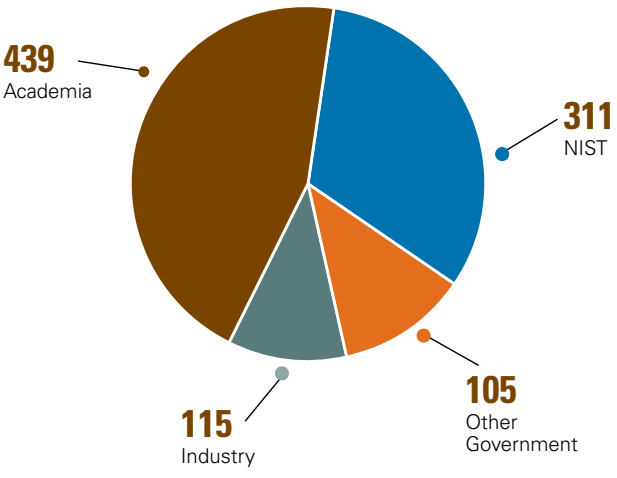
REDUCING MEASUREMENT BARRIERS TO INNOVATION

The organization of the CNST is designed to meet several important goals. To support our central mission, our design must logically support both safe and efficient access to a large tool set and have a significant research capability aimed at furthering the state-of-the-art. We aim to satisfy the first objective through a shared resource, the NanoFab, that operates much like a nanofabrication facility found within the National Nanotechnology Infrastructure Network supported by the National Science Foundation. The cost of maintaining and operating the tools is recovered by charging an hourly rate for tool use that varies depending upon the tool. Users doing proprietary research pay the full cost recovery rate. Otherwise, users supporting the mission of the CNST may qualify for a reduced rate that substantially lowers their charges. We pay the balance of the costs into the NanoFab account from the CNST appropriated research budget. No distinction is made between academia, business, or government in determining the applicability for the reduced rate; the rate is determined by what you are doing, not where you are from. The equitable access policies, especially as applied to industrial researchers, make the CNST a valued resource among nanocenters.

Another goal is to reduce the barriers to using the NanoFab to an absolute minimum. This goal is particularly important because companies, most notably small companies, frequently need rapid access to tools and expertise. Further, many of their needs would not be ranked highly if held to a standard of cutting edge fundamental science in a peer review process. Hence, our entry process uses very simple applications that are reviewed by CNST staff, on a continuous basis throughout the year, for safety, appropriateness, and merit. Typically, only a week or two is required to obtain access; tool use is then on a first come, first served basis via an on-line reservation system.

Researchers working in the NanoFab possess a broad spectrum of knowledge and experience, ranging from seasoned veterans with 30 years of experience to complete novices. The veterans are granted access relatively quickly following a mandatory safety course and examination, and confirmation by our NanoFab staff that they are fully qualified to operate the tools they need. This option is popular with some companies because, if no NanoFab staff members are involved, the company will retain sole ownership of any intellectual property created during that work. At the other extreme, complete novices may choose to be trained on the tools they need to use, have a NanoFab staff member run the process for them, or employ a combination of both alternatives. An initial project discussion with the NanoFab staff and a relevant expert from the research staff helps determine the optimal processing steps. Because many NanoFab researchers will depend on our staff for extensive consultation and help, we have staffed the NanoFab with highly experienced process engineers drawn largely

2010 RESEARCH PARTICIPANT AFFILIATIONS



from the semiconductor industry. Having an experienced staff is also important to maintaining stable and reliable processes in the NanoFab. A small company that has developed a process for making its own structures or device needs to be able to return several months later and be able to duplicate their prior results without having to start a new research project.

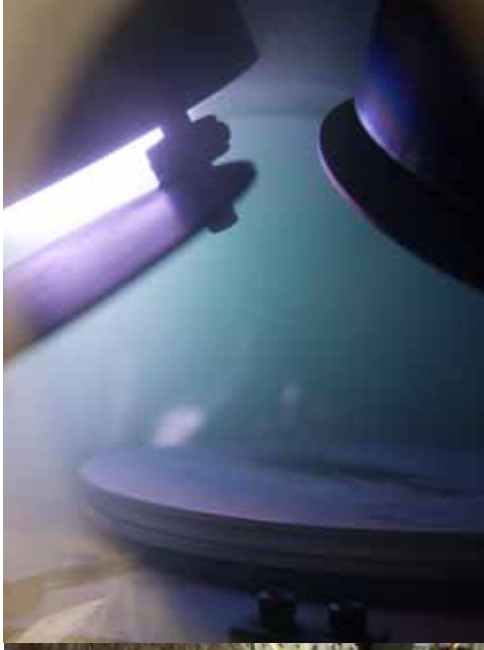
The development of measurements and fabrication methods that go well beyond the current commercial state-of-the-art requires a very different approach. For example, because nanotechnology is a young discipline that is rapidly evolving, it is important that our research approach be agile, allowing it to retarget its research and development objectives relatively quickly in response to perceived need. This need is best met by a relatively flat organizational structure, one that utilizes a multidisciplinary team of core researchers supplemented by a much larger cadre of postdoctoral researchers and a dedicated technical support staff. The frequent arrival of new postdoctoral researchers brings with it new knowledge, experience, and ideas as well as allowing much more rapid changes in direction than might otherwise be possible.

Researchers from outside the CNST can access the advanced tools under development through collaboration: either to aid in their development or to make early measurements using a tool or method not yet available elsewhere. Collaborators include visiting professors, industrial researchers, postdoctoral researchers, graduate students, or undergraduates, with tenures ranging from several days to several years. Even local high school students have successfully participated in CNST projects. Such collaborations are arranged by direct application to the leader of the research project of interest. In addition, formal strategic partnerships have been established. The CNST benefits greatly by the partnerships it has established with other programs.

Nanotechnology being such a broad topic, it is necessary to select carefully from a constantly changing list of priorities for new instrument and method development. One must have taken care, however, to assemble a research staff possessing a broad range of technical expertise and experience in order to maximize the number of tractable problems. For this reason, the CNST has selected experts in a several key nanotechnology fields. Currently, three priority research areas have emerged, based on a gap analysis of nanotechnology measurement needs: future electronics; nanomanufacturing; and nano-enabled energy conversion, storage, and transport devices.

2010 INSTITUTIONS REPRESENTED

Academia	133
NIST	1
Other Government	23
Industry	53
Total	**210**





CNST Measurement Research Staff Expertise

Atomic-scale characterization and manipulation | Joseph Stroscio

Electro-fluidic control of nanoparticles | Benjamin Shapiro

Environmental transmission electron microscopy | Renu Sharma

Fluctuations and nanoscale control | Andrew Berglund

Laser-atom manipulation | Jabez McClelland

Modeling and simulation of nanofabrication | Gregg Gallatin

Nanofabrication and nanomanufacturing | J. Alexander Liddle

Nanomagnetic dynamics | Robert McMichael

Nanomagnetic imaging | John Unguris

Nanomaterials for energy storage and conversion | A. Alec Talin

Nanomaterials for solar fuels and artificial photosynthesis | Veronika Szalai

Nanophotonics | Kartik Srinivasan

Nanoplasmonics | Henri Lezec

Nanoscale electronic and ionic transport | Nikolai Zhitenev

Nanotribology and nanomanufacturing | Rachel Cannara

Optical nanoelectromechanical systems | Vladimir Aksyuk

Theory, modeling, and simulation of nanostructures | Mark Stiles

Theory and modeling of nanomaterials for renewable energy | Paul Haney

Thermoelectrics and photovoltaics | Fred Sharifi

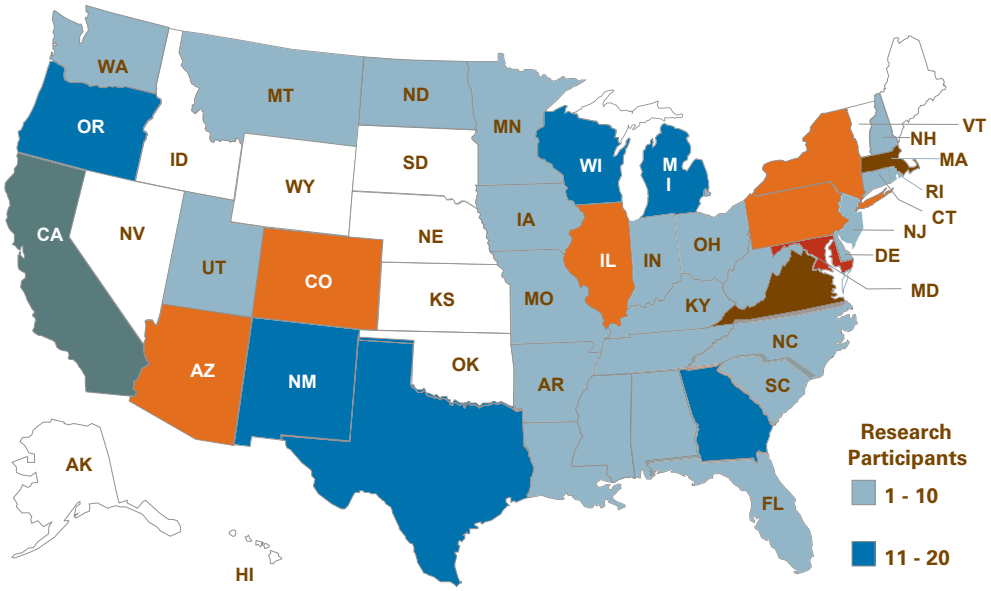
Vibrational spectroscopy and microscopy | Andrea Centrone

KEY METRICS

Over the past two years, we have made major progress in our ability to track key metrics related to the use and impact of CNST resources. This progress results from the development of three databases and associated reporting systems: a comprehensive project management database; a publication and presentation database; and a suite of software tools for managing NanoFab tool usage and accounting.

The project management database, developed specifically for the CNST, is designed to track all CNST-supported projects, the researchers working on each project, and the outputs of each project, including NanoFab projects, CNST staff research projects, and a handful of projects supported extramurally by CNST grants. The database includes detailed technical and administrative information about each project, plus full affiliation and contact information for every research participant. A "Research Participant" is defined as anyone directly involved in a project performed in part at the CNST or through CNST financial support; i.e., someone that would be listed as a coauthor of an associated publication, presentation, patent application, etc. The number of Research Participants is considered the principal metric for the NIST User Facilities, as it is the most direct measure of the extent that industry, academia, NIST, and other government agencies are benefiting from these facilities.

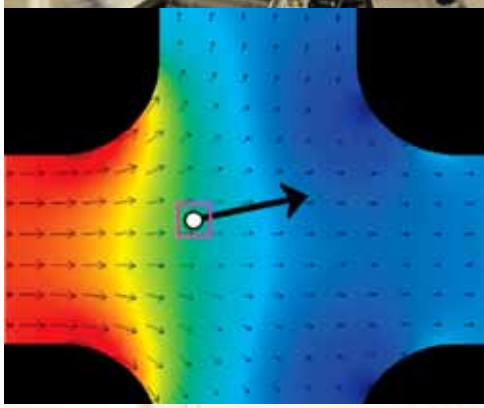
2010 GEOGRAPHIC DISTRIBUTION OF RESEARCH PARTICIPANTS



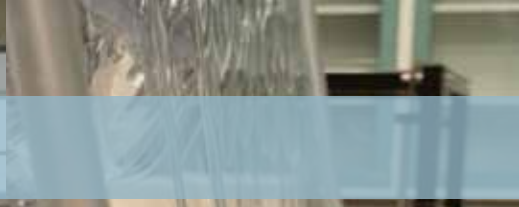
Publications are a trailing indicator of the impact of research, typically reflecting accomplishments in the prior year or two; a complete list of publications in FY2009 and FY2010 is included later in this Report. Although it is still too soon to analyze the impact of this work (e.g., through citation analyses), the year-over-year growth of CNST publications and their quality bodes well for the future, with approximately a quarter of our staff publications each year appearing in high-impact and letters journals.

The third data set we are collecting, from the NanoFab, is extracted from the CORAL suite of software tools (Common Object Representation for Advanced Laboratories) developed at the Stanford Nanofabrication Facility. This suite includes software for scheduling tool use, enabling the access of individual tools on a user-by-user basis (i.e., after training is completed), tracking tool usage time, and billing for each tool usage.

> I had a great visit to NIST, and I was able to quickly develop a process for our samples. This was greatly facilitated by the fact that I was able to leverage previous developmental work that had been performed at NIST. I was very impressed with your cleanroom facilities, the support provided by your staff, and the orientation process (safety classes, tours).
>
> *Lee Oesterling*
> *Electronics, Sensors, & Information Systems*
> *Battelle Memorial Institute*







Over the past year most tools were interlocked with the CORAL system, with others in the process of being added. A dashboard is being developed, expected to be fully operational in FY2011, which will enable enhanced analyses of tool usage, helping us to better optimize the delivery of services to our NanoFab customers. Preliminary analysis indicates that overall use of the NanoFab grew by approximately 80 % in FY2010 over FY2009.

ENCOURAGING A STRONG SAFETY CULTURE

The CNST strives to provide a safe place to work for our staff and visiting researchers, including those using the NanoFab. We have established extensive policies and procedures with the goal of ensuring an open and supportive work environment where all staff members are knowledgeable about Environment, Safety, & Health (ES&H) policies and practices and feel free to report any issues or concerns to the CNST management. We are proud that our safety program was recognized by the recent Final Report of the NIST Blue Ribbon Commission on Management and Safety II, which noted "The Center for Nanoscale Science and Technology (CNST) has an extensive set of rules and procedures for those who work in their facilities… NIST should adopt/adapt these types of policies and procedures for the entire organization."

The main elements of our safety program are as follows:

Defining roles and responsibilities. We assign clear safety roles, responsibilities, accountabilities, and authorities essential to creating and maintaining a safe working environment; for example, each Project Leader is responsible for ensuring that nobody works in his/her lab without the education and training required and specified in the laboratory manual and hazard analyses.

Providing training required to work safely. All technical staff must complete a suite of on-line training modules before starting work in any laboratory. This basic training, typically completed by one's second or third day at NIST, is supplemented by specific, hands-on training on the instrumentation within each laboratory, as specified in each laboratory's manual and hazard analysis.

Before working in the NanoFab, every researcher must complete NanoFab safety training and orientation and pass a safety exam, with annual retesting required to maintain NanoFab access. NanoFab tools are individually interlocked to insure that a researcher can only access tools that he or she has been trained on and is authorized to use.

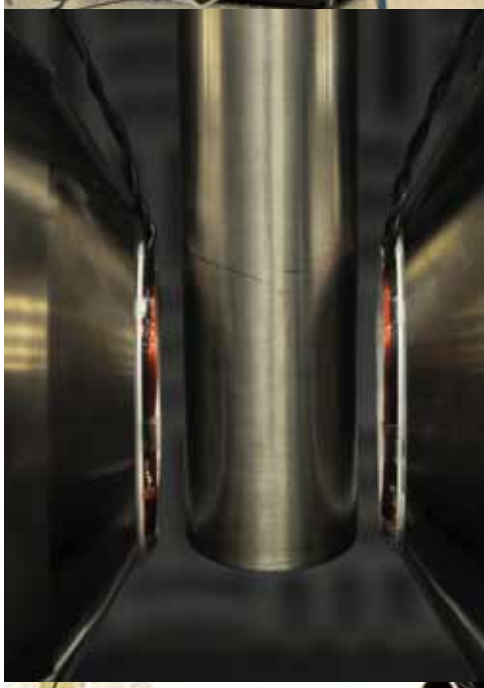
Performing comprehensive hazard analysis and control for all workplace activities. A comprehensive analysis must be completed, including multiple levels of management review, before any new work begins. During this process potential hazards are identified, a detailed analysis of the hazards and worst-case scenarios is performed, and detailed operating procedures, emergency plans, and required controls are developed, documented, and implemented. For laboratory activities involving nanoparticles, the CNST follows best practices as suggested by the National Institute for Occupational Safety and Health (NIOSH). In a bin outside every laboratory there is a laboratory manual that includes an authorized access list, personal protective equipment (PPE) guidance, and hazard assessment and control documentation.

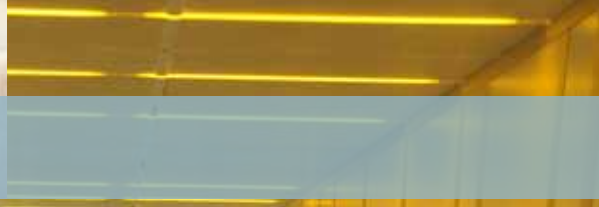
Each bin also holds a notebook containing the appropriate Material Safety Data Sheets. Within the NanoFab, in addition to the tool-by-tool analyses performed as part of the CNST-wide process, the safety of each proposed project is assessed during the technical review.

Conducting management observations and inspections. The NanoFab is monitored at all times via an extensive closed-circuit video monitoring system by either the NanoFab staff or the NIST Emergency Services Division. All CNST managers are responsible for recognizing good safety practices, identifying potential safety improvements, and reporting these to a Center safety email distribution list. All NanoFab laboratories are inspected daily for safety and housekeeping problems, with all other laboratories inspected weekly by the designated manager of each space. In addition, each Group Leader conducts a quarterly safety inspection of all space using a standard checklist, and the Center Safety Representative follows up on outstanding issues. Finally, the Director, Deputy Director, and Group Leaders conduct an annual, all-space safety inspection.

Encouraging communication and a culture of safe work practices. Laboratory safety is considered everyone's responsibility in the CNST, and all staff and researcher participants are expected to help enforce the rules throughout all laboratories. To promote this culture, the CNST has a dedicated, full-time Center Safety Representative who, among other responsibilities, oversees all chemical purchases, including ordering, delivery, and labeling through a centralized chemical receiving room. The CNST Director makes weekly laboratory visits to learn about research activities, with a safety conversation an integral part of each visit.

> "The CNST has an extensive set of rules and procedures for those who work in their facilities… NIST should adopt/adapt these types of policies and procedures for the entire organization."
>
> *NIST Blue Ribbon Commission on Management and Safety II*





The NanoFab: A Shared National Resource

NANOFAB OVERVIEW

The NanoFab provides access to state-of-the-art, commercial nanoscale measurement and fabrication tools and methods, along with associated technical expertise, to industry, academia, NIST, and other government agencies in a shared-access, shared-cost environment. It enables processing and characterization of a wide range of nanoscale materials, structures, and devices critical to the nation's measurement and technology needs. The NanoFab also fosters internal collaboration in nanotechnology across NIST's laboratories and external collaboration with NIST's partners through its shared environment.

The NanoFab has the following key attributes:

- Rapid Access. The streamlined application process is designed to get projects started in a few weeks.
- Extensive Process Support and Development. The NanoFab is operated by a professional staff of process engineers and technicians with over 240 years of collective experience. The NanoFab offers a broad catalogue of established processes, along with assistance in the development of new processes.
- Training and Education. The customer-oriented NanoFab staff members are available for expert consultation and hands-on training for all tools and processes.
- Shared Expertise. As a shared national resource open to all, the NanoFab brings NIST scientists together with industry, government, and academic researchers from across the spectrum of nanotechnology applications, enabling the rapid exchange of ideas and best practices.
- No Inherent Claims on Intellectual Property Rights. The CNST does not claim any rights to intellectual property used or developed in the NanoFab, unless a CNST federal employee is a co-inventor.

The NanoFab consists of a large clean room and several more specialized laboratory modules in an adjacent building. The NanoFab cleanroom occupies 1,800 m^2 (19,000 square feet) of floor area, which includes 750 m^2 (8,000 square feet) of class-100 space. The NanoFab cleanroom contains over 65 fabrication and processing tools, providing electron beam-, photo- and nanoimprint-lithography, laser writing and mask generation, an i-line stepper, field emission scanning electron microscopy (SEM), metal deposition, plasma etching, chemical vapor deposition, atomic layer deposition, and silicon micro/nano-machining. Additional tools, which are located outside the cleanroom, include three dual beam focused ion beam systems, an atomic force microscope, and a Titan 80-300 keV analytical transmission electron microscope (TEM) system with both X-ray and electron energy loss analytical capabilities. The CNST has a particularly strong capability in electron beam lithography. An ultra high resolution electron beam lithography system provides for direct write nanoscale feature development and mask writing capability from small samples up to 300 mm-diameter wafers. A second electron beam lithography tool is designed for direct writing with a 3 nm spot size on substrates up to 150 mm x 190 mm in size. For less demanding lithography tasks, a SEM-based electron beam writing tool is also available. Additionally, a state-of-the-art nano-imprint lithography system can produce nanoscale features in soft materials using a hard mask made with the electron beam writer. These tools are interlocked and controlled using CORAL.

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

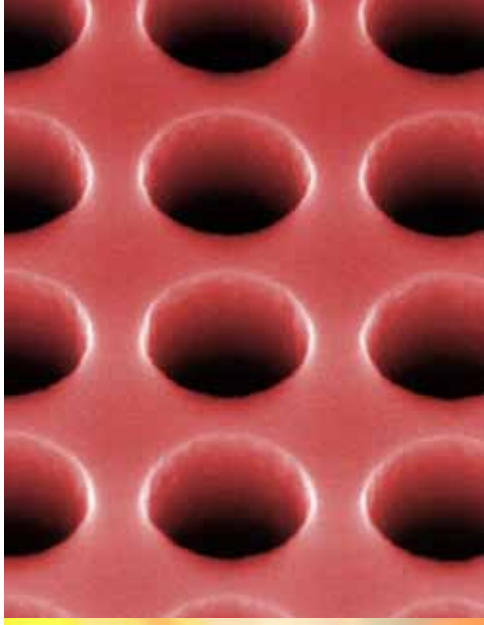
The NanoFab is staffed and open from 7 am to midnight, Monday through Friday, subject to NIST campus access requirements. Access hours for researchers from outside NIST depends on a variety of factors, including citizenship and the length and frequency of anticipated visits to the NanoFab, but the NanoFab Manager works with each researcher to ensure that all work can be scheduled during permitted access times. For researchers with out-of-hours access to the campus, the NanoFab is available at all times, seven days a week, subject to the approval of the NanoFab Manager and with a requirement that work be done under the buddy system, with at least two researchers present at all times.

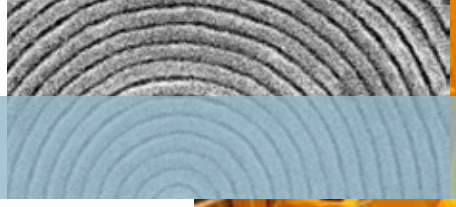
ESTABLISHING A NANOFAB PROJECT

Someone interested in starting a project at the NanoFab is strongly encouraged to begin by discussing the project with the NanoFab Manager. If the project leader is new to nanoscale measurement or fabrication, the NanoFab Manager first discusses possible ways to make or measure the project's nanoscale components. For a researcher experienced in the field, the Manager discusses what tools and processes are available to meet the project's needs. Indeed, the prospective project may well benefit by the processes or know how developed by other NanoFab projects. The NanoFab Manager also provides an overview of the application process and payment options, along with the orientation and training requirements. When someone is ready to begin the new project, the project leader fills out a Project Application and returns it to the Facility User office, where the applicant is guided through the rest of the process.

Each project application is assessed by a Technical Review Committee comprised of NIST experts in nanoscale measurement and fabrication. The project is reviewed to ensure it can be performed safely in the NanoFab, that it will not compromise the use of any tools (e.g., by contamination), that it will not unduly prevent others from using necessary tools, and that it is feasible with the available resources. Finally, it is assessed as to the merit of the overall goal of the project, in order to prioritize projects in the event that needed resources are oversubscribed.

The project application process averages about two weeks from application submission to project approval, at which point an initial payment must be made and an orientation session scheduled. There are a number of factors that affect how quickly someone can come to NIST and get started in the NanoFab. First, we review applications every Tuesday; therefore, if an application is submitted by 5 p.m. on a Monday, it will be reviewed that week. Second, as soon as possible, an applicant should discuss with his or her organization and one of the NanoFab Facility User Coordinators the most efficient way to transfer to the NanoFab funds sufficient to cover the estimated cost of the project. Finally, each researcher planning to work in the NanoFab will need to be cleared by NIST Security to access the NIST campus and NanoFab laboratories. If a researcher already has a NIST badge as an employee or Guest Researcher, there are no additional access requirements. For a U.S. citizen or Permanent Resident, a badge providing access can be issued within a few days of project approval. For a foreign national, it may take at least an additional 30 days to be approved for a badge. However, during that time we can usually arrange for a one or two day




visit to complete orientation and some initial training in order to help the researcher get started as quickly as possible.

New NanoFab researchers are required to complete three levels of training to begin working independently in the NanoFab, as follows:

1. Basic orientation covers operations and safety protocols for use of the NanoFab. The class is a two hour, hands-on training session given at the CNST every Monday morning. Attendance can be scheduled through the NanoFab Facility User office.
2. New researchers must also pass a comprehensive safety exam. The on-line exam takes about 30 minutes and can be scheduled through the NanoFab Facility User office any time after completion of the basic orientation.
3. New researchers are also required to have specific tool training to be certified on each piece of NanoFab equipment their work requires. Tool certification sessions can be scheduled by contacting the primary or secondary owner of the tool you wish to use. Note that a CORAL account is required for tool certification.

NANOFAB COSTS

There are three types of hourly rates charged to every NanoFab researcher to recover the costs of performing the work: Specific Tool Use, Cleanroom Use, and Process Assistance (when applicable). Each rate is computed for full cost recovery, including the cost of the NanoFab staff time required plus the operating costs. The operating costs include the costs of any maintenance contracts, routine maintenance and repairs (both scheduled and unscheduled), and accessories and consumable supplies. After a full cost recovery rate is computed, for projects that advance the CNST mission, a reduced cost percentage is applied to compute the reduced rates charged to those projects. The eligibility criteria for reduced rates are discussed below. As a matter of NIST policy, proprietary projects are not eligible for the lower rates and must pay the full cost for work performed in the NanoFab. The charges for every NanoFab project are based on the same rates, including projects led by NIST employees (CNST research staff included).

NIST requires an initial funding authorization that is sufficient to cover the estimated charges for each new project. However, researchers are only billed for the actual charges incurred. The NanoFab operates on a monthly billing cycle and provides reports of each project's tool usage and costs at the close of each cycle. The NanoFab accepts payment by a variety of methods, including credit card and purchase order. The NanoFab Manager typically works with new applicants during the initial application process to estimate the expected charges for each project.

Non-proprietary projects may be eligible for reduced rates, with the balance of the full cost paid by the CNST from its appropriated research budget. All applicants, including those from NIST, can request consideration during the application process, and the project will be rated on the extent that it contributes to the CNST mission by developing and/or applying nanoscale measurement and fabrication methods to further the development of nanotechnology. All such requests are decided on a case by case basis, typically within 10 days of an application being submitted, following review by a CNST committee and final approval by the CNST Director.

NANOFAB OPERATIONS

The NanoFab is operated by a team of experienced engineers and technicians dedicated to customer service, with two process engineers remaining on site on weekday evenings until midnight. The team is focused on supporting the researchers working in the NanoFab by running the tools and establishing consistent baseline processes. The staff also develops new processes in response to researcher needs. Members of the CNST research staff also apply their expertise to help evaluate and consult on NanoFab projects.

Effective communication between our research participants and the NanoFab staff is essential to NanoFab operations. In addition to periodic informational meetings to which all NanoFab researchers are invited, we maintain a standing NanoFab Researcher Committee. This committee is composed of two representatives from each of the four NIST Laboratories, a representative from the NIST Center for Neutron Research, an external NanoFab researcher, a representative from the NanoFab Facility User office, and the NanoFab Manager. The committee Chair rotates annually among the members. The committee helps keep the Manager informed about general issues in the NanoFab, including the impact of operating policies and procedures, training needs, tool maintenance, new tool requests, and the general level of satisfaction among the NanoFab research community. The Committee also provides a useful conduit for information from the NanoFab back to the research community.

NanoFab Operations Group
December 2010

Vincent Luciani | NanoFab Manager
Matthew Gonzales | Administrative Assistant
Wade Hall | Facility User Coordinator
Jeff Pasternak | Facility User Coordinator

Process Engineers and Technicians

Jerry Bowser
Laurence Buck
Marc Cangemi
Lei Chen
Justin Dickinson
Gerard Henein
Michael Hernandez
Richard Kasica
Chester Knurek
Alline Myers
Eileen Sparks
William Young

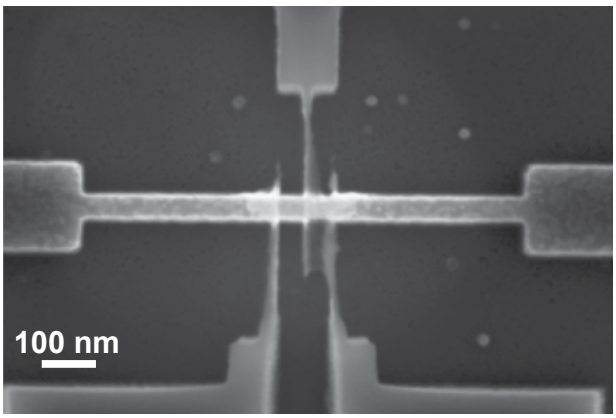
The charges for every NanoFab project are based on the same rates, including projects led by NIST staff members.

NANOFAB CAPABILITIES: TOOLS AND PROCESSES

NanoFab researchers will find the tools well maintained and fully operational, with standard processes well under control. Statistical Process Control charts and standard processes are used to measure and control tool performance. All standard processes are documented for easy reference by users, and new processes are constantly being developed. The NanoFab community also benefits from the many processes developed by non-proprietary projects that are made available to everyone. The NanoFab provides a standard recipe template and encourages all researchers who develop a useful new process to document it for use by others.

The NanoFab is equipped with a comprehensive set of nanofabrication and analytical tools. The toolset is generally equipped to handle wafer sizes up to 150 mm in diameter, including small and irregularly-shaped samples. Many of the tools can handle 200 mm-diameter wafers. A complete list of the toolset is available at the NanoFab web site. An overview of the key capabilities associated with the NanoFab's major tools and processes is provided below, including major new capabilities added in the past two years and those to be installed in 2011.

Lithography—The NanoFab has extensive lithographic capabilities and more on the way, including two full time electron beam lithography systems; a SEM based electron beam writer; nano-imprint lithography; a laser writer for mask or direct write lithography; and two i-line contact aligners. Most standard resists, both positive and negative, and their associated developers are readily available. Ancillary equipment, such as hexamethyldisilazane (HMDS) primers, a critical dimension (CD) measurement tool, and spin coaters is also available.



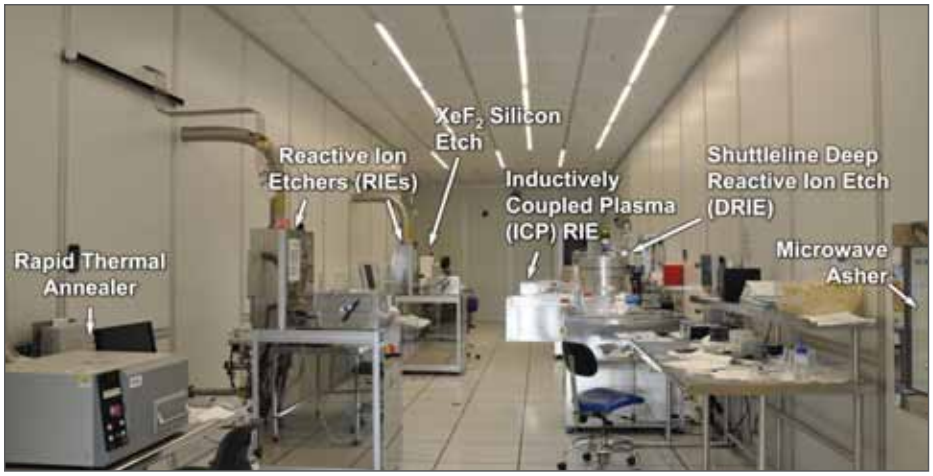
The advanced lithography capabilities in the NanoFab are demonstrated by this silicon nanoelectronics structure, where three separate layers of nanoscale lithography have been implemented with an overlay accuracy ≤ 20 nm. *Courtesy of Neil Zimmerman, NIST.*

New Capabilities:

- A CD measurement tool (mentioned above) that provides fast repeatable line width measurements of developed resist and final patterned structure for accurate calculation of process bias.

- A HMDS vacuum prime oven; vacuum deposition of HMDS resist adhesion promoter maximizes resist adhesion.
- A 5x reduction, i-line stepper will complete the NanoFab's lithography tool set, providing a cost effective solution for creating geometries from the micrometer to nanometer scale. The stepper, along with our mask writer, will make an extremely powerful combination for rapid prototyping (2011).
- An automatic developer will provide the uniform and repeatable development necessary for robust lithography processes (2011).

Reactive Ion Etching (RIE)—The NanoFab has a strong RIE capability that includes two Unaxis parallel plate fluorine based systems; a Unaxis inductively coupled plasma (ICP) chlorine based system; a Unaxis ICP fluorine based deep silicon etcher; a XeF_2 silicon etcher; and an oxygen barrel asher. Capabilities include basic dielectric etching, metal etching, III-V etching, and the Bosch deep silicon etch.



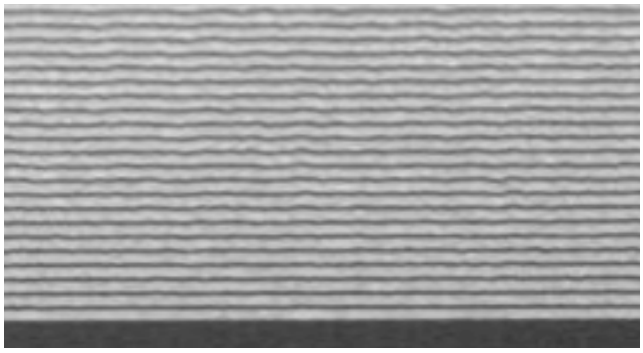
The dry etch bay in the NanoFab cleanroom.

> I would just like to say that my experience at the CNST was phenomenal. I was trained quickly, and it was clear that the priority on your end was to get me in the NanoFab and get my work completed as soon as possible. On that note, Marc and Lei were instrumental in helping me work out recipes quickly so that I could spend my time in production mode rather than trouble shooting. Their knowledge and experience were huge assets. Finally, everyone was extremely nice and pleasant to work with. I won't hesitate to recommend the CNST to anyone in need of a top-notch fabrication facility.
>
> *Jered Haun*
> *Massachusetts General Hospital*
> *Harvard Medical School*

New Capabilities:

- Two Oxford ICP RIE systems were added to our etch suite. These two etchers provide cryogenic etching capabilities, improved chlorine based metal etching, and a III-V etching process that yields super smooth sidewall for photonic devices.
- A new broad beam ion mill etching tool will be invaluable for patterning materials that cannot be etched by RIE (2011).

Thin Film Deposition—The NanoFab can deposit many types of thin films, including metals, metal alloys, various dielectrics, silicon and parylene. The NanoFab staff can help you match the right film with the best deposition technique for your application. Our deposition methods include sputtering; electron beam evaporation; thermal evaporation; plasma enhanced chemical vapor deposition (PE-CVD); and low pressure CVD (LPCVD). Our thin film deposition tools include two Denton sputter systems; two Denton electron beam evaporators; a Unaxis PECVD system; three LPCVD tubes (silicon nitride, silicon dioxide and polysilicon); and a Parylene deposition system.



The NanoFab's process control enables complex layered structures where excellent control over layer thickness, uniformity, and repeatability is essential, such as the structure in this SEM image, composed of 25 periods of alternating 27 nm-thick a-Si and 94 nm-thick Ag films. The NanoFab has deposited similar structures with thicknesses below 3 nm per layer. *Courtesy of Henri Lezec and Kenneth Chau, NIST.*

New Capabilities:

- An Oxford Atomic Layer Deposition (ALD) system uniformly deposits a monolayer at a time of a material across wafers of up to 200 mm in diameter. We currently have processes for hafnium oxide, platinum, silicon dioxide, and aluminum oxide. Several additional materials will be available (2011).
- *In-situ* phosphorus doping has been added to our polysilicon LPCVD system, enabling our complementary metal-oxide-semiconductor (CMOS) researchers to deposit highly doped polysilicon for gate material.

Diffusion/Oxidation—The NanoFab can support diffusions and oxidations at temperatures up to 1200 °C on wafer sizes up to 150 mm in diameter. Processes include normal wet and dry oxidations, forming gas anneals and inert ambient diffusions. Some tubes are equipped with Trans-LC as a chlorine source for high purity oxides, such as gate oxides for CMOS applications. One furnace stack is reserved exclusively for CMOS applications. This stack is subject to regular oxide integrity monitoring by measuring the mobile ion concentration of the resulting oxides. A rapid thermal processor (RTP) is also available for very short, high temperature anneals. Ramp rates of 200 °C per second are easily achieved.

New Capabilities:

- One tube slot has been converted to support temperatures up to 1250 °C for extended periods of times, as is often required for silicon carbide processing.
- Ultra clean internal H_2/O_2 torches are available to improve uniformity, purity, and repeatability of wet oxidation processes.

Wet Processing—There are many nanofabrication processes that involve the use of chemicals to clean or etch various materials. The NanoFab stocks the most commonly used chemicals and the

The inspection bay in the NanoFab cleanroom.

tools needed to use them safely, including several wet benches and fume hoods available for these purposes. Some fume hoods are dedicated for solvent use, whereas others are suitable for the many other aqueous acid and base solutions that are required for processing. The NanoFab also provides dedicated equipment for RCA cleans, KOH etching, and tetramethylammonium hydroxide (TMAH) etching. Ancillary equipment, such as spin-rinse-dryers and critical point dryers, is available as well.

New Capabilities:

- An HF vapor etcher and a second critical point dryer specially designed for very small parts.

Inspection and Metrology—A necessary element of the NanoFab is the ability to inspect and measure Nanoscale structures. The NanoFab provides several different techniques for this purpose, including a field emission SEM (FESEM); a tabletop SEM; a spectroscopic ellipsometer; two reflectometers; a mechanical profilometer; a contact angle goniometer; two atomic force microscopes; a film stress measurement tool; and several optical microscopes. A dual beam FIB augments our inspection capabilities as well as being a process tool.

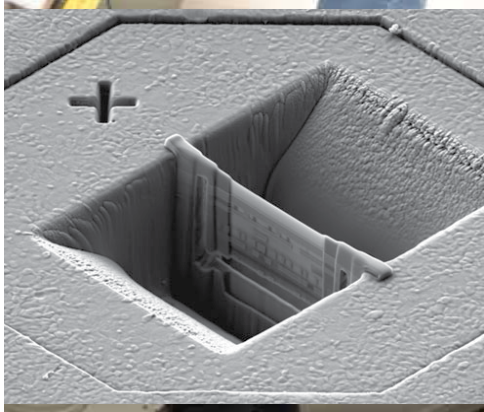
New Capabilities:

- An energy dispersive spectroscopy (EDS) system has been added to our FESEM to provide elemental analysis capability, including a compositional mapping tool.
- A state-of-the-art FEI Titan 80-300 TEM will provide 0.1 nm resolution at magnifications > 1,000,000x (2011).
- Two FEI Helios NanoLab 650 DualBeam FIBs offering extreme high resolution 2D and 3D characterization, 3D nanoprototyping, and high quality TEM sample preparation (2011).

Post Process—When processing wafers, often the researcher will need to thin the wafers, separate the individual dies, and then provide connections to outside circuitry or test fixtures. The NanoFab post process lab is equipped with some specialty tools for these purposes, including a dicing saw and wire bonder.

New Capabilities:

- A chemical mechanical polishing (CMP) tool for thinning wafers or for planarizing interlevel dielectric layers for multi-level metallization (2011).



CNST Research: Creating the Next Generation of Nanoscale Measurement Tools

While the NanoFab provides access to cutting-edge commercial tools and processes, CNST instrumentation scientists and engineers are creating the next generation. These talented staff members make their innovative instruments and methods available through collaboration with the world's leading scientists to make and measure the nanoscale materials and devices of the future. As briefly discussed above, based on the collective input of our stakeholders regarding the key measurement barriers to advancing nanotechnology and the current capabilities and needs of NIST's four Laboratories, we are currently emphasizing the following three research areas.

Future Electronics. In support of continued growth in the electronics industry beyond complementary metal-oxide-semiconductor (CMOS) technology, CNST researchers are developing new methods to create and characterize devices, architectures, and interconnects for graphene, nanophotonic, nanoplasmonic, spintronic, and other future electronics.

Nanomanufacturing. The Center is advancing the state of the art in nanomanufacturing by developing measurement tools and fabrication methods for both lithographic ("top-down") and directed assembly ("bottom-up") approaches.

Nano-enabled Energy Storage, Transport, and Conversion. The CNST is creating new methods for elucidating light-matter interaction, charge and energy transfer processes, catalytic activity, and interfacial structure in energy-related devices.

Although the measurement research staff is organized into three Groups, each staff member applies his or her multidisciplinary expertise across multiple areas. An overview of the ongoing research in each Group follows, including key accomplishments from the past two years and emerging areas for new programs.

ELECTRON PHYSICS GROUP

The Electron Physics Group (EPG) conducts a wide range of cross-disciplinary instrumentation research focused on the development of innovative measurement techniques for nanotechnology. Building on a rich history of influential research in electron-surface interactions, scanned-probe microscopy, electron-atom scattering, electron optics, and electron spectroscopy established while within NIST's former Physics Laboratory, the EPG has expanded over the years into new technology areas as new measurement needs have arisen. Current research constitutes a significant portion of the CNST's major thrust in future electronics, with strong efforts in nanomagnetics measurements for beyond-CMOS state-variable and information storage development; state-of-the-art scanned-probe spectroscopy instrumentation for measurements of novel materials for next-generation devices; and theoretical studies of phenomena such as spin transfer torques and electron transport which guide development of new measurements enabling future device paradigms.

Nanomagnetics—EPG research in nanomagnetics is currently aimed at addressing two of the most important measurement issues in the development of magnetic nanotechnology: imaging the magnetic structure within nanostructures, and measuring magnetization dynamics at the nanoscale. The properties of nanoscale magnetic structures can differ from those of macroscopic magnets for several reasons. Surfaces, edges, and interfaces become dominant, lithographic dimensions approach fundamental magnetic exchange lengths, and nanoscale fluctuations in material properties such as grain structure are also more important.

Ultimately, these nanoscale magnetic properties are not only a source of potential problems, but are also the basis for the development of new, higher density magnetic data storage technologies, and new magneto-electronic and spintronic devices. We seek to develop measurement methods that can be used to provide an understanding of these nanomagnetic properties at a fundamental level by measuring how the electron spins, which are responsible for the magnetic properties, are arranged in a nanostructure, and how these electron spins respond to stimuli such as fields, currents, and mechanical stresses.

Electron Physics Group
December 2010
Jabez J. McClelland | Group Leader
Teresa Figgs | Administrative Assistant

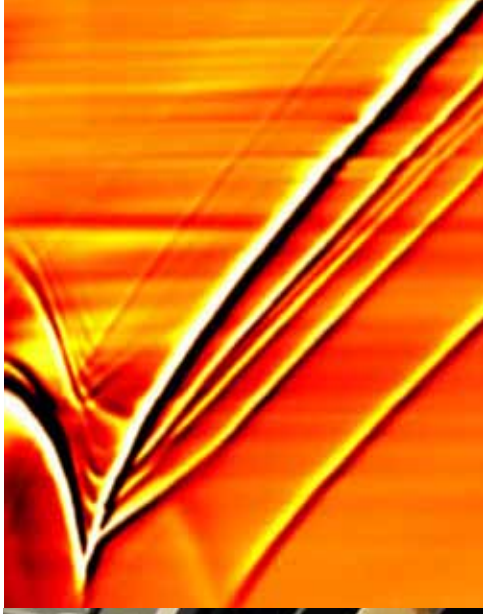
Project Leaders
Robert McMichael
Mark Stiles | NIST Fellow
Joseph Stroscio | NIST Fellow
John Unguris

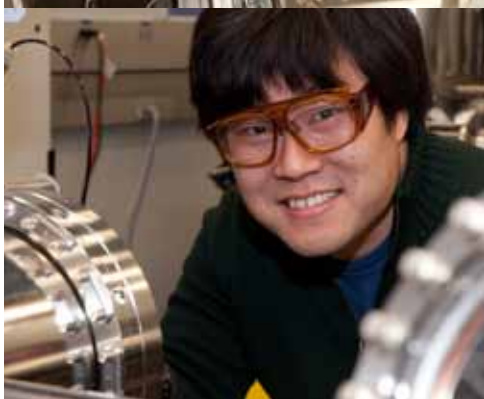
Visiting Fellows
Young Kuk | Seoul National University
David Penn | NIST Emeritus
Daniel Pierce | NIST Fellow Emeritus

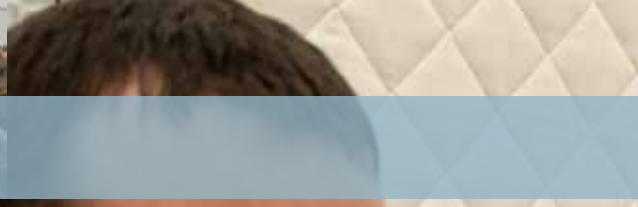
Postdoctoral Researchers
Shaffique Adam
Samuel Bowden
Jungseok Chae
Han-Jong Chia
Nikolai Klimov
Brenton Knuffman
Niv Binyamin Levy
Benjamin McMorran
Adam Steele
Tong Zhang

Graduate Researchers and Interns
Parakh Jain | Poolesville High School
Kevin Kubista | Georgia Institute of Technology
David Lee Miller | Georgia Institute of Technology

Engineering and IT Support Staff
Alan Band
Steve Blankenship
Barbara Coalmon
Frank Hess
Glenn Holland
Matthew Manganello
David Rutter

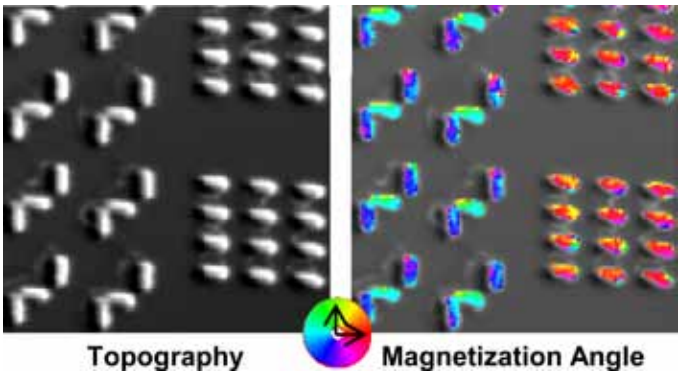






Our primary magnetic imaging technology is the NIST-developed Scanning Electron Microscopy with Polarization Analysis (SEMPA) method, which provides a direct, high resolution view of the magnetization in magnetic nanostructures with 10 nm spatial resolution, 1 nm depth resolution, and equal sensitivity to all three components of the magnetization vector. In recent work we have demonstrated a new approach to depth-profiling the magnetic structure in several materials relevant to perpendicular magnetic storage media and spintronic applications, making use of a combination of SEMPA imaging and low-energy ion milling. For example, SEMPA measurements on patterned Co/Pd multilayer recording media with graded magnetic anisotropy, prepared by our collaborators at the University of California, Davis, revealed how the domain structure evolved at different depths in the graded films (see the Project Highlight, Imaging 3D Magnetic Nanostructure in Ferromagnetic Multilayers). SEMPA was also used to study a synthetic antiferromagnet consisting of an antiparallel coupled Co/Ru/Co multilayer, used by Michigan State University to test ferromagnet/superconductor proximity effects.

SEMPA is also being used to image the magnetic state of nanoscale patterned ferromagnetic thin films that are the building blocks of magnetic logic circuits. Such circuits are potential candidates for use in low power applications in future electronics. So far we have successfully measured static magnetization arrangements in structures provided by the University of Notre Dame and the University of South Florida. We are currently extending the SEMPA measurements to image the magnetic state of active devices—devices in which the magnetic state can be switched using magnetic fields or current-induced spin torques.



SEMPA images from a patterned array of 100 nm-wide elements used for magnetic logic.

As part of our magnetic imaging program we are also pursuing new approaches that will allow us to improve spatial resolution to well below 10 nm. To this end, we have investigated the production and application of electron vortex beams in a TEM. These electron beams have angular momentum that can interact with a ferromagnetic sample in a manner similar to the magnetic circular dichroism contrast obtained using synchrotron radiation, but potentially with the higher spatial resolution of the TEM. Electron diffraction gratings with well defined dislocations were produced in the NanoFab and used to successfully generate electron beams with quantized angular momentum of up to 100 $\hbar$ per electron. These electron vortex beams are analogous to optical vortex beams and are expected to be similarly useful in various electron microscopy applications from magnetic imaging to imaging weak-phase biological objects.

Our research program to develop magnetization dynamics measurements has demonstrated the ability to provide essential information about spin dependent transport, magnetic resonances, and magnetic switching at the nanoscale. Measurements of these properties create a foundation for the design and development of a wide variety of magnetic nanodevices and electron spin-based electronics.

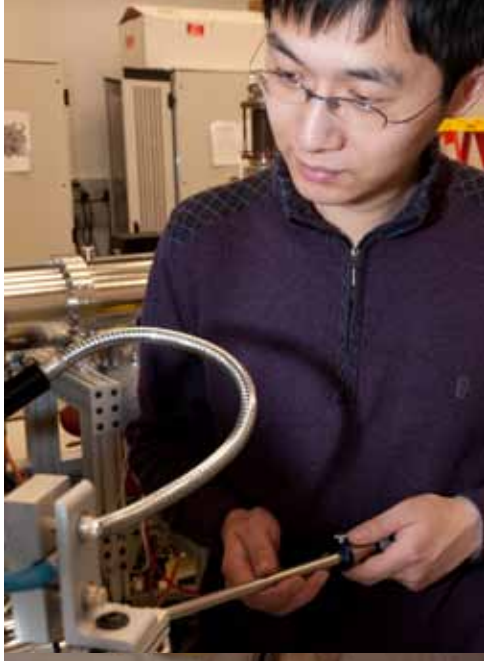
We have developed a method to measure the polarization of electrical current carried in magnetic metals via the "spin wave Doppler effect." In the simplest terms, the electrons that carry charge in an electrical current also transport magnetic moment via the electron spin. The resulting drift velocity of the magnetization adds vectorially to the velocity of propagating spin waves in a way that is very nearly identical to the classical Doppler shift of wave propagation in a moving medium. By measuring how the propagation of spin waves depends on the sign and magnitude of current, it is possible to determine the polarization of the current; i.e., the relative magnitude of currents carried by up and down spins in the magnetic metal. A more detailed description of this experiment is provided in the Project Highlight, Measurement of Current Polarization by Doppler-Shifted Spin Waves.

We have also extended a ferromagnetic resonance spectroscopy technique to enable measurement of the magnetic properties of edges in lithographically fabricated nanostructures. Edges become more important for smaller structures simply because more of the structure is close to an edge. Most recently, we have shown that this technique can be used to observe the effects of oxidation on film edge properties, in structures lightly oxidized in oxygen plasma and in structures annealed in oxygen. These measurements showed that both oxidation methods made the edges less ideal, but that the annealing also caused changes in the film away from the edges. In another set of experiments and models, we demonstrated that the effects of interactions between magnetic layers at the edges of patterned multilayers could be measured.

In a new program aimed at expanding measurements of nanomagnetics dynamics to include high spatial resolution, we have developed a ferromagnetic resonance force microscope system. Working on some of the same principles as electron paramagnetic resonance (EPR) force microscopes that have detected single electron spins, and nuclear magnetic resonance (NMR) force microscopes that have performed magnetic resonance imaging on viruses, this instrument is designed to allow spectroscopy of the magnetization dynamics in individual nanostructures with the goal of facilitating nondestructive evaluation of the magnetic properties of buried magnetic devices. Using this instrument we have recently localized spin wave modes using the dipole field of the cantilever tip. Emerging applications will include such future electronics measurements as performing failure analysis of defective devices in arrays.

Accomplishments:

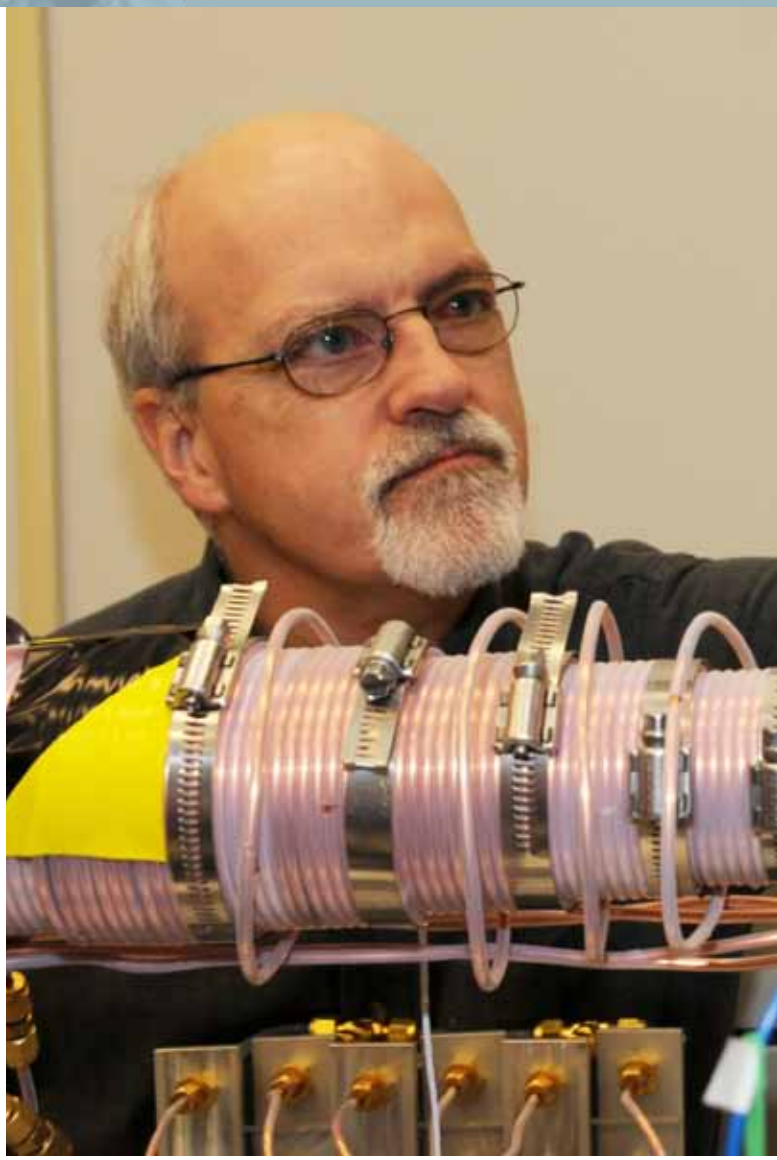
- Demonstrated the ability to measure the three-dimensional magnetization in multilayer magnetic storage media with graded anisotropy.
- Demonstrated the imaging of magnetic logic circuits (in collaboration with the University of Notre Dame and the University of Southern Florida).
- Developed methods to image the magnetic structure of lanthanum-strontium-manganese-oxide (LSMO) and other difficult to prepare materials.



- Produced electron vortex beams with up to 100 $\hbar$ per electron of quantized orbital angular momentum in the TEM.
- Showed that current polarization can be measured by observing the spin wave Doppler effect in magnetic metals (in collaboration with Hitachi Global Storage Technologies and North Carolina State University).
- Demonstrated that edge mode ferromagnetic resonance spectroscopy can be used to measure effects of oxidation and interlayer coupling.
- Designed and installed a ferromagnetic resonance force microscope.

Emerging Research:

- Develop measurements that probe the mechanisms of electrical control of magnetization in nanoscale devices.
- Create new ways to observe how domain-wall chirality behaves in its role as a logic state in nanomagnetic logic circuits.
- Develop a magnetization imaging electron microscope based on using electron vortex beams with orbital angular momentum.
- Demonstrate the detection of ferromagnetic resonance in nanostructures using sensitive cantilever techniques.

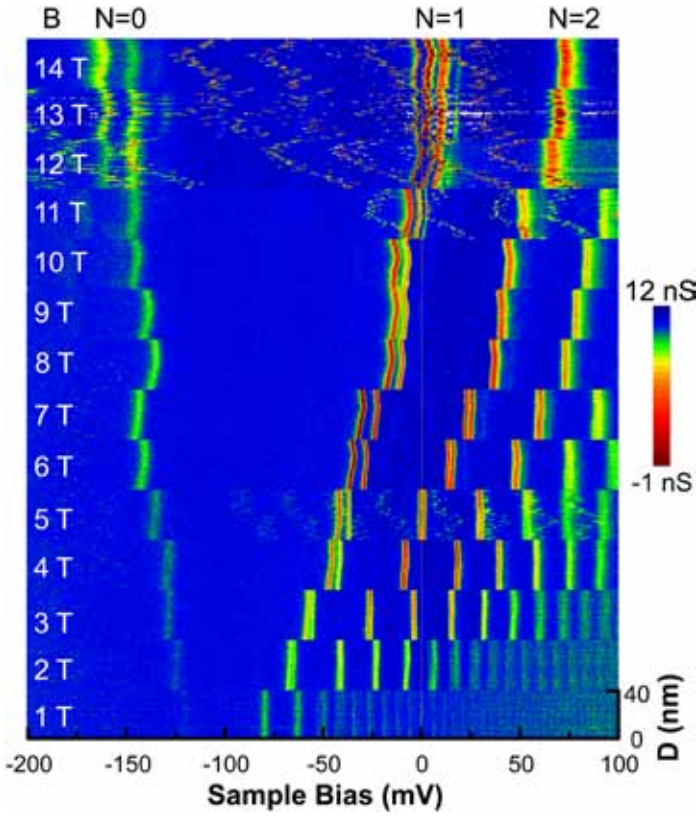


Atomic-Scale Characterization and Fabrication—This research is focused on developing new measurement and fabrication methods with atomic-scale precision. The experimental program emphasizes the design of custom instrumentation intended to push the frontiers of nanoscale characterization. Using state of the art scanning probe techniques, we have developed a new range of measurement capabilities that can provide essential input for a diverse set of new technologies in future electronics.

Recent work has focused on measurements on graphene, a single sheet of carbon with a promising potential for future electronic material applications because of its low scattering rates and high carrier mobilities. Magneto-electronic measurements are made using scanning tunneling microscopy (STM) and spectroscopy measurements in high magnetic fields. Two different types of graphene are used for these studies: epitaxial graphene samples grown on SiC obtained from the Georgia Institute of Technology in collaboration with the research groups of Professors Phillip First and Walter de Heer; and exfoliated graphene devices on SiO_2 substrates fabricated in the CNST NanoFab. With the epitaxial graphene samples, we made direct measurements of the Landau quantization resolving all four quantum states arising from electron and valley symmetries. These measurements were made using a new high resolution ultra-low temperature scanning tunneling microscope operating at 10 mK. The exfoliated graphene devices allowed us to investigate the effect of substrate interactions on the graphene electrical properties. New scanning spectroscopic techniques were developed to map out the electronic structure as a function of carrier density using STM "gate" mapping measurements. This work was performed in collaboration with Nikolai Zhitenev in the CNST Energy Research Group, as well as with David Newell and Angela Hight-Walker in NIST's Physical Measurement Laboratory.

Accomplishments:

- Developed measurements to determine the atomic structure of the edges of graphene islands grown on SiC.
- Measured magneto-tunneling conductance oscillations on graphene and used them to determine the dispersion of graphene's band structure.
- Created a method of analyzing STM moiré patterns to determine very small strains in multilayer graphene.

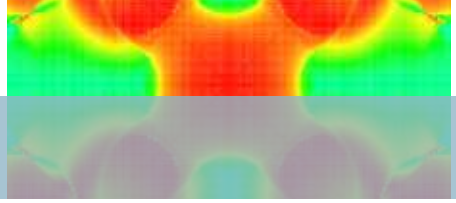


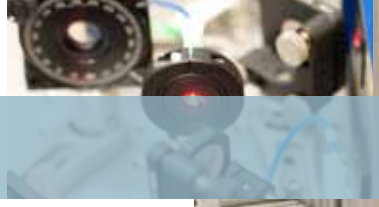
Scanning tunneling spectroscopy map of graphene, showing Landau level dependence on magnetic field up to 14 T.

- Demonstrated measurements of the magnetic quantum energy level structure (Landau levels) in graphene, revealing how the level structure scales with Landau index and magnetic field.
- Measured the spatial properties of the quantum Hall states in graphene, and showed how these properties can be related to geometric structural properties.
- Developed a best-in-the-world, ultra-low temperature, scanning probe microscopy system that operates at temperatures as low as 10 mK and in magnetic fields as high as 15 T for high energy and spatial resolution measurements of electronic structure in nanoelectronics materials and devices.
- Using the new milliKelvin STM, characterized the four-fold internal structure of a graphene Landau level, and determined the relative energy splitting relating to spin and valley degrees of freedom.

Emerging Research:

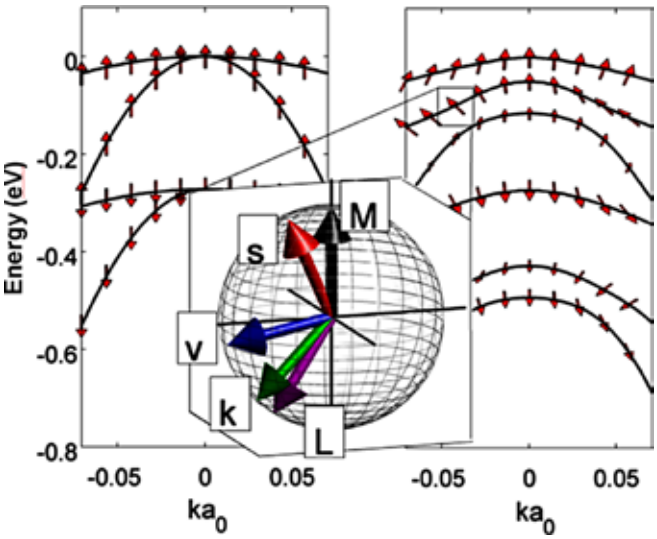
- Develop microscopic measurement methods for future electronic materials, such as graphene and topological insulators, in real device geometries to understand the role of substrates, insulators, and contacts in device performance.
- Create methods to grow and characterize topological insulators, a new class of materials with promising electronic properties.
- Advance spin-polarized scanning tunneling spectroscopy measurements for future electronic materials, such as graphene and topological insulators.





Modeling Nanostructures in Mesoscopic Environments—The EPG is using theory, modeling, and simulation to provide key insight in interpreting measurements of the structural, dynamic, electronic and magnetic properties of nanostructures and associated systems. The goal is to enable and stimulate the development of new measurement capabilities by elucidating the underlying physical processes in these systems. One aspect is to reveal the new physics that becomes important as electronic and magnetic devices approach nanometer length scales. Using various theoretical methods, this research interprets experiments, suggests new directions, and identifies possible improvements in measurements, devices, processes or systems.

One focus has been spin-transfer torques—the "forces" on magnetic systems that arise when electrical current passes through a non-uniform magnetization either in a multilayer or across a domain wall. The torques, which are strong enough to induce magnetic dynamics, are being intensely studied for device applications, with experiments underway at the CNST and around the world. Possible applications include Magnetic Random Access Memory (MRAM), in which memory elements consist of two ferromagnetic elements separated by a thin MgO tunnel barrier, as well as domain wall devices, in which the information is stored in the positions of domain walls that are moved past a sensing element. Motivated by new measurements, EPG theorists have computed the properties of spin-transfer torques in such systems with a range of approaches in order to understand how a variety of extrinsic effects modify the torques. Another important process underlying the performance is the magnetic damping; EPG theorists have addressed this by calculating the variation of magnetic damping that can occur in such nanoscale devices.



Calculations of the effect of spin-orbit coupling on the band structure of ferromagnetic GaMnAs.

An additional focus of our theory effort in the EPG is motivated by CNST's experimental studies of graphene. This research includes developing models to complement the new scanning probe measurement capabilities that have been demonstrated in graphene. A variety of approaches have been used to help interpret the data, including developing models for transport to understand the viability of graphene for use in electronic devices.

Accomplishments:

- Carried out detailed calculations of spin transfer torques in the presence of real world complications, including spin-orbit coupling, finite temperature, finite bias, and disorder.
- Calculated the transport properties of graphene bilayers.
- Calculated the damping parameters for transition metal ferromagnets, including their anisotropy and wave vector dependence, in addition to their variation in the presence of current.

Emerging Research:

- Determine the effect of interfacial spin-orbit coupling on domain wall motion.
- Understand charge transfer in few-layer graphene systems.

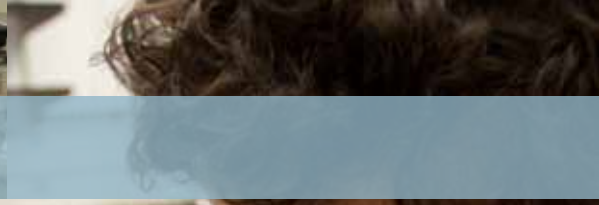
Nanoscale Measurement and Fabrication Using Laser-Controlled Atoms—The central theme of this research lies in taking advantage of the high degree of control afforded by laser manipulation of neutral atoms to develop new measurement and fabrication technologies for nanotechnology. Neutral atoms can be focused, deflected, trapped, state-selected, and cooled with an extraordinary level of control and precision using the mechanical effects of near-resonant laser light. The coldest temperatures on earth, the most precise clocks, and entire new forms of matter are achieved in this way. Such exquisite control opens many opportunities for new nanotechnology measurement applications. Putting these processes to use has enabled us to address such diverse needs as production of high resolution focused ion beams for nanofabrication and imaging, deterministic production of single atoms "on demand," and resist-free *in situ* fabrication of nanoscale features.

Current activities in this research area are concentrated on developing and applying our recently demonstrated magneto-optical trap ion source, or MOTIS (U.S. Patent no. 7,709,807). This source takes advantage of the 100 μK temperatures available in a magneto-optical trap to create an ion beam with very low emittance and high brightness. By ionizing neutral atoms in the trap and extracting them, a beam is created without the need for an extremely small source size such as is found in conventional liquid-metal ion sources. The result is an ion source with focal properties rivaling or surpassing existing sources, with the following added benefits: a wide variety of possible ionic species; a very narrow energy spread; and the potential ability to implant single ions "on demand" with nanometer precision using techniques developed in previous years in the EPG. This new ion source will enable a diverse new set of focused ion beam applications, including contamination-free milling, new beam chemistry mechanisms allowing deposition and removal of material for applications such as integrated circuit repair, damage-free ion microscopy with new contrast mechanisms, and controlled doping of semiconductors.

Our work on MOTIS development has been greatly enhanced by a cooperative research and development agreement with FEI Co., an Oregon-based major supplier of focused ion beam systems. Through this mutually-beneficial and active collaboration, NIST has gained access to equipment in the form of a focused ion beam system in place at the CNST, which we are free to modify and adapt for use with a MOTIS, as well as the expertise of the Beam Development Group at FEI. Researchers at FEI gain early access to our groundbreaking technology.

Having just demonstrated some of the unique capabilities of the MOTIS this year, we are now beginning a series of exploratory research thrusts on a broad range of possible applications—ranging from novel beam chemistries to single ion implantation for color center formation—that will include development of ion sources with other species, experiments with very low and very high energy ions, and pushing the resolution of the focused beam system to its limit.





Accomplishments:

- Constructed a lithium ion source based on the MOTIS principle.
- Carried out a series of calculations and measurements of the effects of Coulomb interactions on Li MOTIS performance.
- Successfully mounted the Li MOTIS on an FEI-provided focused ion beam system, achieving a major milestone in this cooperative research project.
- Demonstrated the first-ever Li ion images, with < 30 nm resolution using 2 kV Li ions.
- Constructed an ion focusing and deflection system for existing Cr MOTIS.
- Demonstrated first-ever focused Cr ion images.
- Conducted exploratory measurements on Cr color centers in diamond.

Emerging Research:

- Exploratory measurements on Cr color centers in diamond.
- Converting Cr and Li ion beam systems to allow operation up to 30 kV.
- Exploring a wide array of imaging and nanofabrication applications using Li and Cr focused ion beams.
- Constructing MOTIS realizations with other ionic species such as Cs and Er, with applications such as secondary ion mass spectrometry (SIMS) in mind.

Energy Research Group

The Energy Research Group (ERG), the newest group in the CNST, develops instruments designed to reveal the nanoscale physical and chemical processes and properties critical to advances in energy conversion, transport, and storage. Research in this group has only recently begun, with most of the technical staff members joining the Center within the past 18 months. The Group's research includes nanoscale characterization of light-matter interaction, charge and energy transfer processes, catalytic activity, and interfacial structure in energy-related materials and devices. The current focus is on the creation of instrumentation for nanoscale characterization of photovoltaic and thermoelectric materials and devices, nanoscale measurements of electrochemical processes for energy storage and conversion, and computational models of energy and charge transfer dynamics.

Nanoscale Optoelectrical Characterization of Photovoltaic Materials and Devices—Large-scale implementation of solar power generation will require photovoltaic (PV) devices with a ratio of efficiency to cost that is much higher than existing silicon-based systems. Such "next generation" devices will depend on new materials and innovative device structures. Thin-film PV devices, also called second generation devices, are becoming competitive with Si-based technologies, but face technical challenges to achieve their potential for high efficiency. Third generation devices are now under development that exploit nanoscale three-dimensional (3D) structures to achieve higher efficiencies at affordable manufacturing cost, but are still less efficient than current commercial solar panels. All these devices feature sophisticated nanometer and micrometer scale structures that have significant effects on overall device performance. The ERG is developing the measurement tools and techniques along with the requisite modeling approaches needed to characterize the physical processes related to light absorption, energy transfer, and carrier dynamics in both second and third generation PV materials and devices.

Energy Research Group
December 2010
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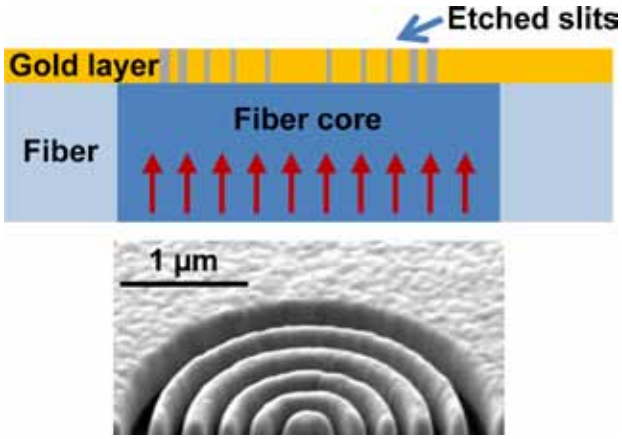
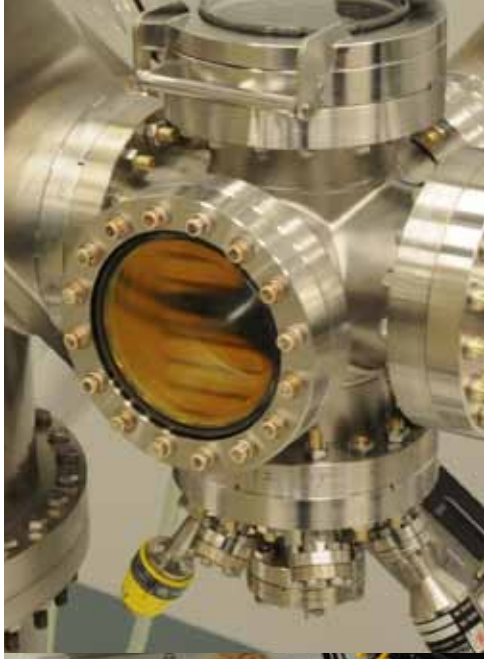
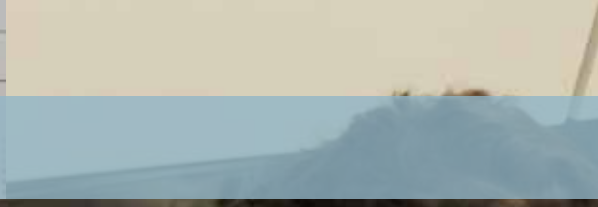
Third-generation cells based on 3D novel nanostructures such as dye-sensitized solar cells, organic bulk heterojunctions, nanocomposite materials, quantum dots, and nanocrystals embedded in polymer matrices typically show low performance caused by a wide range of poorly understood mechanisms, including nanomorphology, inefficient electron transfer at interfaces, charge carrier mobility, and impurities. The ERG is developing multiple probe-based optical and electrical characterizations combined with structural measurements, cross-sectioning of active devices, and nanodevice fabrication, with the goal of obtaining complete information on the relevant physical phenomena at the nanoscale in PV devices. Scanning probe techniques such as conductive atomic force microscopy (AFM) and STM will be correlated with local photoluminescence (PL), surface potential, photocurrent measurements, and local spectral response measurements on third-generation and thin-film PV materials and devices. The measurement development includes the design of advanced local probes such as integrated electrical amplifiers and plasmonic lenses on fibers. (See the Project Highlight, Nanoscale Characterization of Organic Photovoltaic Devices.)



Fabrication of plasmonic lens on a fiber. Top: A gold layer is evaporated onto a cleaved fiber, and circular slits concentric with the fiber core are cut through via FIB. Bottom: SEM image of a lens cross-section.





Another approach to gaining spatial information about the physical processes and characteristic length scales in PV materials is through fabrication and characterization of PV test materials and devices with spatially and morphologically defined 3D structure. The characteristic length scales for energy processes in modern PV materials range from a few tens of nanometers for exciton diffusion, to a few hundreds of nanometers or a few micrometers for grain sizes in thin film materials or carrier diffusion length, respectively. These length scales are well within the capabilities of modern lithographic techniques such as electron beam, imprint, or photolithography. The ERG is collaborating with researchers in the NIST Material Measurement Laboratory (MML) to fabricate and measure regular, 3D-structured PV materials, such as electrochemically-deposited compound semiconductors and chemical vapor deposited nanowires. Another ERG project, in collaboration with the University of Michigan, University of California, Berkeley, and Sandia National Laboratory, has demonstrated and characterized radial *p-n* junction photovoltaic devices with controlled 3D-structure fabricated with simulated metallurgical grade Si (less pure, lower cost). This project developed a quantitative, physics-based model explaining the experimental observations, and thereby led to specific recommendations for optimal device geometry (see the Project Highlight, Efficiency Enhancement of Copper Contaminated Radial *p-n* Junction Solar Cells).

Accomplishments:

- Developed measurements of local photoresponse in polymer solar cells with spatial resolution in the range from 50 nm via conductive AFM, to 200 nm to 1000 nm via nanofabricated contacts.
- Measured the 3D nanomorphology in polymer solar cell through application of AFM and sectioning techniques.
- Developed models to access the impact of inhomogeneous nanoscale morphology on efficiency of organic PV device.
- Created quantitative models for 3D-structured PV materials based on Si radial *p-n* junctions.
- Designed and fabricated plasmonic lenses integrated with optical fibers.

Nanomaterials in Electrochemical Energy Conversion and Storage—A number of new approaches to creating more efficient devices for energy harvesting, storage, and conversion are based on the exploitation of nanostructured materials in electrochemical systems. Incorporating nanostructured electrodes into electrochemical energy conversion and storage devices offers several advantages for a variety of transportation and other "green" energy infrastructure applications, including batteries, fuel cells, and so-called "supercapacitors." In each of these devices, nanostructured materials can be used to increase the surface area where the critical chemical reactions occur within the same volume and mass, thereby increasing the energy density, power density, electrical efficiency, and physical robustness of the system. Such materials also have the potential to lower manufacturing costs.

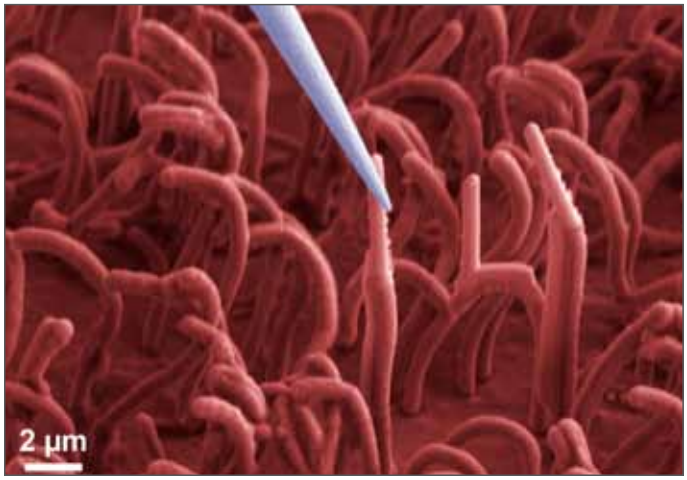
The fabrication and effective use of nanomaterials in electrochemical devices presents formidable challenges, however. In particular, scientists do not yet fully understand the interfacial reactions and phase transformations that accompany charge transfer at electrode/electrolyte interfaces. Controlling these phenomena requires careful measurements and a fundamental knowledge of how nanoscale surface composition, structure, and defects affect these processes.

To address these challenges, the ERG is building several experimental platforms designed for characterizing the physico-chemical processes that accompany charge transfer in electrochemical systems. One system is designed to rapidly transfer within an inert atmosphere a specimen from a liquid

electrochemical cell to a multi-analysis ultra high vacuum chamber, where surface structure and composition can then be determined using electron, scanning probe, and X-ray beam methods. This system will be used to characterize a range of electrochemical energy conversion and storage devices, including batteries, fuel cell electrocatalysts, electrochemical capacitors, and photoelectrochemical cells for hydrolysis. These measurements will provide the key knowledge needed to improve the efficiency and performance of such devices.

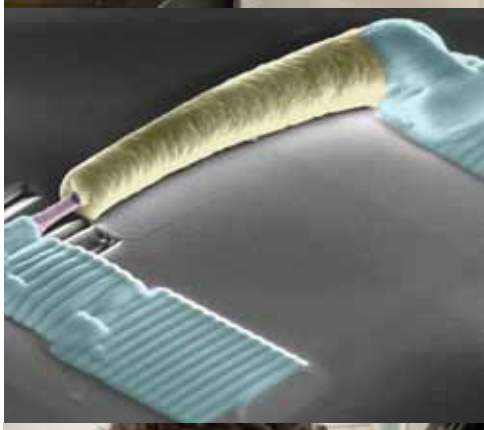
Another system, already operational, uses surface plasmon-polariton waves (SPPs) for real-time, dynamic characterization of processes at electrode/electrolyte interfaces. Because SPPs are confined to the metal/dielectric interface, they are highly sensitive to changes in the electrode surface charge density and the electrolyte dielectric. In addition to SPP spectroscopy, The ERG is also integrating a Raman spectrometer with an atomic force/scanning electrochemical microscope. This system will be dedicated to correlating local photoelectrochemical activity in water splitting catalysts with nanometer scale morphology and chemical composition.

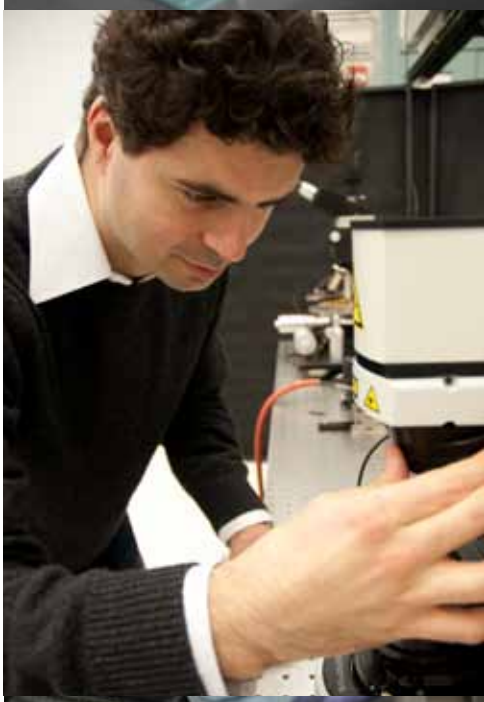
Finally, the ERG is fabricating complete, all-nanowire Li-ion batteries and using these to characterize the physical and chemical processes that accompany the flow of charge directly in the scanning electron microscope. The experimental observations will be used to develop physics-based models to describe the electrochemical behavior of these devices.

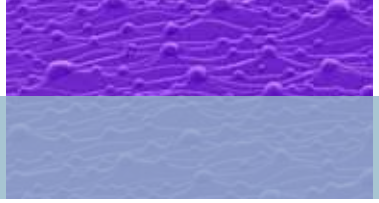


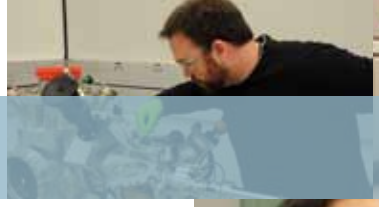
Measurement of nanowire-based battery using nanomanipulator in SEM (false-color image).

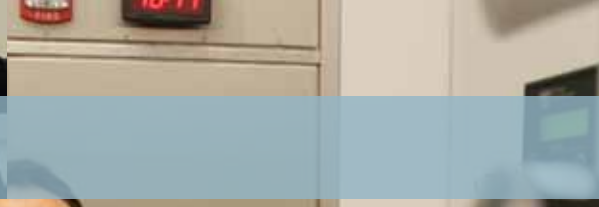
To obtain the detailed mechanisms of electrocatalytic reactions relevant to energy conversion, the ERG is developing spectro-electrochemistry of nanocatalysts by exploiting plasmonic properties and plasmonic antennas. Catalytic nanostructures with well defined geometry, dimensions, composition, and plasmonic resonance will be used as a platform for simultaneous electrochemical and spectroscopic interrogation. The role of plasmon excitations on the rates of charge transfer will be specifically addressed using both experimental and first-principle theoretical methods.











Emerging Research:

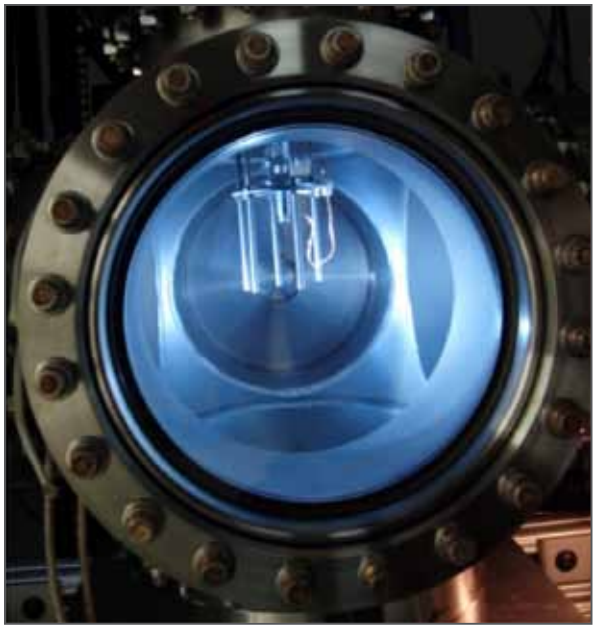
- Characterizing nanowire-based solid-state batteries.
- Measuring charge accumulation and reactions at electrochemical interfaces using nanoplasmonics.
- Using Raman spectroscopy for spatially-resolved strain mapping in nanostructured Li-ion battery electrodes.
- Correlating local activity, morphology, and composition in solar hydrogen electrodes.

Nanostructured Thermoelectrics—Finding alternative energy sources is one approach to satisfying the ever growing demand for energy; another is converting energy that is not used and generated in the form of waste heat. On the large scale, thermoelectric conversion of waste heat is the focus of serious and significant study in industries creating turbine machinery for the electricity generation. These turbines can individually generate power at levels in excess of 500 MW, and newly-constructed power plants often have power production approaching several gigawatts. These turbines are highly efficient, often operating at 60 % efficiency, and approach the Carnot value. A major limiting factor in efficiency occurs at the final stage compressor, where the gas temperature is in the range of 200 °C to 300 °C. At this temperature the low temperature/pressure of the gas does not allow for efficient extraction of mechanical energy and thus it is exhausted as waste heat. Here, thermoelectrics can allow for direct conversion of waste heat into electricity. In a centralized source as power plant, improvements of a few percent in energy production will be significant. In the absence of regulatory edicts, cost-effective implementation of thermoelectrics requires a three-fold improvement over existing thermoelectric material efficiency. There are also several smaller-scale applications of this technology, including in trucks and automobiles. Internal combustion engine platforms convert only about 25 % of the chemical energy from gasoline into kinetic energy; the other 75 % is lost in the form of mechanical friction and exhaust gases. For the latter, thermoelectric generators are being considered for use in converting exhaust heat into electricity, allowing for removal of several components, including on-board generators, and increasing overall efficiency in the range of 5 % to 10 %.

Recent advances in nanostructured thermoelectric materials have increased the conversion efficiency of the thermoelectric devices to the point that they may soon allow cost-effective operation. Several scientific developments have resulted in improvements in the thermoelectric figure of merit, ZT, which is a measure of conversion efficiency. These developments may be broadly categorized into two distinct mechanisms: altered electronic density of states, and altered charge transport. They involve fabrication of reduced-dimensional structures or lamellar-type structures

Characterizing a thermoelectric material requires precise measurements of electrical conductivity, thermal conductivity, and thermopower (the voltage produced per degree temperature difference across the material). To address this need, the ERG is developing new measurement tools and techniques that can be applied to both conventional and unconventional thermoelectric materials, allowing for a more accurate determination of the key properties, including ZT.

The ERG has designed an innovative heat pipe, incorporating high-precision thermometers that can locally probe temperatures ranging from room temperature to 800 K with an accuracy of a few milliKelvin. The design is versatile, allowing for measurements on samples of different structure and composition, ranging from nanowires grown in templates with diameters of approximately tens of nanometers, to macroscopic materials containing lamellar nanostructures. By providing a complete set of measurements, this approach will enable the comprehensive characterization of nanostructured materials needed to advance commercially-promising thermoelectric technologies.



Ultrahigh vacuum instrumentation for measuring the performance of nanostructured field emitters.

Emerging Research:

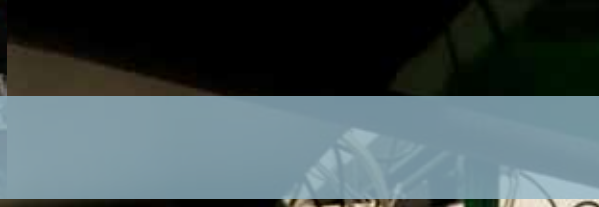
- Developing methods to characterize thermopower.
- Measuring thermal conductivity in nanostructured thermoelectric materials.
- Characterizing chalcogenide lamellar structures.
- Measuring the thermal properties of narrow-band semiconductor nanowires.

Characterizing Field Emitters Based on 1D Structures—Cold cathode emitters could potentially have one of the largest impacts of any nanotechnology, as there are realistic applications across several significant industries. Possible applications range from energy-saving displays, to medical and security imaging, to microwave electronics, to compact free-electron lasers. The goal of this project is to develop methods for measuring the local current density and emittance (transverse kinetic energy or beam spread) of nanostructured emitters.

Accurate measurements on two parameters (local current density and emittance) are required to understand and validate physics models of cold cathode emitters. At present, experimental characterization is limited to measurements of current over large areas, yielding average current density. As current densities of cold cathode emitters are highly non-uniform over the emission area, present techniques do not provide sufficient information so as to enable better understanding of the underlying emission physics of these systems.

Emerging Research:

- Measuring local current density and emittance.
- Fabricating model emitter arrays comprised of carbon nanotubes and InP nanowires.



Nanofabrication Research Group

The Nanofabrication Research Group (NRG) conducts research to support the CNST's nanomanufacturing research thrust through the creation of new measurement methods to enable the development and effective use of nanomanufacturing and nanofabrication processes. These methods include both lithographic ("top-down") and directed assembly ("bottom-up") approaches. Much of the NRG's research is directed towards both measurements capable of elucidating the fundamentals of nanofabrication processes as well those suitable for use in a high-throughput nanomanufacturing environment. The staff members' expertise spans a very wide range, but the following main themes underlie much of the work: nanoscale optical measurements; dynamic *in situ* measurements; nanomechanics; and the stochastic behavior of nanoscale systems. Project leaders in the NRG also make significant contributions to research across the CNST, supporting the energy and future electronics focus areas. Output from the NRG is rapidly growing as the Project Leaders' laboratories come on line.

Nano-Optics—Light in or near the visible range offers tremendous opportunities for sensitive measurements of the properties of nanostructures. Typically, however, the diffraction-limited optical spot size of about a wavelength makes measurements of single nanoscale objects problematic. Nanophotonics and nanoplasmonics both provide the means by which light can be confined to the nanoscale, enabling such measurements.

Sub-wavelength features etched into high-index semiconductor and dielectric materials such as silicon, silicon nitride, and gallium arsenide can be used to produce waveguides and resonant cavities that allow for the precise manipulation of the flow of light at very small dimensions. With advanced fabrication techniques, the losses can be minimized, enabling the production of devices with extremely high quality factors. The NRG's research is focused on the fabrication and characterization of such structures and their use in measuring phenomena related to cavity quantum electrodynamics involving individual quantum dots, single-photon emitters, and signal transduction.

Current activities in this area make use of a novel cryogenic fiber-taper probe system. This system uses the evanescent field extending from an optical fiber (adiabatically tapered to a diameter on the order of a micrometer) to interact with microcavity devices and nanostructures. Light injection and extraction is very efficient with this device, enabling precise spectroscopic measurements to be made on semiconductor waveguides and resonators containing single quantum dots. Measurements exploring novel strong light-matter interaction effects in these systems have been performed in collaboration with the University of Rochester. Individual solution-synthesized quantum dots can also act as single photon sources and have the potential to be integrated with conventional silicon semiconductor devices, bringing quantum information transmission a step closer to practice. In collaboration with Columbia University, a low density of quantum dots was deposited on a Si photonic crystal and the fiber-taper waveguide was used to probe their spectroscopic behavior close to the 1.55 μm telecommunication wavelength. (See the Project Highlight, Measuring Light-Matter Interactions in Chip-Based Optical Cavities.)

Nanofabrication Research Group
December 2010
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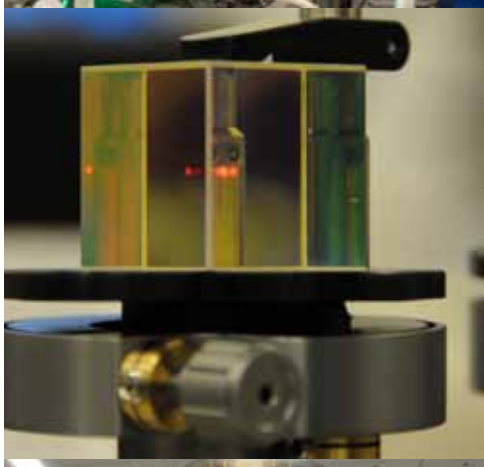
In collaboration with NIST's Information Technology Laboratory, we have developed a method for taking 1.3 μm wavelength photons, which have good long-distance transmission properties, and efficiently up-converting them to easy-to-detect photons at 710 nm. Significantly, the quantum, single-photon properties of the source are preserved in this process. This work clearly demonstrates that it is possible to integrate the disparate quantum systems needed for information transmission and computation.

Metal nanostructures can also be used to guide light at the nanoscale by means of surface plasmon-polaritons (SPPs)—waves confined to the interface between metals and dielectrics. Optical confinement takes place in all three dimensions, leading to extremely large field enhancements that can be used to create strong interactions with non-linear materials. The enhanced effect of small external perturbations

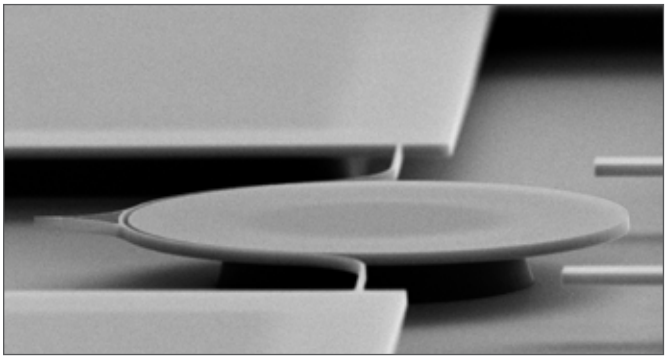



on the behavior of the light, such as the absorption of a single molecular layer, can be used both in sensing and switching applications. NRG research is focused on the design and fabrication of novel components for measurement and communications based on nanoscale plasmonic effects, thereby essentially recreating the toolkit of macroscale optical devices in the deep sub-wavelength regime. SPPs also offer the ability to create novel devices unlike anything available at the macroscale. The unique dispersion characteristics of SPPs traveling in nanostructured metallo-dielectric materials enable metamaterials with properties such as negative refractive indexes to be created. These materials are the subject of intense study and may have significant potential for imaging and manipulating matter at the nanoscale.

The utility of plasmonic devices for sensing and measurement applications relies on a clear understanding of how light behaves in the near and far-fields, both theoretically and experimentally. Recent work has focused on advancing this understanding for simple geometries such as slits and holes that are often used in sensor applications. We have used a combination of modeling and far-field measurement to demonstrate how the illumination of a single slit can efficiently generate surface waves. The phase shift (close to $-\pi$) of the surface waves compared to the reference wave creates interference that, in the case of periodic slit arrays, controls the transmission profile. The surface waves can readily be perturbed by, for example, small changes in the surface chemistry—the reason they may be used in sensing. However, we can also use the change in chemistry induced by those waves to learn about the field distributions of the plasmonic devices themselves. We are working with collaborators at the University of Texas at Austin to develop a sublimation-switch resist material that is sensitive at long wavelengths (≈700 nm) to map surface fields. Initial results qualitatively agree with modeling results, but show many interesting features that may relate to small imperfections in the actual experimental devices. The technique is simple and can be used over large areas, and we are currently working to make it fully quantitative.

Accomplishments:

- Developed novel cryogenic optical fiber-taper probes for characterizing nanophotonic devices.
- Demonstrated single-photon up-conversion that enables efficient measurements of nanoscale, near-infrared quantum emitters.
- Performed detailed spectroscopy of single PbS quantum dots in collaboration.
- Developed a novel, broadband-sensitive resist that can be used to quantitatively determine time-averaged field intensities in nanoplasmonic devices.
- Created techniques to enable the measurement of the optical and optomechanical response of 3D plasmonic metamaterials with a negative index of refraction.



SEM image of an on-chip cavity optomechanical transducer for atomic force microscopy applications. A high quality factor silicon microdisk optical resonator is used to measure the megahertz frequency vibrations of an adjacent nano-cantilever, providing high displacement sensitivity, bandwidth, and dynamic range.

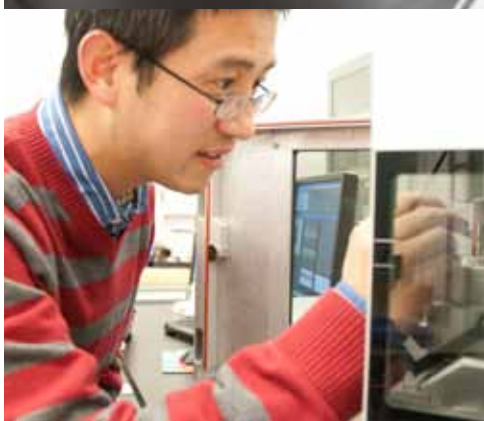
Nanomechanics—Nanomechanical systems can be used for both sensing and actuation. Their small size can render them extremely sensitive to the smallest perturbations, or capable of the most delicate manipulation. They are thus both objects of study and used for the investigation of other nanoscale devices.

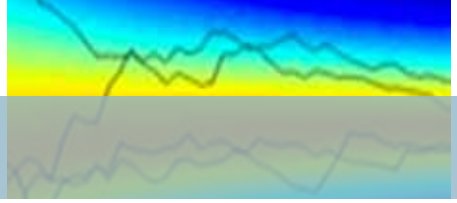
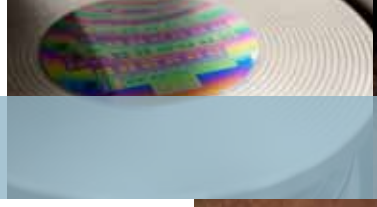
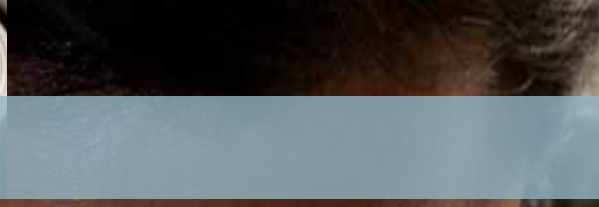
Friction accounts for much of the energy wasted in industry, and it has a strong impact on the reliability of devices. This impact is true down to the nanoscale, where surface interactions dominate bulk forces, and the effects of friction, adhesion, and wear hinder the development of complex nanomechanical devices. The NRG's research is directed to developing new ways to measure the fundamental mechanisms governing friction. One area of interest is the loss of waste heat to phonons generated at sliding interfaces. A greater understanding of friction can help improve the energy dissipation and wear characteristics of mechanical systems (including those at the macroscale), as well as help guide the engineering of electromechanical devices that require a balance between frictional properties and thermal management.

AFM friction image (2 μm × 2 μm) showing a dependence on the different structural properties of a patterned substrate.

The AFM is the workhorse instrument for nanoscale friction and adhesion measurements; however, even the most advanced AFM lacks the ability to perform detailed characterization of thermal properties and local thermal profiles in combination with force measurements. By integrating Raman spectroscopy with an AFM customized for measuring thermal properties, the NRG is building a powerful new measurement tool to investigate nanoscale energy dissipation with the potential to affect a variety of technologies.

Nanomechanical devices can be used to effectively emulate the sense of touch at the nanoscale. Although much has been achieved with scanning probe microscopy in its numerous incarnations, there is still a great deal to be done to increase the speed and sensitivity with which such measurements can be made, and to make those measurements with compact devices that can be easily integrated into more complex systems. Small-scale mechanical systems can influence and be influenced by the intense local fields produced by microphotonic and nanoplasmonic structures, allowing for the development of entirely



novel devices. In a new program that is already yielding exciting results, the NRG is creating integrated optical microelectromechanical systems (MEMS) with nanoscale elements (NEMS) that will enable imaging, metrology, manipulation, and assembly techniques.

Although AFM has proven its utility in nanoscience many times over, conventional AFMs are still relatively slow and, because of the free-space optics typically used to detect the cantilever motion, they are limited in the combination of spring constants and cantilever dimensions that can be used. By integrating the cantilever with a whispering gallery resonator the NRG has been able to achieve displacement sensitivities of 10^{-15} m/√Hz in extremely compact devices that have GHz bandwidths. As an added benefit, the light is confined in these devices and so does not affect the sample under study.

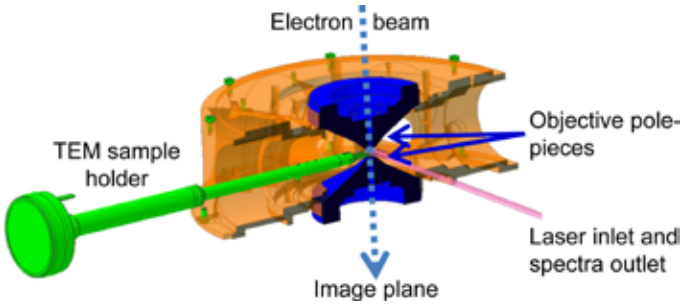
Although increasing the speed of a single probe can yield dramatic improvements, using parallel arrays of probes can result in qualitatively different measurements. In a new project, the NRG is exploring the potential for arrays of near-field optical reference structures to enable high-throughput measurements of structures such as photomasks. Careful choice of the reference structure geometry with respect to the relatively constrained set of patterns on the mask allows the far-field optical signal to provide detailed nanoscale information, once the appropriate deconvolution has been performed.

Accomplishments:

- Developed an integrated micro-optomechanical system capable of high-speed, high-sensitivity displacement measurements for scanned probe microscopies.
- Revealed the strong influence of applied load and adhesion on the frictional properties of graphite, and demonstrated a switch in friction contrast between single and multilayer graphene for suspended versus supported structures.

Emerging Research:

- Measuring nanoscale friction and adhesion.
- Developing nanoscale thermal properties characterization by combining AFM with local *in situ* thermometry.
- Creating holographic optical tweezers for manipulating MEMS and NEMS.
- Designing and fabricating optical reference nanostructures for high-throughput, near-field optical imaging.



A cross-sectional illustration of the sample area in the ETEM with integrated Raman spectroscopy.

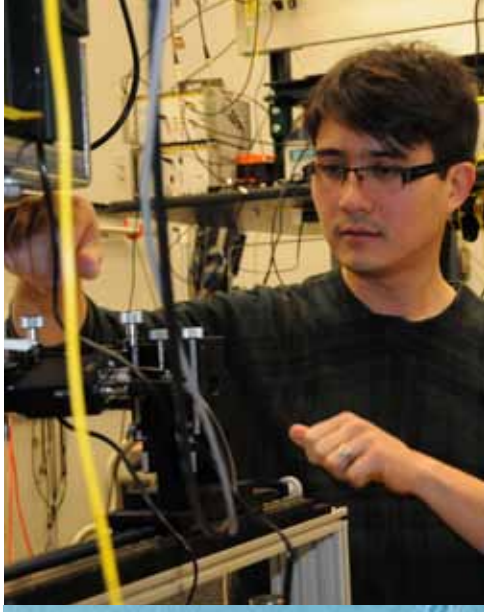
***In Situ* Measurement**—Many of the most important and interesting nanoscale phenomena are highly dynamic in nature, and capturing the details of these dynamic processes is essential to successfully exploit them. Aside from the intrinsic difficulty imposed by the small length scale, there are many measurement challenges that arise from the environment in which these phenomena occur.

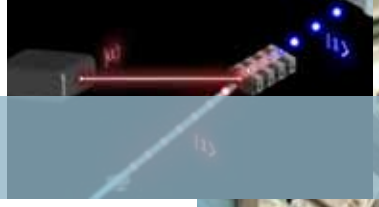
Transmission electron microscopy and its associated analytical techniques have proven their value many times over in materials science. The NRG is in the process of adding an environmental (scanning) TEM [E(S)TEM] to the CNST's suite of major equipment. This instrument will allow for the study of atomic-scale processes occurring, for example, during the growth of carbon nanotubes (CNTs) from individual catalyst nanoparticles at realistic process temperatures and in relevant gaseous environments. The microscope's capabilities are being enhanced by the integration of a custom-built *in situ* Raman spectroscopy system that will enable, for example, the identification of the types of carbon nanotubes being formed during growth, or precise local temperature measurements. (See Project Highlight, *In Situ* Measurements of Thermodynamics and Reaction Kinetics During Nanomaterials Synthesis and Catalysis.) The NRG's research, leveraging the capabilities of the NanoFab, is directed towards the development of new types of sample cells that will enable *in situ* TEM and also SEM measurements of bistable electronic switches and electrochemical processes relevant to batteries, super capacitors, and fuel cells.

Molecular processes that take place in a liquid environment are relevant in biology, but are also important in many self-assembly processes. Ensemble measurement techniques can be used to provide information integrated over large numbers of individual systems but can miss the diversity of behavior that can occur in single events. The NRG is focusing on developing methods that can track and interrogate the behavior of molecules and nanoparticles as they interact while freely diffusing in solution or in proximity to a functionalized surface. We have recently developed the models to understand how to control both the position and orientation of nanowires in solution using electro-osmotic flow and are now experimentally controlling metal, dielectric, and semiconducting materials. These methods can be used to control and study the interactions of nanoparticles with each other and with functionalized surfaces. They may even lead to the development of "free-floating" scanned probe techniques that can enable minimally perturbative measurements of nanoscale local environments. (See the Project Highlight, Particle-Tracking Measurements of Nanoparticle Dynamics in Fluids.)

Accomplishments:

- Developed the theory of simultaneous object orientation and position control.
- Demonstrated tracking of individual nanoparticles in three dimensions using a microfabricated optical system.
- Created improved statistical methods to analyze the behavior of diffusing nanoparticles to enable accurate extraction of diffusion coefficients.




Emerging Research:

- *In situ* scanning and transmission electron microscopy.
- Designing and microfabricating sample environments and measurement platforms.
- Fluorescence correlation spectroscopy.
- Advanced methods to track and interrogate single molecules and nanoparticles in real time.

Nanofabrication and Nanomanufacturing—The ability to control the detailed configuration of matter at the nanoscale is at the heart of nanoscience and nanotechnology. Novel, specialized measurement methods and fabrication techniques provide new ways of accessing, investigating, and directing nanoscale phenomena. Methods that can be scaled for high throughput enable new technological applications of these phenomena.

Optical lithography is still the mainstay of the current major nanomanufacturing industry—integrated circuit (IC) fabrication—and is likely to be so for the foreseeable future. However, in order to maintain the current rate of progress, continuous improvements in the lithographic process must be made. These improvements depend on developing an ever more detailed understanding of nanoscale stochastic and molecular effects. The NRG is focused on creating the measurement methods that are needed to extend the lifetime and applicability of these existing techniques. (See the Project Highlight, The Effect of Resist on the Transfer of Line-Edge Roughness Spatial Metrics from Mask to Wafer.)

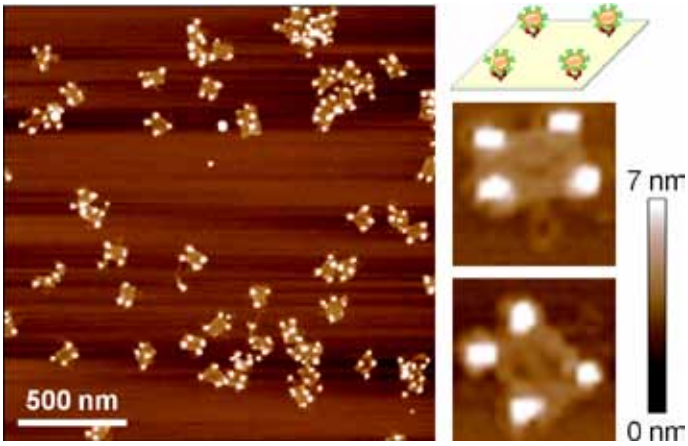
In a new research effort, the NRG is applying the technique of photoactivated localization microscopy (PALM) to map the distribution of acid catalyst species in chemically amplified resist materials. The acid catalysts enable each photon incident during the exposure process to cause a significant chemical change. This change increases the speed of the photoresist, but the acid diffusion that occurs can compromise the resolution. Until now, there has been no way of monitoring this process, with the acid behavior only inferred from the latent or developed image. The NRG has been able to perform measurements of the acid-diffusion blur using very dilute concentrations of fluorescent molecules (which therefore do not affect the resist). The technique works by recognizing that knowledge of the initial lithographic pattern functions as a maximum likelihood estimator for the acid-diffusion blur. In this way, a very sparse sampling of the diffusion profile can still yield nanometer-scale accuracy and precision. This technique may also be applicable to systems such as lithium-ion batteries, where detailed knowledge of ion distributions is important.

Novel fabrication techniques that do not have the same overlay and defectivity requirements as those used in integrated circuit production will address new applications; in particular, those that require low-cost, large-area, high-throughput patterning such as data storage, photovoltaics, and batteries. These nanomanufacturing methods present unique metrology challenges in terms of monitoring the behavior of nanoscale structures at extremely high speeds. The NRG's research in this area is aimed at creating the measurement and fabrication methods needed for approaches such as roll-to-roll directed self-assembly, which combines existing patterning methods with self-organizing systems like diblock copolymers.

The NRG has developed a resonant-scattering X-ray diffraction technique that enables the measurement of the domain profile and interfacial roughness of diblock copolymers assembled on lithographically patterned substrates. The use of resonant scattering enables the generation of high contrast between the two phases of the diblock by tuning the X-ray energy to specific chemical bonds. The resulting high diffraction efficiency yields data sets with enough resolution to perform

detailed parameter extraction with high confidence. (See the Project Highlight, Line-Edge Roughness Measurements in Diblock Copolymers.)

The precise assembly of nanoscale components represents one of the major challenges in nanomanufacturing. Molecular assembly methods based on DNA are gaining in popularity because of their ability to produce well-defined and highly-functional architectures. In another new project, the NRG is developing ways to measure the yield and speed of nanoparticle assembly on DNA origami. The measurements provide insight into the design rule constraints that apply to these kinds of processes and will serve as a guide to those attempting to create manufacturable devices based on this type of directed self-assembly.



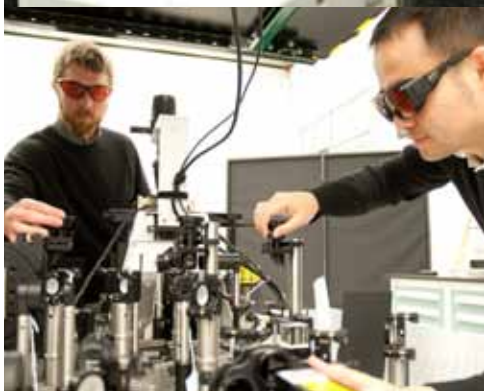
AFM images of quantum dot nanopatterns on DNA origami templates. The cartoon illustrates the locations of the binding sites for this origami design, with successful self-assembly demonstrated by the two higher magnification images of individual templates, each with quantum dots decorating all four corners.

Accomplishments:

- Applied novel estimators for characterizing a particle control system to enable the deposition of single quantum dots to an accuracy of less than 50 nm.
- Developed a resonant X-ray scattering technique to measure the detailed domain shape and interfacial roughness in diblock copolymers assembled on lithographic patterns.

Emerging Research:

- Metrology for high-throughput nanomanufacturing.
- Stochastics of nanoscale systems.
- Directed assembly of diblock copolymers.
- Molecularly-precise, templated assembly.
- Super-resolution optical imaging for lithographic process analysis.
- Nanoparticle tracking for inter-particle force measurements.
- Active control of nanoparticle assembly.
- Focused-ion beam nanofabrication, analysis, and metrology.



Project Highlights

10In order to provide a more complete overview of the breadth of activities supported by the CNST as a national User Facility, the following pages provide more in-depth highlights of a selection of projects. These projects represent a cross-section of measurement research conducted by the CNST staff, along with projects in the NanoFab led by academic, industrial, and NIST researchers.

Measuring Quantum Electronic Properties of Graphene

Research Participants: Y. J. Song,[1,2] A. F. Otte,[1,2] Y. Kuk,[1] Y. Hu,[3] D. B. Torrance,[3] P. N. First,[3] W. A. de Heer,[3] H. Min,[1,2] S. Adam,[1] M. D. Stiles,[1] A. H. MacDonald,[4] and J. A. Stroscio[1]

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[3]School of Physics, Georgia Institute of Technology, Atlanta, GA 30332

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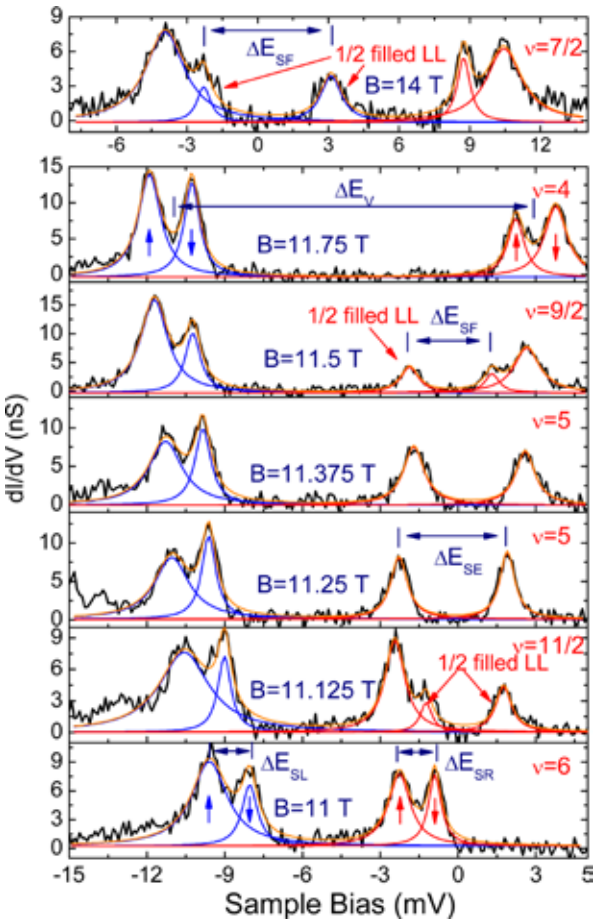
Key Accomplishments:

- Resolved the "quartet" electronic structure of graphene magnetic quantum states.
- Discovered new fractional many body quantum states in graphene.

Background: Graphene, a single atomic layer sheet of carbon, has unique electronic properties with potential applications in future electronics, sensors, and composite materials. Electrons in graphene behave quite differently from those in traditional 2D electron systems. Like massless relativistic particles, they have linear dispersion and chiral eigenstates. In addition, there are two sets of electrons that have this dispersion centered at different valleys in reciprocal space. The symmetry between valleys, together with the two possible spin states, leads to a four-fold degeneracy of the Landau levels. Understanding how electron-electron interactions lift the degeneracies in graphene's electronic states has been an intense goal in graphene research, with quantitative measurement of these interactions important for future applications. The main experimental methods for investigating these issues have been electrical transport measurements. Because graphene is an exposed 2D electron system, low-temperature scanning probe measurements offer an opportunity to probe the electronic structure of graphene with atomic-scale spatial resolution, thereby measuring the local variations in properties that are typically averaged over by traditional transport measurements.

Approach: We apply scanning tunneling spectroscopy at milli-Kelvin temperatures to resolve the graphene "quartet" electronic structure of magnetically quantized states and directly measure the energy separations when the degeneracies are lifted in an applied magnetic field. These are the first measurements from the recently constructed ultra-low temperature scanning probe microscopy system at the CNST, and are the lowest temperature scanning probe measurements ever performed.



High resolution Landau level spectroscopy of the four-fold states that make up the N=1 Landau level (LL) at different applied magnetic fields. At fields of 11.125 T, 11.375 T, and 14 T, new half-filled quantum states are observed at filling factors 11/2, 9/2, and 7/2 respectively. The electron spin splitting is enhanced at the odd filling factor of 5 at 11.25 T, while the valley splitting is enhanced at filling factor 4 at 11.75 T.

Results and Discussion: We measured the tunneling spectra of epitaxially grown graphene on the carbon face of SiC as a function of magnetic field at 13 mK. The spectra showed the evolution of the Landau levels with magnetic field. The field dependence of the orbital quantum number determined the graphene carrier velocity and demonstrated single layer graphene electronic behavior. This results from the multilayer graphene film being electronically decoupled due to the small rotational misalignments between the layers.

With the higher energy resolution obtained at these temperatures, the spectra showed interesting features relating to energy splitting within a single Landau level. The single N1 Landau level was observed to be

split into four sub-levels due to the lifting of the electron spin and valley degeneracies. When the Fermi level lies inside the four-fold Landau manifold, significant electron correlation effects result in an enhanced valley splitting at even filling factors, and an enhanced electron spin splitting at odd filling factors. Between integer filling factors, we discovered a series of new fractionally filled levels at filling factors of 7/2, 9/2, and 11/2. These measurements, which resolve spectroscopic features which are enhanced and fractional, suggest that new many body quantum states occur under these extreme conditions of ultra-low temperatures and high magnetic fields.

Recent Publications:

High-resolution tunnelling 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).

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).

A 10 mK scanning probe microscopy facility, Y. J. Song, A. F. Otte, V. Shvarts, Z. Zhao, Y. Kuk, S. R. Blankenship, A. Band, F. M. Hess, and J. A. Stroscio, *Review of Scientific Instruments* **81**, 121101 (2011).

Localization in Graphene Devices Probed by STM

Research Participants: S. Jung,[1,2] G. M. Rutter,[1] N. N. Klimov,[1-3] D. B. Newell,[3] I. Calizo,[3] A. R. Hight-Walker,[3] N. B. Zhitenev,[1] and J. A. Stroscio[1]

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[3]*Physical Measurement Laboratory, National Institute of Standards and Technology, Gaithersburg, MD 20899*

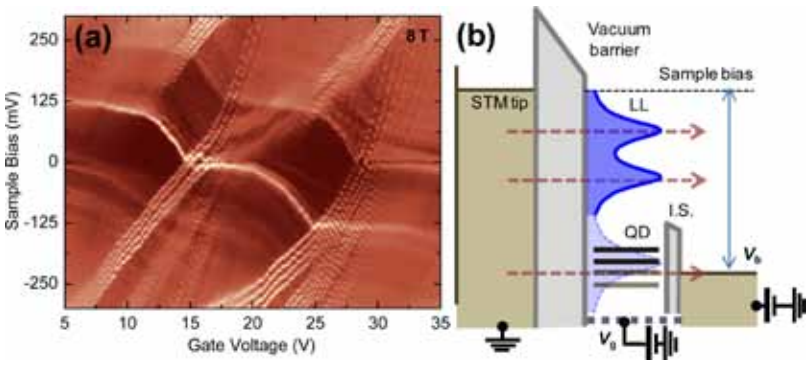
Project Goal: To develop a microscopic understanding of the scattering processes and localization phenomena determining the metrology-relevant half-integer quantum Hall effect of Dirac fermions and defining the transport properties of graphene devices for future electronic applications.

Key Accomplishments:

- Developed a fabrication process for graphene devices that maintains the high-degree of surface cleanliness required for STM experiments.
- Measured the evolution of scattering and localization using scanning tunneling spectroscopy in an applied magnetic field.

Background: Graphene is a unique two-dimensional material whose emerging physical properties hold great promise for electronic device applications. In realistic devices, physical phenomena such as the half-integer quantum Hall effect and high carrier mobility depend on interactions with impurities and substrates and on the localization of Dirac fermions. For example, plateaus in the quantum Hall effect are caused by the carrier localization, but very disordered samples do not display the effect at all. While achievable carrier mobility in graphene is extraordinary high, the mobility in research devices is much lower because of difficulties of integration and unintended disorder. Fortunately, direct access to a tunable exposed two-dimensional electronic system of Dirac fermions in graphene by scanning probes allows for the measurement of these carrier interactions and of the localization in greater detail than in conventional semiconductor devices, where the carrier transport layers are buried below the surface.

Approach: To avoid possible contamination caused by conventional wet-chemistry processing, graphene devices were fabricated from pristine flakes exfoliated on a SiO_2 substrate using stencil masks. Scanning tunneling spectroscopy (STS) was used to measure the local tunnel current-voltage (I-V) curves at a fixed distance between the tip and the sample. In these measurements,



(a) Tunneling spectra at different gate voltages combined in a "gate map" at 8 T. The dI/dV curves taken at different gate voltages are color-coded (white corresponds to peaks in the conductance) and plotted vertically with the horizontal shift proportional to the gate voltage. Two sets of features are seen in the map: the "horizontal" peaks are the Landau levels, and groups of "vertical" peaks result from single-electron charging of the QDs. (b) The tunneling current is defined by the density of states between the Fermi energy (E_F) of the tip and the sample. Tunneling channels at E_F of the tip are seen as horizontal lines in the gate map, while the channels at E_F of the sample appear as vertical lines.

the differential conductance, dI/dV, is proportional to the local density of states. Controlling the charge density of the Dirac fermions using an electrostatic back gate, we investigated the local density of states and localization in graphene at the atomic scale while varying the Fermi energy (E_F) with respect to the charge neutral Dirac point (E_D). Varying the magnetic field from zero to the quantum Hall regime strongly affected the electronic behavior of the graphene, and the states condensed into well-defined Landau levels with dramatic changes in the localization.

Results and Discussion: In an ideal graphene layer, the carrier density can be continuously tuned from hole to electron doping, through zero density at E_D. Local disorder gives rise to a spatially varying electrostatic potential that changes the relative position of E_D with respect to E_F. If E_F is close to E_D, then spatially alternating patterns of electron and hole puddles are formed. The distribution of the density set by the back gate voltage and spatially modulated by the disorder potential can be determined by locating E_D, a local minimum of the differential conductance dI/dV.

At zero magnetic field, we mapped the density fluctuations arising from the disorder potential variations. We found resonances in the density of states associated with the scattering from the disorder potential, as supported by the correlation between the spatial properties of the disorder potential and the spatial variation of the resonances.

At higher magnetic fields, discrete Landau levels (LLs) were resolved with both electron and hole states following single-layer graphene scaling. The LL spectra were dramatically different from previous STS measurements of epitaxial graphene on SiC and of graphene flakes on graphite, which showed characteristics of weakly disordered systems. Besides broader LLs due to disorder, we observed an additional set of localization resonances in the tunneling spectra governed by single electron charging effects. Effectively, the localization in graphene can create local quantum dots (QDs) with current flowing through two tunnel barriers in series: the vacuum tunnel barrier between the probe tip and a local graphene QD, and a barrier originating from the resistive incompressible strips that isolate the QD in a high magnetic field. Therefore, our STS measurements not only detect local density of states variations in graphene with and without magnetic fields, but also measure the electronic structure with sufficient sensitivity to probe single-electron charging phenomena at the Fermi level.

Recent Publications:

Evolution of microscopic localization in graphene in a magnetic field: from scattering resonances to quantum dots, S. Jung, G. M. Rutter, N. N. Klimov, D. B. Newell, I. Calizo, A. R. Hight-Walker, N. B. Zhitenev, and J. A. Stroscio, *Nature Physics* **7**, 245-251 (2011).

Development of Si-Based Single-Electron Devices (SEDs) for a Fundamental Current Standard and for Quantum Information

Research Participants: M. J. Stewart,[1,2] J. Bowser,[3] and N. M. Zimmerman[1]

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[3]*Center for Nanoscale Science and Technology, National Institute of Standards and Technology, Gaithersburg, MD 20899*

Project Goal: To develop Si-based single-electron devices, based on the Coulomb blockade, for applications in a fundamental electrical current standard based on the charge of the electron, and in charge or spin qubits for quantum computing.

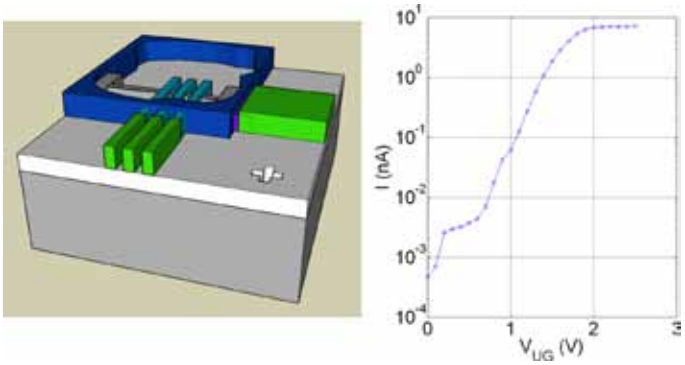
Key Accomplishments:

- Designed and implemented a process flow for SOI (silicon-on-insulator) MOSFETs, integrating ion implantation, polySi deposition, doping and patterning, and contact to metal leads.
- Demonstrated up to factor of 10^7 in on-off current ratio.

Background: Single-electron devices (SEDs) offer the amazing ability to move electrons around one-by-one. We have two long-term goals for our project. The first goal is the development of a quantum current standard. If we can develop a current standard based on the value of the electron charge, *e*, we could test Ohm's law with all three parameters being based on fundamental constants of nature; however, we need a standard with a current value of at least 100 pA, which is much larger than presently available. Si-based SEDs (essentially nanoscale MOSFETs with multiple gate layers) offer two potential advantages in this regard: higher operation frequency, and the ability to parallelize multiple devices. The second goal is to develop SEDs for quantum coherence applications. Many different experimental approaches are being pursued to develop a usable quantum computer. One main approach is semiconductor-based quantum dots, with the quantum mechanical degree of freedom being either charge position or electron/nuclear spin direction. In this approach, one important real-world source of undesirable decoherence is defect-mediated charge noise in the surrounding material. Our planed approach to addressing this problem is to fabricate high-quality devices with low defect density and then perform both decoherence (T^1 and T^2 times) and standard Si microelectronics industry measurements; e.g., C-V, G-V, DLTS, ESR, SDR, *etc.* In this way we hope to elucidate the sources of decoherence and their coupling mechanisms in order to ameliorate or eliminate this problem.

Approach: A schematic of one of our devices fabricated in the CNST NanoFab is shown in the figure. The device consists of the following three basic parts: a mesa-etched single crystal Si wire on a SOI wafer; two separate layers of doped polySi gates, with gate oxide and isolation oxides; and ion-implanted source and drain regions with metallization on the front and back surfaces. The fabrication process flow requires between 60 and 80 individual steps, requiring the overall reliability and yield performance maintained in the NanoFab.

The basic electrical operation of the device is as follows. For these enhancement-mode devices, the upper gate, which extends from the source to the drain, is biased positive to turn on overall conduction. The lower "finger" gates are biased negative in order to inhibit conduction underneath the individual fingers. The combination of these biases forms conducting regions separated at low temperatures by tunnel barriers, such that individual electrons have to quantum-mechanically tunnel from one conducting region (quantum dot) to another. Because of the Coulomb blockade, electron flow on or off the dot in these regions is quantized in units of 1 *e*, and therefore can be made into a current standard. By trapping one or two electrons localized on one or both of the dots, we can start to perform quantum-mechanical manipulations; for instance, by looking for the signature of a quantum coherent oscillation of the position of the electron between the two dots.



Schematic of a full device, including a mesa-etched SOI wire (gray), lower finger gates (aqua), upper gate (blue), source/drain (purple), and metallization (green). The MOSFET control curve is shown as a semi-log plot of current versus gate voltage for a device with no lower gates, and exhibits a 10^4 turn-on ratio.

Results and Discussion: Having started the development of the process about one year ago, we have refined choices for many of the individual process steps, and completed some of the block tests. Fabricating the MOSFET block tested the integration of oxide and polySi growth, large (not nm scale) gate patterning, source and drain implant and activation, and metallization. The graph shows good subthreshold slope over a factor of about 10^4. The Si nanoscale patterning block test gave us the ability to combine negative tone electron beam lithography with dry etching. By going from a fluorine-based to a chlorine-based chemistry, we have been able to achieve this combination.

Recent Publications:

Quantum electrical standards, N. M. Zimmerman, *Physics Today* **63**, 68 (2008).

Lack of charge offset drift is a robust property of Si single electron transistors, E.I Hourdakis, J. A. Wahl, and N. M. Zimmerman, *Applied Physics Letters* **92**, 062102 (2008).

Laser Direct Write to Enhance Inter-Chip IO Bandwidth

Research Participants: R. R. Yu, E. G. Colgan, S. Purushothaman, D. S. Nielsen, R. Wisnieff, and M. M. Denneau
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Project Goal: The goal is to develop a characterization methodology for inter-chip input/output (IO) positional variability in highly integrated composite wafer substrates. This includes developing methods for IO positional correction and IO density enhancement by pattern recognition using laser direct writing (LDR). The intent is to apply this research to future manufacturing processes.

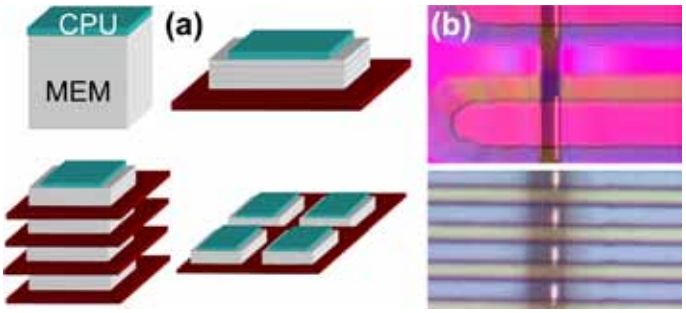
Key Accomplishments:

- Substrate characterization results met target.
- Pattern recognition and laser direct write met requirements.
- Laser direct writing achieved better resolution than monolith.
- Square substrate demonstrated improved process-ability.

Background: Since 2004 device parallelism scaling (multi-cores) has replaced frequency scaling for performance gains in chips within a relatively stable operating system clock frequency range. Several options are currently being actively pursued to increase this parallelism through system integration methods, such as 3D integration (3DI) and system-in-package (SIP). These are alternatives that provide higher system contents in both logic cores and memories than the traditional system-on-chip (SOC) configuration. Structures with high contents in close proximity are of particular interests for super systems aimed at exa-scale computing where a large number of logic cores require an even larger number of supportive memories which are difficult to achieve with SOC on a planar configuration. One of the key development goals for a composite system like 3DI and SIP is the IO bandwidth enhancement between the component wafers. In SOC the IO are well defined and optimized. With integrations, a large number of component device wafers are brought together from different wafer sources to form a composite substrate as a hybrid system. The variations in each component wafer in the composite substrates are magnified and make it difficult to form the required fine pitch IO connections between the component

wafers with a monolithic lithography. This difficulty reduces the IO density. In this study, laser direct write is being evaluated as a mean to re-write the IO connections in the substrates to enhance the IO density in a highly integrated composite system.

Approach: In this work we are evaluating structures that have higher multiple contents than the standard SOC density and are assembled into square substrates which themselves are each a composite assembly of many wafers. Each wafer contains various structural variabilities that occur in different process stages and during substrate assembly. These structural variabilities include those due to processing, overlay tolerance, wafer thinning, wafer dicing, and substrate assembly. The test structure contains various IO pitches which are first overlaid with images by monolithic lithography. The standard monolithic lithography is insufficient for our application needs. The same structure is then scanned using the NanoFab Heidelberg laser direct write pattern recognition system to locate the IO and to record the offsets of the IO from the design positions. The measured offsets are fed into the laser write file to make the necessary correction for each IO and re-direct the IO to the new, ideal locations. The IOs are then again imaged with the monolithic lithography to the new locations and the IO pitch density is evaluated for pitch density improvement.



(a) Some 3DI and SIP options identified in E*xaScale Computing Study: Technology Challenges in Achieving Exascale Systems* (DARPA/IPTO, 2008). (b) Resolution improvement shown by comparing monolithic lithography (top) with laser direct write (bottom).

Results and Discussion: For a full field exposure, the Heidelberg laser writer offers several writing head options for various writing-field areas. The highest resolution is the 2 mm head with 0.6 µm lines at 1.2 µm spacing with a small writing field. At the lowest end is the 20 mm write head with 4 µm lines at 8 µm spacing with a much larger writing field. There is a trade-off between the write speed and the write resolution. We select the maximum write field (20 mm head) for our substrate due to the production throughput consideration. The substrates are first scanned for their positional accuracy using the patterning recognition feature in the Heidelberg tool. This data allows us to characterize and improve our assembly method. We are able to identify at this stage the impact of substrate format on the planar resolution. The square format substrate is then selected over the round format used in the early builds for the better topography, the ease of handling, and for the higher area utilization and design plan for the devices. Based on the structural scan results, a correction factor is uniquely generated for each IO and is directly written to the IO to make the connective offsets for all IO and lead the IO to the new locations, which are located in the ideal design positions. Because each IO offset is different, a large number of scans and re-directs are needed for the entire substrate. The scan results also help us to identify previously unknown substrate variations, such as the size difference between the design data and the actual wafer images between different tool sets, and the positional shift due to the image quality. All these variabilities affected the early work and limited the required IO density; these limitations are being remedied with direct write. As a result, the IO resolution has improved by three times with the 20 mm writing heads as compared with the early monolithic lithography. The direct write also enables us to use square format substrates, resulting in a significant improvement in surface planarity. It is expected that with further learning, the substrate quality and the IO resolution can be further improved to the levels of SOC BEOL.

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Measurement of Current Polarization by Doppler-Shifted Spin Waves

Research Participants: M. Zhu,[1,2] C. L. Dennis,[3] B. D. Soe,[4] M. J. Carey,[5] S. Maat,[5] J. R. Childress,[5] R. Thomas,[6] V. Misra,[6] and R. D. McMichael[1]

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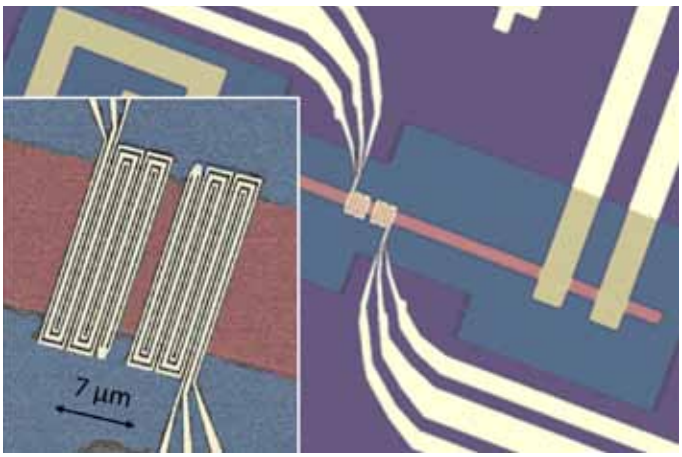
Project Goal: To develop methods for measuring the spin polarization of current in ferromagnetic metals, an often-estimated materials parameter that plays a pivotal role in the spin-torque driven dynamics of magnetic nanodevices.

Key Accomplishments:

- Designed and fabricated devices to measure spin wave propagation at sub-μm wavelengths in current-carrying ferromagnetic metals.
- Measured current polarization in $Ni_{80}Fe_{20}$ (Permalloy) with two times the precision of existing measurements, and provided the first measurements of temperature dependence in current polarization.

Background: Spin-polarized current provides an important and relatively new mechanism for changing the direction of magnetization in novel nanodevices. Spin-polarized current transports spin angular momentum that exerts a "spin transfer torque" on magnetization, providing a low-power alternative to Ampere's-Law fields for flipping "bits" in novel nonvolatile memory technologies, such as MRAM and racetrack memory. The current polarization, *P*, quantifies the relative magnitude of the currents carried by up-spin and down-spin electrons. Despite its importance, until now, measurements of *P* have been primarily limited to interface-sensitive and low-temperature techniques.



False color SEM image of a spin wave Doppler device. The central red stripe is a $Ni_{80}Fe_{20}$ (Permalloy) magnetic metal wire. DC contacts at the ends of the wire in yellow inject current and allow voltage measurements. Curved triple lines are coplanar microwave waveguides connected to antennas. The inset is an expanded view of the antennas that launch and detect spin waves showing the periodic structure that determines the spin wave wavelength. A dielectric layer, shown in blue, insulates the antennas from the wire.

Approach: The magnetic moments of up and down spin electrons are proportional to their angular momentum. Therefore, spin-polarized currents transport both angular momentum and magnetization. In fact, in the limit where the current polarization direction is parallel to the local magnetization, the magnetization in a long, uniform wire should behave exactly as if the magnetization were being carried "downstream" by the electron current at a drift velocity that is proportional to both the current density and the polarization, but inversely proportional to the magnetization. In the spin-wave Doppler measurements, we measure the polarization of the current by observing the effect of the magnetization drift velocity on the propagation of spin waves in magnetic wires. A pair of antennas with periodic structures launches and detects the spin waves while fixing their wavelength. A peak in the transmission between the antennas determines the spin wave frequency, which is Doppler shifted by the magnetization drift velocity.

Results and Discussion: Our initial measurements were performed on $Ni_{80}Fe_{20}$, where we measured Doppler shifts corresponding to drift velocities on the order of 4 m/s in current densities of 10^{11} A/m^2. Using a cryogenic probe station with an integrated electromagnet and both microwave and DC probes, we measure the Doppler shift of the spin wave frequency to obtain the drift velocity and the current polarization. Our room-temperature polarization value of $P = 0.58 \pm 0.02$ is in fair agreement with an independent spin wave Doppler measurement. Between 340 K and 80 K, the polarization increases to 0.75 ± 0.05, trending toward values obtained by earlier analysis of current-perpendicular transport measurements at 4.2 K.

Our second set of experiments, done in collaboration with Hitachi GST, measured the current polarization in $(CoFe)_{1-x}Ge_x$ alloys. These alloys exhibit many of the properties of half-metal Heusler alloys such as high polarization and low damping. They are potentially useful for manufacturing applications because they require annealing at a modest temperature of 245 C that is compatible with established fabrication processes. We found that for increasing Ge concentrations in the range of $0.0 < x < 0.3$, the reduced magnetization causes the drift velocity to increase from 3.1 ± 0.2 m/s to 8.2 ± 0.6 m/s at a current density of 10^{11} A/m^2, but that the polarization increases from 0.84 ± 0.04 at $x = 0.0$ to a broad maximum of 0.95 ± 0.05 at $x = 0.25$. These results are consistent with the first-principles predictions of a pseudogap or low density of states in the down-spin band.

Acknowledgments: This work has been supported in part by the National Science Foundation.

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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).

Enhanced magnetization drift velocity and current polarization in $CoFe_{1-x}Ge_x$ alloys, M. Zhu, B. D. Soe, R. D. McMichael, M. J. Carey, S. Maat, and J. R. Childress, *Applied Physics Letters* **98**, 072510 (2011).

The Effect of Disorder on Magnetic Dynamics

Research Participants: H. Min,[1,2] R. D. McMichael,[1] M. J. Donahue,[3] J. Miltat,[1,2,4] and M. D. Stiles[1]

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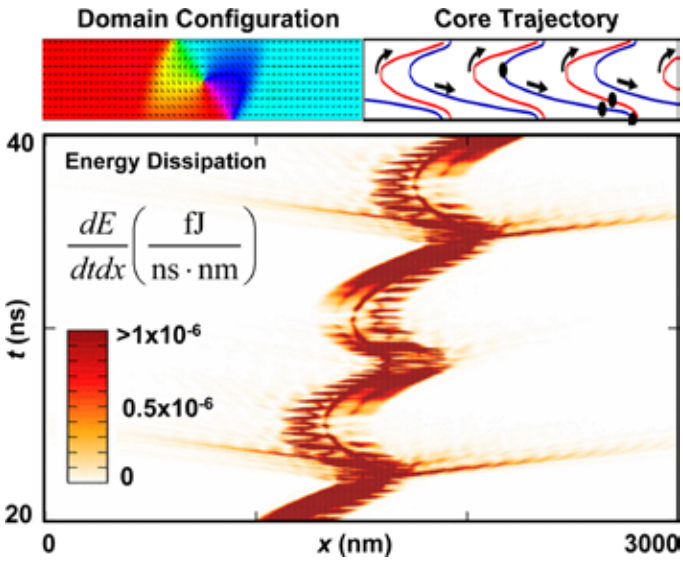
Project Goal: To determine the extent to which disorder can confound the determination of material properties through measurements of magnetic domain wall motion.

Key Accomplishments:

- Simulated vortex wall propagation in a disorder magnetic wire and showed how the results could be interpreted as an increase in the effective damping.
- Determined that similar effects are much smaller for the vortex gyration in a magnetic disk.



Calculation of the energy dissipation during vortex domain wall motion. The magnetic configuration of a vortex wall is shown in the upper left. The magnetization curls around a vortex. When a magnetic field is applied, the domain wall moves down the wire while oscillating from side to side. This motion is shown in the upper right in the location of the vortex core as the wall moves. The wall moves from side to side where the core collides with the edge of the wire and then it reverses direction and moves back. Without energy loss, the wall would simply oscillate in place. The main panel shows the energy loss as a function of time and position along the wire. The energy loss is localized around the vortex core.

Background: Magnetic bits store most of the world's information, either in magnetic tapes or in hard disk drives. Magnetic materials are excellent for storing information because in a small volume, the magnetization typically points in one direction (which can signify a "1") or its opposite (which can signify a "0") and will stay in that state unless power is applied. With the semiconductor road map rapidly approaching difficult physical limits, there is significant effort to develop new ways of storing and processing information. One of the most recent approaches is to store information in magnetic domains in wires made of ferromagnetic materials. Magnetic domains are regions of a sample in which the magnetization is uniform. Domains are separated from each other by domain walls. Manipulating magnetic domains for storing information revives an old technology – magnetic bubble memory, but with a new twist. When a magnetic wire gets small enough, it becomes possible to move the domains, or rather the walls between the domains, by running a current through the wire rather than by applying a magnetic field. This new approach scales better to lower sizes and raises the possibility of high density, non-volatile, and fast memory. Designing such devices requires the ability to measure the appropriate material properties.

Approach: We take a theoretical approach to understanding domain wall motion. In particular, we use calculations to facilitate the interpretation of measurements. There have been a number of measurements of domain wall motion driven by current. Typically the experiments measure the domain wall velocity as a function of the current and an applied magnetic field. These measurements are then interpreted in terms of models of domain wall motion in idealized magnetic wires. However, imperfections have a strong effect on the motion and therefore on the interpretation of the experimental results. To understand the consequences of disorder on the measurement, we have carried out micromagnetic simulations of domain wall propagation and developed simpler models to interpret the results in a framework that can be used to interpret experiments. Micromagnetic simulations are based on a continuum description of magnetic materials on an intermediate length scale. We use the Object Oriented MicroMagnetic Framework, software developed at NIST, for the simulations and run them on a Beowulf cluster at NIST. We analyze the results of the simulation using a model in which all the details of the domain wall are reduced to two numbers giving the wall's position in the wire.

Results and Discussion: For a domain wall to move down the length of a wire due to an applied magnetic field, the magnetic system has to lose energy to the lattice. If there were no energy loss, the domain wall would just oscillate without making any forward progress. The figure shows how energy loss near the vortex core allows the wall to move forward.

Repeating the calculation in the presence of disorder generates an important caveat to the interpretation of such experiments. We find that when the vortex core and other sharp features of the vortex structure move through the disorder, the wall gets excited and loses energy faster. Since the wall loses energy faster, it moves down the wire more rapidly, giving the counterintuitive result that, up to a limit, the wall moves faster the more disorder there is in the wire. It turns out to be possible to analyze this behavior using a simple description in terms of an increased effective damping. The fact that the intrinsic damping in the system can differ significantly from the value that would be interpreted from experiment is important both for the analysis of experiments and for the design of devices.

Recent Publications:

Effects of disorder and internal dynamics on vortex wall propagation, H. Min, R. D. McMichael, M. J. Donahue, J. Miltat, and M. D. Stiles, *Phys. Rev. Lett.* **104**, 217201 (2010).

Effects of disorder on magnetic vortex gyration, H. Min, R. D. McMichael, J. Miltat, and M. D. Stiles, *Physical Review B* **83**, 064411 (2011).

Ultra-High-Density Patterned Magnetic Recording Media

Research Participants: E. C. Choi,[1,2] M. Young Oh,[1,2] S. Jin,[1,2] R. Kasica[3], and L. Chen[3]

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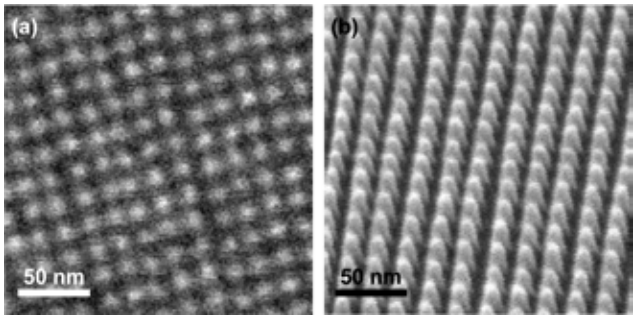
Project Goal: The ultimate objective of this project is to develop large-area, nanoimprint lithography techniques for applications such as ultra-high-density, patterned magnetic recording media with at least 1 terabit per square inch (Tb/in^2) recording density—about five times higher than the current hard disk memory in the market. Such a high recording density requires patterned media with a recorded magnetic island bit size of ≈12.5 nm in diameter or smaller, arranged in a periodic fashion.

Key Accomplishments:

- Nano-patterning of a Si substrate and patterned island formation with a bit density as high as 1.6 Tb/in^2 has been accomplished in the CNST NanoFab using electron beam lithography and nanoimprinting. A bit size on the order of 10 nm in diameter has been achieved with a spacing of similar dimension. The magnetic switching behavior of patterned bit islands of $(CoPd)_n$ perpendicular anisotropy magnets has been investigated.
- A planarization technique to remove the topographically uneven surface configuration of magnetic island arrays has also been demonstrated, which eliminates the magnetic material outside the pillar top bit locations to reduce magnetic signal interference, and also to allow the read/write recording head to easily fly on the hard disk media surface.

Background: Significant advances have been made in recent years in the field of nanomaterials and nanodevices, with their size scales continuing to decrease. While exciting nanomaterials concepts have been demonstrated with carbon nanotubes, semiconductor nanowires, quantum dot particles, and so forth, the progress toward large scale integration of these materials and devices into useful electronic devices and system arrays has been rather slow, largely due to the lack of industrially viable and efficient nanofabrication techniques. For hard disk drive magnetic recording, one of the key components in computer technologies, a storage density of 1 Tb/in^2 or higher (approximately five times current hard disk drive media) is desired, and a viable fabrication technique is needed to achieve this density.



SEM images showing fabrication of bits at 1.6 Tb/in^2 by (a) electron beam patterning of HSQ resist at 20 nm pitch and (b) Si pillar array from the exposed HSQ created by RIE pattern transfer, with pillars 10 nm in diameter and 20 nm high.

Approach: In order to accomplish such a large memory density, bit-patterned media having periodically spaced islands of magnetic recording material are preferred because the undesirable magnetic interaction between adjacent bits and associated reduction in signal-to-noise ratio can be minimized. To obtain such a high density memory, advanced electron beam lithography is used and the pattern transferred to form periodic silicon pillar arrays. The behavior of magnetic islands deposited on the pillar top is also investigated. Furthermore, planarization of topographically uneven features has been achieved using SiO_2 filling of the valley space, to obtain cleaner magnetic signals and read/write head flyability from the hard disk surface.

Results and Discussion: As shown in the figure, a negative resist, hydrogen silsesquioxane (HSQ), was used for electron beam lithography, with different island size and density. A patterned/developed HSQ resist island array was then pattern transferred to silicon surface to obtain a periodic Si pillars, obtaining a bit density as high as 1.6 Tb/in^2.

In the patterned magnetic media, the magnetic islands need to be separated with minimal magnetic material in between spaces to minimize magnetic signal interference. In addition, the topographical configuration should be planar on the surface so as to allow the read/write head slider to fly. As a demonstration, we fabricated a ≈100 nm-diameter diameter periodic array of Si nano pillars using electron beam lithography and nano imprint lithography (NIL). Each of the samples contained approximately 100 million Si nanopillars over a relatively large area of 0.6 cm x 0.6 cm. The vertically anisotropic magnetic material, [Ta 3 nm\Pd 3 nm\[Co 0.3 nm\Pd 0.8 nm]$_8$] was sputter deposited on top of the Si islands as well as in the valleys (trenches). A geometrical planarization process was then applied, using HSQ spin coating, back etch, and conversion of the trench HSQ into SiO_2 by annealing. Because the HSQ gets preferentially etched over the magnetic material, the end result is a planar surface with islands of high coercivity magnetic recording material exposed only on top of the pillars. Much cleaner magnetic force microscopy images were obtained, with no obvious inter-bit magnetic interactions, and excellent read/write head flyability was demonstrated. Patterned media without such planarization did not allow the recording head to fly off the disk surface at all.

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Planarization of discrete track recording media to improve flyability of magnetic recording sliders, Y. Yoon, C. Choi, Y. Oh, D. Hong, S. Jin, and F. E. Talke, *IEEE Transactions on Magnetics* **45**, 3527-3530 (2009).

Highly self-assembled nanotubular aluminum oxide by hard anodization, K. Noh, K. S. Brammer, H. Kim, S.-Y. Jung, T.-Y. Seong, and S. Jin, *Journal of Materials Research* **26**, 186-193 (2011).

Magnetic Nanopillars Fabricated Using Electron Beam Lithography

Research Participants: R. B. Comes,[1] M. Gu,[1] M. Khokhlov,[1,2] R. Kasica,[3] G. Henein,[3] J. Lu,[1] and S. A. Wolf[1]

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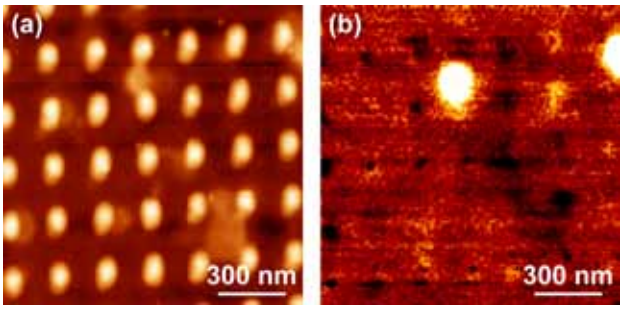
Project Goal: $CoFe_2O_4$ nanopillars will be fabricated with perpendicular magnetic anisotropy to produce magnetic quantum cellular automata.

Key Accomplishments:

- Fabricated smallest reported $CoFe_2O_4$ nanopillars using electron beam lithography.
- Demonstrated magnetic force microscopy imaging of nanopillars and dipole interactions between pillars.

Background: Spintronic logic presents an exciting magnetic alternative to traditional CMOS-based transistor logic. The magnetic quantum cellular automata (MQCA) approach is a leading candidate for this type of magnetic switch. A bit is represented using 4 magnetic nanopillars in a square array. There are two possible minimum energy ground states for this system, with one state representing a '0' and the other a '1'. Our work attempts to develop an MQCA system using $CoFe_2O_4$ (CFO) for the magnetic pillars because of its potential as a magnetoelectric material—a material whose magnetic moment can be controlled using an electric field.

Approach: CFO thin films were deposited on MgO and $SrTiO_3$ single crystal substrates using a pulsed electron deposition (PED) system at the University of Virginia (UVA). A Si capping layer was deposited on the surface of the CFO films via radio frequency (RF) sputtering in the CNST NanoFab to promote resist adhesion and for use as a sacrificial mask during etching. Electron beam lithography (EBL) was performed using hydrosilsesquioxane (HSQ) as a negative-tone electron beam resist with the NanoFab's Vistec EBL system. The samples were then etched using the Cl_2 ICP/RIE system to selectively remove the Si layer in the unexposed areas, leaving nanopillars in the area where the HSQ resist was exposed. An Ar mill was performed to etch through the CFO film to the substrate to transfer the nanopillar pattern into the film. The resulting CFO nanopillars were then characterized using a magnetic force microscope (MFM) system at UVA to measure the effects of dipole coupling between neighboring pillars.



(a) AFM and (b) corresponding MFM image of CFO nanopillar array. Pillar heights are approximately 35 nm above the surface, with diameters of approximately 75 nm and a pitch of 200 nm between the center of neighboring pillars. The MFM image shows magnetic contrast from the pillars, the first such demonstration. There is also evidence of magnetic dipole interactions between the pillars in some cases, which is a good first step towards the eventual goal of achieving an MQCA system.

Results and Discussion: The films were successfully grown in the PED system at UVA and were characterized to determine their magnetic and crystalline properties. Films on MgO substrates exhibit excellent epitaxial properties and have high perpendicular magnetic anisotropy when measured using a vibrating sample magnetometer (VSM). Films on $SrTiO_3$ were also epitaxial, but did not exhibit significant perpendicular anisotropy in VSM scans. Both films were patterned to create nanopillars, but for brevity only the pillars on MgO will be discussed here.

Using the Vistec, we were able to pattern pillars in the HSQ resist as small as 20 nm in diameter, with dot-to-dot pitch of 60 nm. After etching the resulting pillars have heights of approximately 35 nm above the substrate surface. However, the Ar milling process leads to an increase in the pillar diameter due to re-deposition of the etched material onto the sidewalls of the pillars. The resulting pillars have diameters of approximately 75 nm at the end of the process. For this reason, MFM characterization focused on pillars with a pitch of 200 nm for the initial testing. While larger than the EBL patterned pillars, these pillars still represent the smallest features produced yet on a CFO film. We found that the pillars maintain their magnetic moments after processing and exhibit dipole interactions even on the relatively large length scale of 200 nm spacing.

Future work will focus on ways to enhance the magnetic properties of the films to demonstrate dipole coupling for MQCA logic, while also reducing the dimensions of the system by improving the Ar milling process. We will also conduct experiments to produce self-assembled CFO pillars by templating a $SrTiO_3$ substrate using the Vistec EBL and depositing additional material after patterning.

Acknowledgments: R. B. Comes gratefully acknowledges support through the National Defense Science and Engineering Graduate Fellowship Program. This research was supported by Nanoelectronics Research Initiative, the National Science Foundation, and the Defense Advanced Research Projects Agency.

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The promise of nanomagnetics and spintronics for future logic and universal memory, S. A. Wolf, J. Lu, M. R. Stan, E. Chen, and D. M. Trager, *Proceedings of the IEEE* **98**, 2155-2168 (2010).

Imaging 3D Magnetic Nanostructure in Ferromagnetic Multilayers

Research Participants: B. McMorran,[1] D. T. Pierce, [1] J. E. Davies,[2,3] P. Morrow,[3] C. L. Dennis,[3] J. W. Lau,[3] R. K. Dumas,[4] P. Greene,[4] K. Liu,[4] and J. Unguris[1]

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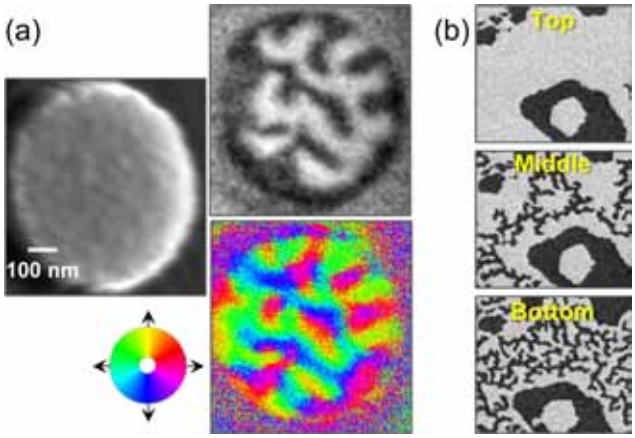
Project Goal: To develop sample preparation methods that allow for direct imaging of the detailed nanomagnetic structure in delicate magnetic materials and devices that are used for magnetic storage and magneto-electronics.

Key Accomplishments:

- Developed plasma-based procedures for cleaning and depth profiling ultra-thin magnetic multilayers while minimizing interfacial mixing and roughening.
- Imaged the complete magnetic nanostructure, including all three components of the magnetization vector, as a function of depth in patterned and unpatterned CoPd multilayers grown with graded magnetic anisotropy.

Background: Imaging magnetic structure at the nanoscale is a challenging and essential part of developing new magnetic materials and devices. Modern nanofabrication techniques are pushing magnetic structures towards smaller feature sizes and higher densities which test the spatial resolution of current magnetic imaging techniques. Magnetic recording media are a prime example. Demand for higher storage densities has resulted in multilayer nanostructures for perpendicular anisotropy, nanolithography for patterned media, and graded or exchange coupled multilayer films for combining thermal stability with write ability.



SEMPA images of (a) a patterned Co/Pd multilayer film showing the topography, perpendicular magnetization (gray scale), and in-plane magnetization direction (color); and (b) the depth dependent magnetic structure of a graded Co/Pd multilayer film near the top (low anisotropy), middle (medium anisotropy), and bottom (high anisotropy) of the film.

Our goal is to develop tools and procedures to image the magnetic structure within these materials and devices, both with high spatial resolution and, where possible, with nanometer depth resolution. We previously showed that conventional argon ion sputtering can be used to depth profile magnetization in 6 nm thick layers of Co/Cu mulitlayers. However, the layers in perpendicular media such as Co/Pd are ten times thinner and much more susceptible to ion damage. We have therefore developed low-energy ion milling techniques that reduce the extent of the ion damage and enable magnetic depth profiling of these materials.

Approach: Patterned and continuous film test samples based on $[Co\ (0.4\ nm)/Pd\ (0.6\ nm)]_{60}$ multilayer films were grown at room temperature using dc magnetron sputtering. During deposition, the Ar gas pressure can be varied to change the interfacial roughness and grain size, producing a thickness dependent anisotropy in the film. Optical lithography and ion milling through an alumina mask are used to pattern some of the films, and all of the films are surface treated with a 6 nm thick Pd film.

We use scanning electron microscopy with polarization analysis (SEMPA) to image the magnetic structures. SEMPA images the magnetization by measuring the spin polarization of secondary electrons generated in a scanning electron microscope. The secondary electron spin polarization is directly related to the magnitude and direction of the sample magnetization. Our SEMPA tool can measure all three components of the magnetization vector. The spatial resolution of our SEMPA is at best 10 nm, while the probing depth is approximately 1 nm, determined by the escape depth of the polarized secondary electrons. The small probing depth provides excellent thin film sensitivity, but also demands atomically clean surfaces for imaging. Samples were cleaned and milled using low-energy (50 eV) Ar ions produced by a microwave coupled plasma. The average milling rate was 2.5 nm/min.

Results and Discussion: The ion milling procedures were first tested by comparing SEMPA images from samples milled using low energy ions to samples cleaned using conventional higher energy (> 1 keV) ion beam sputtering. The samples cleaned with high energy ions clearly showed degradation of the perpendicular anisotropy. The high energy ion milling disturbed the well-ordered multilayer structure which diminished the perpendicular anisotropy, and the deeper we milled, the worse the damage. Ion milling with low energy ions, on the other hand, produced no observable damage even when we milled through most of a uniform sample. An example of a SEMPA image from a patterned Co/Pd sample after removing the Pd capping layer with low-energy ion milling is shown in the figure.

Low-energy ion milling was then used to depth profile the magnetic structure of Co/Pd films grown with a thickness dependent graded anisotropy. The figure shows depth dependent SEMPA images of the magnetic structure of a graded Co/Pd multilayer film with low anisotropy near the top, medium anisotropy in the middle, and high anisotropy near the bottom of the film. The SEMPA images show the domain fine structure increasing as expected with increasing anisotropy.

Recent Publications:

Measuring the effects of low energy ion milling on the magnetization of Co/Pd Multilayers using scanning electron microscopy with polarization analysis, B. J. McMorran, A. C. Cochran, R. K. Dumas, K. Liu, P. Morrow, D. T. Pierce, and J. Unguris, *Journal of Applied Physics* **107**, 09D305 (2010).

Reversal of patterned Co/Pd Multilayers with graded magnetic anisotropy, J. E. Davies, P. Morrow, C. L. Dennis, J. W. Lau, B. McMorran, A. Cochran, J. Unguris, R. K. Dumas, P. Greene and Kai Liu, *Journal of Applied Physics* **109** (2011), *in press*.

Measuring Light-Matter Interactions in Chip-Based Optical Cavities

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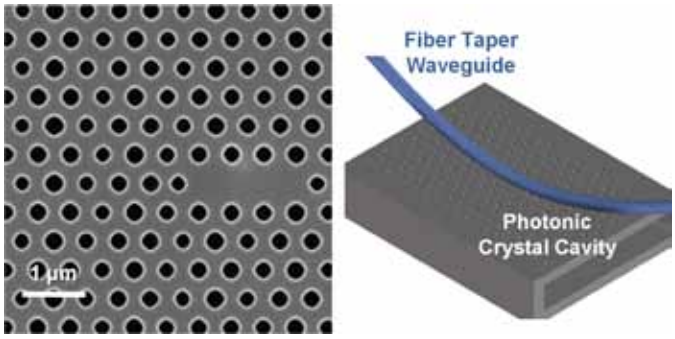
Project Goal: To develop special near-field probes and nanofabricated optical cavities for measuring light-matter interactions with exquisite sensitivity, down to the single photon, single emitter level, and to thereby enable potential applications in classical and quantum information processing.

Key Accomplishments:

- Designed and constructed a cryogenic fiber probe station for efficient nanophotonic device characterization.
- Measured optical properties of PbS colloidal quantum dots integrated with silicon nanophotonic resonators.

Background: Measuring interactions between light and matter has both fundamental and practical importance. For example, lasers have had a profound influence on science and industry over the past 50 years. More recently, devices exploiting light-matter interactions at the smallest scale, between a single photon and a single atom, have been a proving ground for ideas in quantum mechanics and a test bed for quantum-information-processing applications. Our goal is to develop measurement tools to probe these interactions in solid-state systems. We focus on nanoscale light-emitting materials such as epitaxially grown quantum dots and solution-processed colloidal nanocrystals. These are solid-state structures that exhibit many of the important properties of isolated atoms. We measure these nanostructures both as-grown and also when integrated into nanofabricated devices like optical cavities, where light can be confined to a small volume for many thousands of optical cycles. Placing quantum dots in optical cavities enhances their interaction with light, and gives rise to a variety of physical phenomena that can be used in information processing devices.



Scanning electron microscope image of a silicon photonic crystal cavity containing PbS quantum dots, and a schematic of the fiber coupling approach.

Approach: We fabricate optical cavities in the semiconductors GaAs and Si using electron beam lithography and plasma etching. The cavities are integrated with quantum dots in two ways. For GaAs, a layer of InAs quantum dots has been embedded in the GaAs device layer during its epitaxial growth. For Si devices, solution-processed PbS colloidal quantum dots are spin-coated on the samples after device fabrication (see figure). The quantum dots emit in the 980 nm/1300 nm (InAs/GaAs) and 1550 nm (PbS/Si) wavelength bands. To measure these structures, we have built a custom cryogenic fiber probe station, whose key component is an optical fiber taper waveguide, fabricated out of standard telecommunications optical fiber. The waveguide diameter starts at 125 μm, standard for an optical fiber, and gradually decreases to about 1 μm, before gradually increasing back to 125 μm. It is positioned so that its narrowest section is adjacent to the cavity (see figure). At this juncture, a fraction of the light sent through the waveguide tunnels into the cavity, where it interacts with the quantum dot(s). Some of the light then tunnels back into the waveguide, and we measure the properties of this exiting light to identify the quantum dot interaction. Working in a cryostat provides access to temperatures between 6 K and 300 K, a range over which the quantum dot behavior varies widely. The optical fiber interface allows for a variety of different systems to be interrogated by simply exchanging the excitation source and detector.

Results and Discussion: In the InAs/GaAs system, we have focused on single quantum dots, measuring their fluorescence spectra, time-resolved emission, and photon statistics. Cavities fabricated in the CNST NanoFab have exhibited very high quality factors (low optical loss), and will be used to follow up on previous results in which we demonstrated that a single quantum dot in a cavity can strongly modify its response and forms a coupled quantum-dot-photon quantum system, similar to two atoms forming a molecule. The net result is a device in which a single semiconductor quantum dot, a structure with a characteristic length scale of 10 nm, can be effectively connected to the macroscopic fiber optics that are ubiquitous in modern communications. Through a detailed characterization of the device's electromagnetic properties, we have shown that this structure can perform highly efficient fluorescence spectroscopy and resonant scattering measurements on a single InAs quantum dot. We are also investigating other device architectures, including single quantum dots embedded in dispersion-engineered semiconductor waveguides.

In the PbS/Si system, we have interrogated a low density of PbS quantum dots that have been spun onto silicon photonic crystal cavities (see figure). We have confirmed that the quantum dot integration does not degrade the cavity's loss, and have performed measurements of cavity-filtered photoluminescence, looking at effects such as quantum dot "blinking" (fluorescence intermittency) and saturation. Going forward, we hope to improve the sensitivity of measurements on this system, eventually achieving the capability to characterize a single PbS quantum dot.

Recent Publications:

Spectroscopy of 1.55 μm PbS quantum dots on Si Photonic crystal cavities with a fiber taper waveguide, M. T. Rakher, R. Bose, C. W. Wong, and K. Srinivasan, *Applied Physics Letters* **96**, 161108 (2010).

Fiber-coupled semiconductor waveguides as an efficient optical interface to a single quantum dipole, M. Davanço and K. Srinivasan, *Optics Letters* **34**, 2542-2544 (2009).

Linear and nonlinear optical spectroscopy of a strongly-coupled microdisk-quantum dot system, K. Srinivasan and O. Painter, *Nature* **450**, 862-865 (2007).

Probing Plasmonic-Photonic Interactions in Periodic Nanowire Arrays

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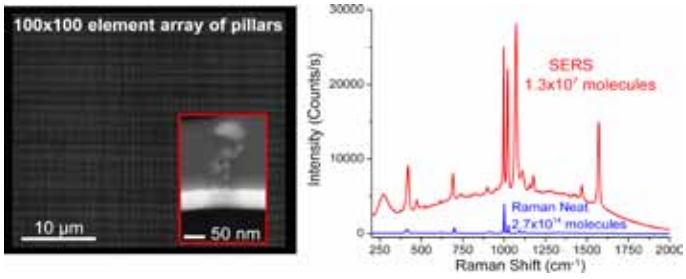
Project Goal: To investigate the influence of photonic and interparticle effects on plasmonically-enhanced optical processes in periodic nanowire arrays for improved optical sensors and enhanced optical emitters.

Key Accomplishments:

- Fabricated periodic arrays of metal-coated semiconductor nanowires that exhibit average surface enhanced Raman scattering (SERS) enhancements greater than 1×10^8.

- Observed plasmonic interactions between Au/Ag nanowire "caps" and underlying films that lead to over an order of magnitude increase in SERS output.

Background: Plasmons are coherent, resonant oscillations of electrons within metals, typically confined at surfaces and/or interfaces with dielectric materials. These oscillations induce large, localized electric fields that enhance optical processes such as the absorption, Raman scattering (SERS), fluorescence (SEFS), and/or luminescence. In nanostructures made of plasmonic metals, typically gold and silver, plasmons can be optically stimulated at the surface plasmon resonance (SPR) frequency, which is defined by the nanoparticle geometry. Research in the field of plasmonics has led to the development of plasmonic waveguides, superlenses, enhanced solar cells and optical antennas, and dramatic advancements in molecular spectroscopy, and has led to new fields of study, such as metamaterials. However, until very recently the vast majority of efforts in the field of SERS has focused either on isolated single or coupled plasmonic particles. This focus led to a great deal of basic physics being explored and the observation of single molecule detection within tightly spaced (< 10 nm) nanoparticles due to plasmonic coupling. However, the enhancements that were observed are highly localized within that small gap between the particles and therefore not ideal for attaining the large average enhancement factors that are necessary to realize enhanced optical sensors and emitters desired in most commercial and military applications. Our goal is to design and fabricate large-area, periodic arrays of plasmonic nanowires with varying particle dimensions, inter-particle separations, and array sizes. We are focusing on Au- and Ag-coated semiconductor nanowire arrays, probing the role that the array periodicity and nanowire architecture have upon the properties of the plasmonic fields and optical enhancements observed.



SEM image of 100 × 100 array of Au-coated Si nanowires. INSET: TEM image of individual nanowire. The bulk Raman spectrum of Neat (blue) and SERS spectrum of benzene thiol on Au nanowires (red), shows the 7 orders of magnitude increase in the number molecules detected.

Approach: We fabricate periodic and aperiodic arrays of semiconductor nanowires such as Si, GaN, ZnO, and SnO_2 using electron beam lithography, reactive ion etching, and vapor-liquid-solid growth. These nanowires are overcoated with Au or Ag using a variety of deposition techniques, including electron beam evaporation, sputtering, and atomic layer deposition. These efforts create arrays of core-shell, Au- or Ag-coated nanowires (see figure). These structures may be used as substrates for SERS detection of adsorbed molecular species or in the case of direct-band-gap semiconductor nanowires, as enhanced emitters. We probe the SERS intensity using both micro- and telescopic Raman spectroscopy, where the enhancement factors of the array structures may be probed as a function of nanowire diameter and height, inter-wire gap, array size, and the characteristics of the plasmonic metal coating (metal thickness, uniformity, conformality, and roughness). These measurements are carried out at a variety of wavelengths within the UV-Vis-NIR range. Telescopic Raman spectroscopy is also utilized to probe the efficiency of these arrays for use in stand-off detection of dangerous molecular compounds such as explosive or chemical warfare agents. The emission efficiency of luminescence nanowire arrays is measured with micro-photoluminescence spectroscopy.

Results and Discussion: In the Si nanowire arrays, we have focused on probing their use in SERS detection schemes. Periodic arrays of nanowires that were fabricated at the CNST NanoFab and NRL Nanoscience Institute have exhibited extremely high, uniform SERS enhancements, with average enhancement factors approaching 10^9 (see figure). These structures have a peak response that is strongly dependent upon the nanowire diameter, with the peak diameter being dependent upon the metal used for the coverage and the incident laser wavelength. The enhancement factor is also weakly dependent upon the interparticle gap. Theory predicts that as the particles are spaced closer to one another, coupling between the stimulated plasmons will lead to significantly larger electric field intensities and therefore much larger enhancement factors. This effect has previously been observed in coupled clusters of plasmonic particles; however, in the case of the periodic arrays, the largest enhancement factors were observed for the most widely spaced arrays. Our array structures have also been used to detect SERS from various compounds at distances of 10 m.

Recent Publications:

Plasmo-photonic nanowire arrays for large-area surface-enhanced Raman scattering sensors, J. D. Caldwell, O. J. Glembocki, R. W. Rendell, S. M. Prokes, J. P. Long, F. J. Bezares, *Proceedings of SPIE* **7757**, 775723 (2010).

Probing plasmonic confinement: the role of propagating and localized surface plasmons for SERS enhancement in periodic nanostructures, F. J. Bezares, J. D. Caldwell, O. J. Glembocki, R. W. Rendell, M. Feygelson, M. Ukaegbu, R. Kasica, L. Shirey, N. D. Bassim, and C. Hosten, *Nano Letters, submitted.*

Efficiency Enhancement of Copper Contaminated Radial *p-n* Junction Solar Cells

Research Participants: A. Boukai,[1] P. Haney,[2] A. Katzenmeyer,[3] G. M. Gallatin,[2] A. A. Talin,[2] and P. Yang[4]

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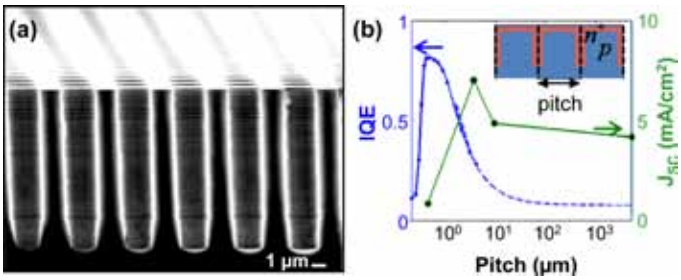
Project Goal: To demonstrate and characterize radial *p-n* junction photovoltaic devices with simulated lower cost and less pure metallurgical grade Si, and to develop a quantitative, physics-based model to explain the experimental observations.

Key Accomplishments:

- Fabricated and characterized the performance of a Si-based array of radial *p-n* junctions with high Cu contamination.
- Demonstrated its superior photovoltaic performance compared to a planar Si *p-n* junction device.
- Developed a two dimensional model of a *p-n* junction array to explain the dependence of performance on junction areal density.



(a) SEM cross section of a radial junction Si solar cell; (b) Calculated internal quantum efficiency (IQE) and measured short circuit current density (J_{sc}) versus pitch of the nanowire array. Both show a similar trend of a maximum at twice the depletion width. Note the model is 2D while the sample is 3D, and the model parameters are such that it has a smaller depletion width than the experimental system.

Background: The need to employ high purity semiconductor grade Si in the fabrication of Si solar cells significantly increases their production costs. Given silicon's long optical absorption length, high purity Si is used to provide the diffusion length needed for efficient carrier extraction. Unfortunately, using cheaper, metallurgical grade Si substantially degrades cell performance due to its shorter carrier diffusion length. Radial *p-n* junction solar cells based on vertical arrays of Si nanowires orthogonalize, to a first approximation, the direction of the light absorption and the minority carrier diffusion necessary to reach the junction. This approach has the potential to circumvent the requirement for high purity silicon in photovoltaic devices. We performed the first experimental measurements on radial *p-n* junction arrays. To simulate the effects of impurities in metallurgical grade Si, we intentionally contaminate our devices with Cu impurities. Radial junctions contaminated with Cu impurities show roughly a two-fold increase in efficiency compared to similarly contaminated planar *p-n* junction solar cells; however, the enhancement is a strong function of the radial junction pitch, with maximum enhancement occurring at a pitch that is twice the carrier diffusion length.

Approach: Radial junction arrays were fabricated by a combination of lithography and deep reactive ion etching to form 10 μm deep vias in boron doped Si wafers. This was followed by forming *p-n* junctions using a spin-on glass phosphorus source. The vias had diameters of 2 μm and 0.13 μm, and a pitch of 10 μm, 4 μm, and 0.5 μm (see figure). To simulate the effects of Cu impurities in metallurgical grade Si, we deposited Cu films on the backside of our devices and then thermally annealed them. Cu contamination decreased the electron diffusion length from approximately 500 μm for clean Si to approximately 0.3 μm for contaminated Si, as measured using an electron beam induced current technique. The solar-to-electrical power conversion efficiency was measured in a system calibrated to deliver 100 mW/cm^2 photon flux (1 sun) through an atmospheric mass 1.5 filter.

Results and Discussion: The shorter minority carrier diffusion length in Cu-contaminated Si lowered the efficiency of planar junction devices by an order of magnitude, approximately from 8 % to 0.7 %. As expected, the efficiency of similarly contaminated radial *p-n* junction arrays with 10 μm pitch improved to 1.1 %. Reducing pitch to 4 μm increased efficiency to 1.8 %, an improvement of approximately 40 %. This improvement can be attributed to carriers having a shorter distance to diffuse before being collected. Surprisingly, the device with the shortest distance between adjacent *p-n* junctions showed a dramatically decreased efficiency, performing even worse than the planar device. To explain this decrease, we developed a two dimensional model with heavily *n*-doped Si 'fingers' inserted into a lightly *p*-doped Si matrix, and calculated the internal quantum efficiency by numerically solving the drift-diffusion and Poisson equations. Varying the distance between adjacent junctions from a few to thousands of nanometers reproduced the experimentally observed trend. At a large pitch, decreasing the junction to junction distance dramatically improves efficiency by increasing the fraction of collected carriers, or decreasing the fraction lost to non-radiative recombination at Cu centers. This trend begins to saturate when the distance between the junctions approaches the carrier diffusion length, because further decreases do not improve collection efficiency. The performance drops sharply when the distance between the junctions falls below twice the depletion width. At that point, the *p-n* junction is not fully formed. This deficiency increases the minority carrier concentration and therefore the recombination rate for the light-generated electron-hole pairs.

Recent Publications:

Efficiency enhancement of copper contaminated radial *p-n* junction solar cells, A. Boukai, P. Haney, A. Katzenmeyer, G. M. Gallatin, A. A. Talin, and P. Yang, *Chemical Physics Letters* **501**, 153-158 (2011).

Nanoscale Characterization of Organic Photovoltaic Devices

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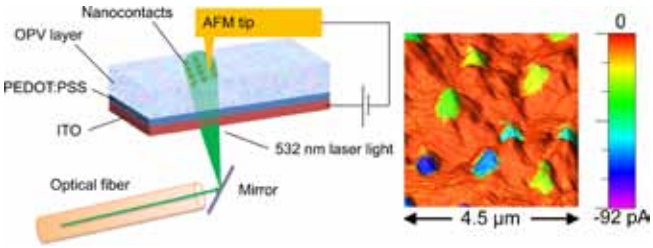
Project Goal: To develop measurement techniques capable of mapping the functional properties of novel nano-structured photovoltaic (PV) materials at the relevant length scales.

Key Accomplishments:

- Used nanocontacts fabricated with a minimally invasive processing scheme to probe the photoelectric response in organic PV (OPV) materials at 200 nm to 1000 nm length scales.
- Explored the applicability of conducting AFM probes for characterizing photo-response down to a 30 nm length scale.

Background: The performance of bulk heterojunction (BHJ) organic solar cells has recently improved significantly and much effort is under way to further explain the photophysical phenomena and mechanisms responsible for charge generation and transport in these systems. In these materials, electron donors (usually polymers) and acceptors (fullerene derivatives or other small molecules) are intimately mixed throughout the material. Recent performance improvements have been associated with better active layer ordering in both the polymer and the fullerene regions, resulting in enhanced optical absorption and increased carrier mobility. These improvements have led to increased interest in the role of the nanomorphology of the active layer on exciton dissociation and on the transport of free charge carriers inside the material and across collecting contact interfaces.

Approach: When investigating the role of nanomorphology on charge transport in organic photovoltaic films, greater insight is obtained when structural characterizations are combined with optical or electrical measurements at the nanoscale. We use a photoconductive AFM (see figure) to probe arrays of nanoscale metal electrodes (Ag, Ca, Au) fabricated directly on top of the active layer without lithographic patterning on the sensitive organic film. These nanodots act as miniature versions of macroscopic devices and therefore make data interpretation more straightforward. The nanoscale contacts were deposited by metal evaporation through silicon nitride stencil masks fabricated in the CNST NanoFab with arrays of small openings in the shape of dots as defined by electron beam lithography and an etch process. We compared the photoconductive AFM (PCAFM) measurements with different conductive tips to the properties of these nanodevices. We investigated BHJs based on the blends of poly(3-hexylthiophene) (P3HT) and [6,6]-phenyl C61-butyric acid methylester (PCBM) that have produced the most efficient devices.



Schematic of the photoconductive AFM measurement configuration, and the measured photo response of a 300 nm nanodot array under short circuit illumination conditions. The photocurrent color-coded map is superimposed directly on top of the 3D topography.

Results and Discussion: The nanocontact data and photoconductive AFM maps both consistently reveal that high efficiency electron collection is limited to sparse hot spots. Among different dots, a significant variation (up to a factor of 10) in the photocurrent response is observed. The surface topography by itself is very rough with peak to valley variations of up to 40 % of the total film thickness. We found no correlation between the topography and the photocurrent data. This suggests that variations in the material organization that affect topography have only minor effects on device performance. An analysis of nanodot photoresponse as a function of size showed that as the nanodot size increases, fluctuations in the short circuit current among different dots are reduced.

The diamond-coated tips used on the nanodots also worked well for direct contact and photocurrent measurements on the bare OPV film. The variations in photocurrent can be as large as an order of magnitude over lateral dimensions as small as 100 nm. The largest photocurrent is observed in "hot spots" of current which vary from tens of nanometers up to 200 nm across. The PCAFM results from the top surface are consistent with an electron blocking skin (P3HT) and dilute hot spots corresponding to PCBM nanocrystals on the top surface.

In an attempt to access the bulk nanomorphology, we used ultra-low angle microtomy cut the OPV film, removing the surface layer and exposing wedges along the cut directions. High-resolution AFM imaging along each slice shows that the top morphology is enriched with a P3HT skin. The bulk morphology transitions abruptly into the

top film morphology, suggesting a very thin skin layer. Using several line scans across this transition region, we estimated a top layer thickness of 10 nm or less, confirming that the gross inhomogeneities occur at the surface rather than in the bulk organization. These results are consistent with published spectroscopic data revealing an abundance of polymer species at the air/OPV interface, as well as tomography data showing fine blending within the bulk.

Recent Publications:

Origin of nanoscale variations in photoresponse of an organic solar cell, B. H. Hamadani, S. Jung, P. M. Haney, L. J. Richter, and N. B. Zhitenev, *Nano Letters* **10**, 1611-1617 (2010).

Self-Assembly of Lithographically Patterned Cubic Nanostructures

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Project Goal: To develop strategies for mass fabrication of precisely patterned 3D nanostructures.

Key Accomplishments:

- Demonstrated the use of nano-imprint lithography and self-assembly to synthesize 3D nanostructures.
- Applied nanoimprint lithography to create nanoporous membranes.

Background: Present day nanofabrication methods such as electron beam and nanoimprint lithography enable exquisite patterning, but are limited to planar fabrication. We have been exploring the use of electron beam and nano-imprint lithography to precisely pattern 2D structures enabling them to be structured in 3D. Patterning of heterogeneous materials is very important to the creation of electronic, optical, and biomedical devices. In the absence of patterning, device functionality is severely limited. The state of the art in the creation of nanoparticles is limited to simplistic shapes like spheres and cylinders with only limited patterning. To overcome this challenge, we are utilizing a combination of lithography and self-assembly to enable precisely patterned 3D nanoparticles and nanostructures composed of metals, dielectrics (including polymers), and semiconductors to be created. Because of the high surface area to volume ratio in 3D particles, we anticipate novel properties for solar, plasmonic, and drug delivery devices.

Approach: We are utilizing a self-folding approach, developed in our laboratory at the Johns Hopkins University, which enables precisely patterned 2D lithographic templates to be curved or folded into the third dimension. The structures curve or fold due to intrinsic or extrinsic stress. Specifically, we have been building on initial published work achieved using electron beam lithography to nanoimprinted samples thereby enabling cost-effective mass fabrication of these structures.

Results and Discussion: To date, we have been able to self-assemble 100 nm scale polyhedral particles with precise patterns in all three dimensions. We have also been able to create curved nanostructures with radii as small as 20 nm. These structures have been fabricated with precise patterns with a resolution of 15 nm, in all three dimensions, which is unprecedented. However, in our prior work we utilized electron beam lithography which limited us to the creation of small numbers of particles. In order to enable such an approach to be widely used, there is a need to enable mass production. Using the Nanonex imprinter in the NanoFab, we have been trying to enable such production of these nanostructures. We have been able to create molds of 2D templates and self-assemble 3D structures. One challenge that we seek to overcome is the ability to enable multilayer registration using nanoimprint lithography, which is challenging at the present time. However, we have succeeded in creating nanoimprinted lines and discs and are exploring the self-assembly of these structures. The advantage of nanoimprint lithography is really in throughput; i.e. we have shown that nanostructures can rapidly be fabricated over areas as large as 1 cm. Such parallel fabrication is not possible (cost-prohibitive) using electron beam lithography.

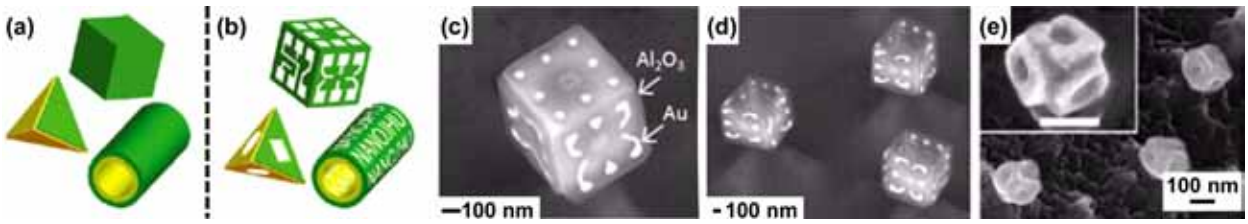
Recent Publications:

Self-assembly of lithographically patterned nanoparticles, J. H. Cho and D. H. Gracias, *Nano Letters* **9**, 4049-4052 (2009).

Curving nanostructures using extrinsic stress, J.H. Cho, T. James, and D. H. Gracias, *Advanced Materials* **22**, 2320-2324 (2010).

Three dimensional nanofabrication using surface forces, J.H. Cho, A. Azam, and D. H. Gracias, *Langmuir* **26**, 16534-16539 (2010).

Plastic deformation drives wrinkling, saddling, and wedging of annular bilayer nanostructures, J.-H. Cho, D. Datta, S.-Y. Park, V. B. Shenoy, and D. H. Gracias, *Nano Letters* **10**, 5098-5102 (2010).



(a) Present day nanoparticles have limited patterns (b) Our proposed nanoparticles and (c-e) fabricated nanoparticles, including precisely patterned polyhedral nanostructures.

Line-Edge Roughness Measurements in Diblock Copolymers

Research Participants: G. E. Stein,[1,2] A. L. Aquila,[3] E. M. Gullikson,[4] and J. A. Liddle[1]

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Project Goal: To develop a technique for measuring the intrinsic line-edge roughness in diblock copolymer systems targeted for integrated circuit manufacture.

Key Accomplishments:

- Developed precise and robust sample fabrication methods to enable transmission X-ray scattering measurements of polymer thin films.
- Developed resonant soft X-ray scattering measurement and data analysis techniques to determine line-edge roughness in block copolymers and latent-image photoresist samples.

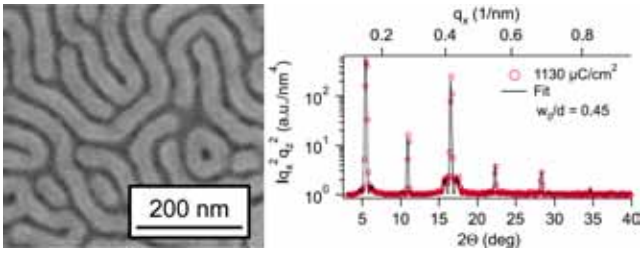
Background: Diblock copolymers are materials in which two immiscible polymer chains are covalently linked, leading to the formation of self-assembled phase-separated structures in which the domain size is controlled primarily by the size of the two component polymers. These materials have rich phase diagrams and can be used to generate nanoscale repeating structures with precisely defined domain sizes and spacings. In principle, because the system's morphology is determined by its thermodynamics, guided self-assembly of diblocks on lithographically patterned surfaces may eliminate the statistical effects that plague conventional patterning methods. These materials may be important for fabricating integrated circuits and future generations of patterned magnetic media for data storage. In both cases, the variability in nanoscale features can lead to an unacceptable inconsistency in individual bit or transistor performance. In integrated circuit manufacturing, it is essential to determine which process steps significantly add to the line-edge roughness (LER) of the completed devices in order to develop strategies to mitigate them to satisfy current and future device requirements. Measuring the intrinsic LER in a diblock, and the LER after guided self-assembly, provides a way of screening candidate material systems in order to find those with the lowest LER. It also enables the material and process contributions to be separated.

Approach: Nanoscale polymer structures are notoriously difficult to measure using techniques such as scanning electron microscopy because the materials are frequently radiation sensitive, causing them to change and deform while being imaged. Other metrology methods, including atomic force microscopy, only probe the upper layer of the material. To avoid these problems, we instead used X-ray scattering over a large area of material with relatively low fluence to minimize the radiation damage. X-rays also penetrate the material, sampling the structure through its entire thickness.

Diblocks as used for these kinds of patterning are typically thin films, roughly a domain-spacing thick. With low atomic number elements and film thicknesses less than 50 nm, the two components in diblocks typically have low X-ray absorption cross-sections. However, the level of X-ray scattering from the film and the contrast between the two phases can be dramatically improved using resonant scattering. This method involves using low-energy X-rays and tuning their energy precisely (within 0.1 eV) to match a particular resonance in the molecular structure of one of the diblock components; for example a C=O bond. In addition, in order to eliminate background scattering from the substrate, the diblock films are created on SiN membranes only 100 nm thick fabricated in the CNST NanoFab.

Results and Discussion: Alignment of the diblock to an underlying lithographic pattern results in the formation of a diffraction pattern whose major features are determined by the periodicity of the lithographic pattern, but whose details are controlled by the diblock's morphology. The net outcome is extremely high-quality X-ray diffraction data, with good contrast and low noise out to large scattering angles. This data quality has enabled sophisticated fitting algorithms to be used with confidence to extract parameters including LER and the interfacial width between the two phases. Because the X-rays sample the full film thickness, the technique can also determine the sidewall angle between the two phases— something that was previously only modeled.

Future directions in this area will include the use of this technique to measure the extent of the deprotected, latent image in chemically amplified resists, a measurement of significant interest to the semiconductor industry. In addition, we plan to explore the potential of guided self-assembly to produce large areas of orientated nanostructures for enhanced X-ray and neutron scattering experiments.



The electron microscopy image shows a *randomly* oriented, 50:50 lamellar poly(styrene-b-methymethacrylate) diblock. (Note that the image appears to show that the relative proportions of the two phases are not 50:50.) The X-ray diffraction pattern obtained from an *aligned* sample of the same material exhibits diffraction peaks out to 6th order and extremely low background scattering.

Acknowledgments: Soft X-ray scattering measurements were performed at the Advanced Light Source at Lawrence Berkeley National Laboratory.

Recent Publications:

Measuring the structure of epitaxially assembled block copolymer domains with soft X-ray diffraction, G. E. Stein, J. A. Liddle, A. L. Aquila, and E. M. Gullikson, *Macromolecules* **43**, 433-441 (2010).

The Effect of Resist on the Transfer of Line-Edge Roughness Spatial Metrics from Mask to Wafer

Research Participants: G. M. Gallatin[1] and P. P. Naulleau[2]

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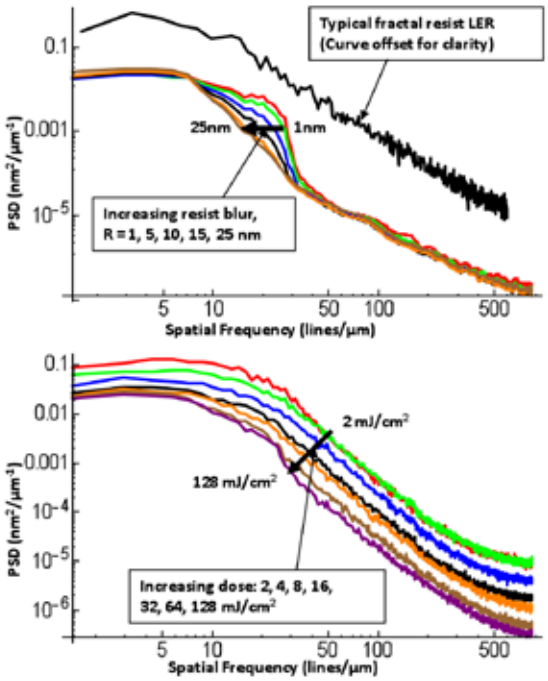
Project Goal: To develop an algorithm and associated computer code for predicting the extent to which both surface and edge roughness on an extreme-ultraviolet (EUV) mask is transferred to line edge roughness (LER) in the exposed and developed photoresist patterns on a wafer. The computational tools will need to account for shot noise effects in the exposure process, as well as diffusion and acid-base neutralization in the photoresist.

Key Accomplishments:

- Early aerial-image modeling results have demonstrated the importance of mask-induced LER, and have informed the development of methods for experimentally determining the presence of such effects.
- Exposure statistics have been added to the model, and have shown that these proposed experimental methods are viable only in cases where the contribution to total wafer LER due to acid shot noise is negligible compared to that due to mask induced LER.
- The model has predicted that in certain cases, even with the mask LER being the dominant contributor, the wafer LER remains indistinguishable from that produced by exposure statistics alone.

Background: Many next generation computer chips, with features on the scale of 22 nm and below, will be fabricated using extreme ultra-violet (EUV) lithography. The tolerances for feature size, shape, placement, etc., for these devices range from a maximum of a few nanometers down to less than 1 nm. One critical parameter, the LER, is required by the International Technology Roadmap for Semiconductors to be less than 2 nm. Unfortunately, LER values are still several nanometers, with the best case a little over 2 nm, for processes that meet all other requirements. To reduce this value to below 2 nm, its sources must be determined. Mask features themselves have an LER of about 10 nm which, in a 5x reduction EUV imaging system, is reduced to 2 nm on the wafer. Additionally, modern photoresists are "chemically amplified;" i.e., the absorption of a photon releases an acid whose concentration is proportional to the image intensity. The photoresist then undergoes a post exposure bake process during which the acid diffuses and "deprotects" the resist by converting insoluble molecules to soluble ones. The photoresist is developed by washing away the soluble regions, leaving the insoluble regions as the pattern. Base quencher, which neutralizes acid, is often added to the photoresist to limit acid diffusion, which degrades the resist resolution, and to control exposure dose levels.



The lower cluster of curves in the top figure shows the power spectrum density (PSD) of the wafer LER without exposure statistics (i.e., no acid shot noise). The frequency contribution from the mask LER is clearly visible as the bump in the PSD at frequencies of about 20 lines/μm. But, the bump disappears as the acid diffusion range, R, increases. The upper gray curve is the standard shape produced by acid shot noise alone. The lower figure shows computed wafer PSDs including both mask effects and acid shot noise for R = 10 nm, for various exposure doses. These curves are essentially indistinguishable from those created by acid shot noise alone.

Approach: Our model predicts the variation in the statistics of the wafer LER as the relative contributions from the mask LER and the statistics from the exposure and processing of the photoresist are varied. Following the tenets of quantum mechanics, the image intensity acts mathematically as a probability distribution for the positions where photons will be absorbed and an acid molecule will be released. These exposure statistics are often termed "acid shot noise" since the number of acid molecules generated in any small volume obeys Poisson statistics. Coupled partial differential equations describe the reaction and diffusion of the acid, along with the deprotection of the photoresist. These equations are solved by a combination of analytical manipulation and numerical evaluation. The final step, photoresist development, is highly nonlinear and difficult to model. However, most modern photoresists respond primarily in a binary fashion. Regions where

the deprotection density is above a given value wash away and regions below that value do not. The LER can therefore be determined by simply thresholding the deprotection density.

Results and Discussion: In the absence of acid shot noise, the contribution of mask LER is clearly detectable as a bump in the measured power spectral density (PSD) of the wafer LER. However, when acid shot noise is included, the bump can vanish, indicating that although mask-induced LER is still dominant, in this case it would be impossible to determine this from just the wafer LER PSD since it is virtually indistinguishable from the PSD produced by acid shot noise alone. Therefore, the PSD of the wafer LER cannot be used reliably to determine the contribution to the wafer LER coming from roughness on the mask.

Recent Publications:

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

Measurement Platforms to Facilitate Nanoimprint Lithography

Research Participants: H. Ro,[1,] H. J. Lee,[1] L. Chen,[2] K. Kearns,[1] T. Germer,[3] W.L. Johnson,[1] Y. Ding,[4] K. Alvine,[5] and C.L. Soles[1]

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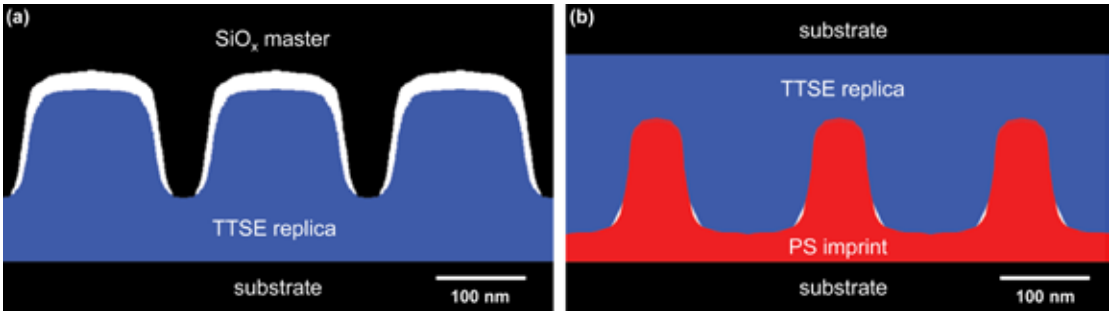
Project Goal: To develop, advance, and demonstrate measurements that facilitate nanoimprint lithography (NIL) as a viable technology for the patterning of robust, reliable, and functional nanostructures with dimensions smaller than 25 nm. These measurements will be critical as NIL transitions from a novel laboratory patterning technique to a commercial one for producing nanostructures for applications as diverse as semiconductors, bit-patterned magnetic media, high brightness LEDs, biotechnology, and other emerging technology sectors.

Key Accomplishments:

- Combined critical dimension small angle X-ray scattering (CD-SAXS) shape measurements with a finite-element inversion analysis of the acoustic modes from Brillouin light scattering to identify finite-size effect reductions of the elastic constants of polymeric nanolines fabricated by NIL.
- Established a structure-property-processing understanding of how the rheology of polymer flow in the nanoscale cavities of a NIL mold lead to residual stresses and physical instabilities in NIL generated patterns.
- Used X-ray based pattern shape measurements to quantify the patterning fidelity of cubic silsequioxanes (SSQs) as NIL template replication materials; validated their subsequent performance as high performance, low-cost secondary imprint templates.

Background: Nanoimprint Lithography (NIL) was originally developed as a versatile, low-cost, and high-resolution patterning alternative for optical lithography in CMOS fabrication. However, it is becoming apparent that NIL has great potential for nanotechnology in general. It is capable of patterning sub-10 nm features directly into a range of materials, even functional materials, and not just sacrificial resist formulations. Intense research and development activities are currently centered on not just CMOS logic devices, but also bit patterned data storage media, high brightness light-emitting diodes (LEDs), patterned biological devices, optical devices, and a range of emerging technologies.

Approach: Our approach is to develop measurement platforms to quantify the quality of the pattern or feature that has been fabricated by NIL. These quantitative assessments of the imprint process are necessary inputs to optimize the development of NIL and move from novel demonstrations or devices in the lab to the high volume nanomanufacturing required for commercialization. To accomplish this goal we leverage our expertise in accurately measuring physical shape and properties of nanoscale structures. For the shape measurements we utilize our critical dimension X-ray scattering (CD-SAXS) and X-ray reflectivity methods to non-destructively determine the 3D shape of both the mold cavities and the imprinted structures with nanometer-scale resolution. This approach provides us with a quantitative assessment of the fidelity of pattern transfer. Then we quantify the way that the NIL process itself influences properties of the imprinted material and determine if this influence has an impact on the performance, robustness, and



(a) Primary SiOx imprint into TTSE. (b) Secondary Imprint using TTSE replica.

quality of the imprinted device. By providing the measurement portion of the processing–properties–structure paradigm, we facilitate the development of technologies based on NIL patterning.

Results and Discussion: A central theme in our research is the development and use of X-ray methods, including CD-SAXS and specular X-ray reflectivity, to quantify the physical shape or profile of nanoscale structures with sub-nm resolution. These methods are non-destructive and applicable to nanostructures in a wide range of materials, allowing us to quantify the pattern shape in both the mold and the imprint, and thereby establish the fidelity of the pattern transfer process without sacrificing the molds or patterns. This approach is a powerful tool for improving and evaluating NIL processes.

Recently, we used these non-destructive and quantitative pattern shape measurement to address several materials issues related to NIL. One notable example, briefly summarized here, uses the quantitative fidelity of pattern transfer concept to determine the feasibility of using solution processable sol-gel cubic SSQs as template replication materials for generating low-cost secondary imprint molds. A SiO_x imprint master was fabricated by conventional 193 nm optical lithography and imprinted into the spin-on SSQ material (referred to as TTSE) film at low temperatures. The as-imprinted TTSE patterns were then heated to convert the TTSE precursors into a hard cross-linked organosilicate. This material showed excellent thermal stability, a high modulus, a low surface energy, and quartz-like transparency in the ultraviolet (UV) spectrum. The figure summarizes the different cross-sectional line-space patterns obtained by fitting the X-ray data. Part (a) reveals a small amount of shrinkage in the TTSE replica that occurs after imprinting, during the high temperature vitrification. However, the TTSE replica can be used to directly imprint polystyrene (PS) patterns, shown in part (b), with an extremely high fidelity of pattern transfer.

Recent Publications:

Elastic constants and dimensions of imprinted polymeric nanolines determined from Brillouin light scattering, W. L. Johnson, S. A. Kim, R. Geiss, C. M. Flannery, C. L. Soles, C. Wang, C. M. Stafford, W. Wu, J. M. Torres, B. D. Vogt, and P. R. Heyliger, *Nanotechnology* **21**, 075703 (2010).

Stability and surface topography evolution in nanoimprinted polymer patterns under a thermal gradient, Y. Ding, H. J. Qi, K. J. Alvine, H. W. Ro, D. U. Ahn, S. Lin-Gibson, J. F. Douglas, and C. L. Soles, *Macromolecules* **43**, 8191-8201 (2010).

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* **23**, 414-420 (2010).

Novel Sources for Focused Ion Beams

Research Participants: A. V. Steele,[1] B. Knuffman,[1] J. Orloff,[2] and J. J. McClelland[1]

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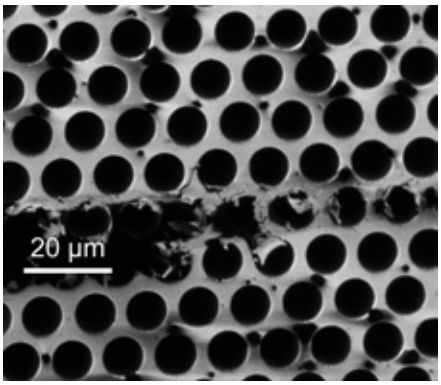
Project Goal: To explore applications of the CNST-developed magneto-optical trap ion source that make use of its unique brightness and range of ionic species to realize new measurement and fabrication methods, enabling new nanotechnology tools for future electronics, nanofabrication, and energy.

Key Accomplishments:

- Designed and constructed a laser-cooled, magneto-optical trap-based chromium ion source and demonstrated first-ever images with a focused Cr ion beam.
- Designed and constructed a laser-cooled, magneto-optical trap-based lithium ion source mounted on a commercial focused ion beam column, creating the first Li ion microscope.



MOTIS image of a damaged multichannel plate acquired using a 2 kV, 10 pA beam of Li ions.

Background: Focused ion beams (FIBs) are a proven tool for nanoscale surface modification and interrogation, having been used for over two decades for material removal (milling), material deposition, microscopy, and ion implantation. Collectively, commercial tools that use focused ion beams make up a $300 million to $600 million industry, with customers ranging from circuit manufacturers doing fabrication-line diagnostics and circuit edits, to basic researchers making new discoveries in nanoscale material development. However, FIBs are generally limited because they lack source flexibility. The commonly used liquid-metal ion source (LMIS) uses ions from the metal gallium (Ga), which is useful for milling because of its relatively large mass. It has also seen wide application in material deposition by induced beam chemistry using precursor gases. Unfortunately, gallium can contaminate nanostructures by implanting, and is destructive in microscopy because it erodes the sample. Expanding the available ion species beyond gallium would create a suite of focused ion beam tools with a range of new nanofabrication and imaging possibilities, including high-resolution microscopy, new beam chemistry processes for material removal and deposition, and entirely new applications in nanoscale analysis and fabrication. Focused to the nanometer scale, light ions could image without 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 perform localized chemistry, deposit high purity nanoscale conductors, or implant dopants.

Approach: For an ion source to be useful for nanoscale probe formation, it is essential for it to have a very small emittance, a high brightness, and a narrow energy spread. The magneto-optical trap ion source, or MOTIS, developed in the CNST, achieves these characteristics in an approach very different from the LMIS. Neutral atoms from an atomic beam are first slowed and then trapped in a magneto-optical trap, where they are laser-cooled to temperatures as low as 100 μK. A laser then ionizes the atoms in the presence of an extraction field, which accelerates them into a beam. The extremely low temperature the ions have when they are created translates into a very small spread in transverse velocities. This results in a very small emittance, which combined with an achievable current of as much as 100 pA makes for a very high brightness. The energy spread, fundamentally limited only by the cold atom temperature, but practically limited by source geometry, is also very low compared with the LMIS.

Having first demonstrated this source in 2008, we are currently undertaking two closely related projects aimed at characterizing its performance and exploring applications. In one project, a chromium MOTIS has been built, complete with a simple focusing and deflection system and a channel electron multiplier to detect secondary electrons. In the second project, we have constructed a lithium MOTIS that is completely compatible with a FIB system supplied by FEI, Co., our partner under a cooperative research agreement. This FIB system provides a high-quality focusing column, including beam alignment, stigmation and scanning, as well as an easy-to-use specimen chamber for rapid sample interchange.

Results and Discussion: The focused chromium ion system has recently come on line, allowing us to generate images and to study probe size as a function of source operating parameters. We have created ion beams with energies ranging from 0.5 keV to 3 keV, and currents of a few tenths of a picoampere. We observed probe sizes as small as 205 ± 10 nm (one-standard deviation radius), a spot size that is probably limited by the precision of our rudimentary ion optical system. The Li ion system has also become operational in recent months, and has generated the first high-quality images with resolution in the sub-100 nm range using 2 keV Li ions. Detailed studies of the source behavior are currently underway.

With the introduction of the MOTIS as a viable source for nanoscale focused ion beams, we are now poised to begin a wide range of research paths. In the near future, we will explore higher acceleration energies (up to 30 keV), novel beam chemistries with Li ions, and direct deposition of metal with Cr ions. Beyond this, by exploiting the unique ability of the MOTIS to produce single ions "on demand" for doping materials, we see a large number of possibilities with new ionic species such as Cs, noble gases, or rare earths.

Recent Publications:

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).

Magneto-optical-trap-based, high brightness ion source for use as a nanoscale probe, J. L. Hanssen, S. B. Hill, J. Orloff and J. J. McClelland, *Nano Letters* **8**, 2844-2850 (2008).

In Situ Measurements of Thermodynamics and Reaction Kinetics During Nanomaterials Synthesis and Catalysis

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Project Goal: To measure how growth and pressure affect the thermodynamics, reaction rate, structure, and morphology of nanostructured zero- and one-dimensional materials.

Key Accomplishment:

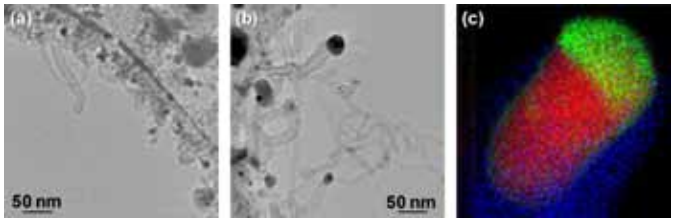
- Catalytic activity of Ni catalyst for carbon nanotubes formation was improved by doping with 10 % to 20 % mole fraction of Au.

Background: Nanomaterials range from catalyst nanoparticles, where the surface area and atomic structure of the exposed surfaces control their activity and reactivity, to magnetic nanoparticles, where the properties are a function of their composition, size, and assembly. Recent advances in nanotechnology require very stringent control on the synthesis of nanomaterials so that they can be directly incorporated in the fabrication process. There is a growing need for developing and characterizing active nanostructures where the system components may be functionalized to interact in a controlled manner with the ambient environment (pressure, fields, stress, heat, *etc.*). To achieve this goal, it is essential to develop atomic-scale measurements of the

interactions with the ambient during controlled synthesis. Evolution of the morphology and the associated chemical and physical properties takes place both during the nanomaterial synthesis process and in response to external stimuli from the ambient; therefore, measurement of these complex transformations requires the use of advanced *in situ* methodologies so that the associated dynamic processes can be identified, understood, and measured.

Approach: We use a modified TEM to elucidate the nucleation and growth mechanism as well as the reaction kinetics. Most of the experiments to date have been performed using environmental scanning TEM (ESTEM) at either the Technical Institute of Denmark (DTU) or Arizona State University. Thin Ni/Au films (1 nm to 2 nm) with varying mole fractions were deposited using physical vapor deposition under high vacuum conditions. Samples were heated under the reactive gas environment and the dynamic changes recorded using electron diffraction, high resolution electron microscopy images (including videos), and electron energy loss spectra. This approach allows us to characterize the materials during synthesis. Post-synthesis analysis was also performed using energy-dispersive X-ray analysis at NIST.

Results and Discussion: For carbon nanotube growth, we have focused on the effect of Au doping in Ni on the activity of catalyst particles for the formation of carbon tubular structures. We find that the number of catalytically active particles for carbon nanotubes increased by a considerable amount for Ni thin films containing small amounts of Au (less than 20 % mole fraction). With higher Au content, the activity began to decrease, and pure Au samples were observed to be inactive for nanotube formation.



Low magnification images of the samples with (a) 100 % Ni, and (b) 80 % mole fraction Ni and 20 % mole fraction Au recorded after the thin films were exposed to approximately 0.3 Pa of C_2H_2 at approximately 510 °C. (c) High magnification STEM-EDS elemental map of a single catalyst particle showing high concentration of Au (green) at the tip and Ni (red) in the center.

Acknowledgments: Financial support from NSF-CBET (#0625340) and use of facilities in the LeRoy Eyring Center for Solid State Science at Arizona State University are gratefully acknowledged.

Recent Publications:

A spray drying system for synthesis of rare-earth doped cerium oxide nanoparticles, V. Sharma, K. M. Eberhardt, R. Sharma, J. B. Adams, and P. A. Crozier, *Chemical Physics Letters* **495**, 280-286 (2010).

In situ evaluation of the role of Au in improving catalytic activity of Ni nanoparticles for the formation of 1-D carbon nanostructures, R. Sharma, S. W. Chee, P. Rez, R. Miranda, and A. Herzing, *Nano Letters, submitted.*

Particle-Tracking Measurements of Nanoparticle Dynamics in Fluids

Research Participants: P. Carmichael,[1] K. Du,[1,2] J. Juarez,[3] M. D. McMahon,[1] P. Mathai,[1,2] M. Bevan,[3] B. Shapiro,[4] E. Waks,[5] G. Gallatin,[1] J. J. McClelland,[1] A. J. Berglund,[1] and J. A. Liddle[1]

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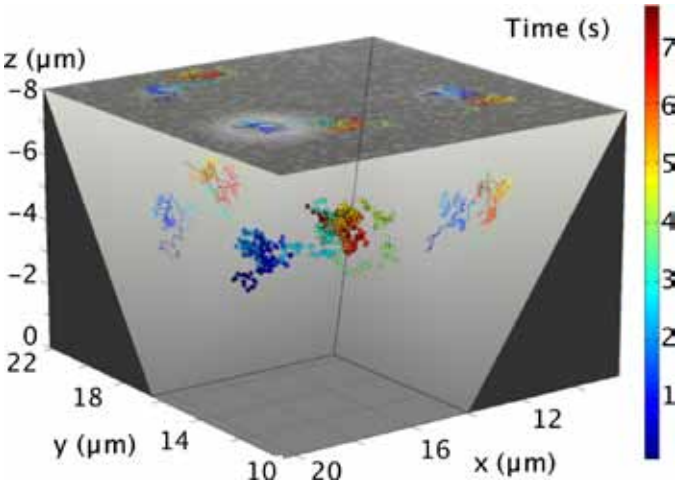
Project Goal: To develop faster, more reliable, and more sensitive methods for measuring, understanding, and controlling the forces and interactions experienced by nanoparticles in a fluidic environment.

Key Accomplishments:

- Developed "Orthogonal Tracking Microscopy" method that uses an optical microscope to repeatably resolve nanoparticle positions with 20 nm resolution in three dimensions.
- Developed a suite of statistical analysis tools for fast, robust, and accurate single particle tracking measurements.

Background: A sizable library of nanoparticles has been developed over the last few decades, having novel thermal, optical, mechanical, and chemical properties relative to their macroscopic counterparts. Assemblies of these fluid-based components promise disruptive improvements in several technological areas, including optics, electronics, catalysis and medicine, provided they can be organized into structures with nanometer precision on an industrial scale. This goal is hindered by the lack of measurement methods with suitable resolution (both in space and time) for monitoring assembly processes. By tracking and analyzing the motion of individual nanoparticles moving in solution and interacting with other particles and surfaces, we can determine the forces and interactions that govern the motion, internal dynamics, and fluidic self-assembly of nanoparticles, including large biomolecules, into organized structures.



Reconstructed trajectory of a 190 nm-diameter fluorescent particle undergoing Brownian motion near two micromirror faces. The 3D positions are determined with a precision of 20 nm, significantly below the optical diffraction limit, and at frame rates greater than 300 Hz.

Approach: We use single-particle tracking methods to observe nanoparticle dynamics using an optical microscope and a sensitive CCD camera. The resulting data are analyzed to determine a nanoparticle's position in each camera frame and to link these positions together into a trajectory containing information about a particle's size, shape, and local environment. These methods are used widely to monitor transport properties in cell-scale biological processes and to determine rheological properties of complex fluids. We are working to extend and improve these techniques through experimental design, device fabrication, and theoretical analysis with the goal of rendering nanoparticle tracking a quantitatively reliable measurement method for resolving the nanometer-scale dynamics in engineered nanoparticle systems.

Results and Discussion: We have successfully addressed several outstanding measurement problems, developing techniques for resolving three-dimensional particle trajectories, actuating and controlling particle motion based on real-time data processing, and analyzing data quantitatively with statistically optimal methods.

The interaction of a single nanoparticle with a patterned substrate is inherently a three-dimensional problem, since motion both within and normal to the substrate plane affects the dynamics. In order to monitor the full three-dimensional trajectory of a nanoparticle near a surface, we developed the "Orthogonal Tracking Microscopy" method in which a particle is observed in the vicinity of a pyramidal micromirror etched in a silicon substrate. The micromirrors, fabricated in the CNST NanoFab, provide a direct and reflected image in the microscope, which can be reconstructed to provide position information in three dimensions. We fabricated devices and performed an extensive analysis of systematic and random errors, demonstrating device performance with repeatable sub-20 nm, three-dimensional particle localization capability.

We also developed a suite of methods for extracting information such as particle position, orientation, and diffusion coefficient from the tracking data. We developed an optimal diffusion coefficient estimator, updating a decades-old theory to account for important technical considerations in modern experiments. The new method has been applied to analyze the nanometer-scale motion of quantum dots in an electrofluidic control setting.

Future directions in this area will include using real-time feedback control to track a particle's position and simultaneously perform high-resolution spectroscopy, increasing spatial and temporal resolution for *in situ* measurements.

Recent Publications:

Statistics of camera-based single particle tracking, A. J. Berglund, *Physical Review E* **82**, 011917 (2010).

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).

3D particle trajectories observed by orthogonal tracking microscopy, M. D. McMahon, A. J. Berglund, P. T. Carmichael, J. J. McClelland, and J. A. Liddle, *ACS Nano* **3**, 609-614 (2009).

Atomic-Scale Silicon Surface Control to Enable Atom-Based Dimensional Metrology

Research Participants: R. M. Silver,[1] P. Namboodiri,[1,2] K. Li,[1,3] S. Chikkamaranahalli,[1,2] and J. Fu[1]

[1]Physical Measurement Laboratory, National Institute of Standards and Technology, Gaithersburg, MD 20899
[2]KT Consulting NV, Antioch, CA 94509
[3]Department of Physics, University of Maryland, College Park, MD 20740

Project Goal: Develop a process to fabricate atomic-scale structures that can be accessed externally using robust fiducial marks, and demonstrate atomic-scale control of the surface morphology with large step-free atomically-flat regions on silicon for dimensional metrology.

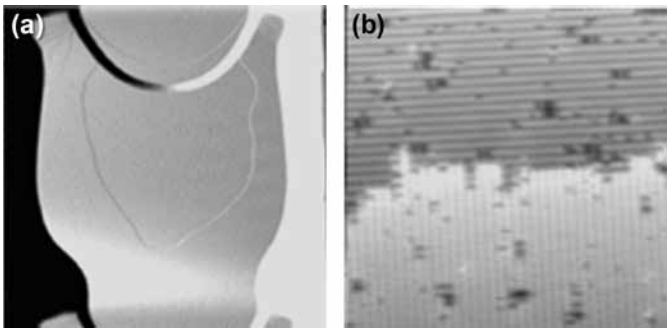
Key Accomplishments:

- Achieved atomic-scale control of step flow patterns on the wafer scale.
- Produced atomically flat and extremely wide terraces (larger than 10 μm).
- Patterned silicon at the atomic scale with robust fiducials.

Background: An essential element of this project is the fabrication of test artifacts and structures for the development of high resolution calibration artifacts and methods. Atomic-scale fabrication and manipulation typically requires well ordered surfaces with control of the step-terrace morphology at the atomic scale. Moreover, in this work these samples need to be dimensionally stable and allow transfer to other measurement

tools that can measure the artifacts with dimensions known on the nanometer scale. Control of atomic-scale morphology of the substrates in the presence of fiducial marks that can withstand high temperature processing has been a long standing challenge. The ability to produce repeatable, large terrace patterns over an entire wafer in the presence of fiducials, while maintaining the ability to control the terrace width, would represent an exciting new advance to meet the needs of atomically precise manufacturing. Creating a process that leads to the spontaneous arrangement of atomic terraces and surface structure is a key objective of this project.

Native surfaces are not atomically flat and contain a variety of atomic steps. Even with the lowest miscut angle available, surface dynamics preclude atomically flat terraces stretching over a micrometer. However, it is known that the surface morphology can be altered with thermal processing procedures giving mobility to the surface atoms. The atoms have different dynamics depending on local bonding forces and on the process temperatures. A key achievement this year is the use of fiducial structures etched in the surface, which after thermal processing serve as boundaries for the evolution of step flow by confining the dynamics into individual cells.



(a) AFM image (25 μm × 25 μm) of two neighboring 25 μm cells prepared on a Si(100) sample after annealing at 1030 °C for 48 hr. The surface has only one atomic step, and terraces more than 10 μm wide. (The step flow patterns are nearly identical in every cell defined by the fiducial marks.) (b) STM image (20 nm × 20 nm) showing atomically-ordered, reconstructed surface.

Approach: We have fabricated micrometer sized fiducial patterns of 25 μm and 50 μm square in two orientations on a Si(100) sample using standard lithographic procedures and dry (plasma) etching in the CNST NanoFab. The patterns are formed by creating trenches or walls (2 μm wide). The samples were resistively heated by passing current and flash heating at 1230 °C for a few seconds, and then annealed at 1030 °C for 48 hours. When heated, the micrometer scale patterns induce different mass transport kinetics due to the thermodynamics and electromigration effects.

The morphology of the step-terrace patterns can be controlled by optimizing the size and orientation of the fiducial patterns on the surface. The patterns provide boundary conditions and change the dynamics of the atomic processes by pinning the steps during high temperature processing. Control is achieved by optimizing the size, shape, and orientation of the patterns to obtain uniformly sized and spaced atomic steps on the surface.

Results and Discussion: We have been able to obtain nearly identical step-terrace patterns in all the individual cells of the patterned sample. The atoms flow in the direction of the current [bottom-up in figure (a)], causing the steps to move in the opposite direction. Steps are accumulated at the boundaries defined by the fiducial patterns. As seen in the figure, a 25 μm cell has only one atomic step. The terrace width is larger than 10 μm, which is by far the largest terrace width reported. The step flow pattern is largely associated with the orientation of the micro-scale features. The patterns confine the steps to smaller regions, which help to remove the random fluctuations (usually associated with very long steps) and facilitate the formation of regular step arrays.

As shown in the figure, this method produces atomically ordered surfaces that are ideal for atom based metrology applications. Diffusion on a Si(100) surface is anisotropic because of the 1 × 2 and 2 × 1 surface reconstruction. When a sample is heated by passing a direct current through it, the electromigration of Si atoms occurs. The electromigration introduces a bias in the diffusion so that the steps are swept aside gradually, leaving behind very large atomically flat terraces. The bias in the diffusion also drives the surface to a single domain, either 1 × 2 or 2 × 1 depending on the direction of the current.

This method offers vast potential for 'engineering surfaces at the atomic scale' and for enabling their use as templates for various applications related to atomic-scale fabrication. It will be also possible to fabricate step-bunch heights by controlling the pitch (depth of the etched trenches). Step bunches of various heights (typically 5 nm to 20 nm) could be produced in a single sample and be used as height standards. These structures are robust and can be persevered in a moderately controlled environment.

Nanoscale Linewidth Standards for AFM Calibration

Research Participants: C. McGray,[1] R. Kasica,[2] G. Orji,[1] R. Dixson,[1] M. Cresswell,[1] R. Allen,[1] and J. Geist[1]
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[2]*Center for Nanoscale Science and Technology, National Institute of Standards and Technology, Gaithersburg, MD 20899*

Project Goal: To refine AFM tip width calibration techniques and improve the traceability of the meter for nanoscale measurements by developing linewidth standard materials with near-atomically-smooth surfaces.

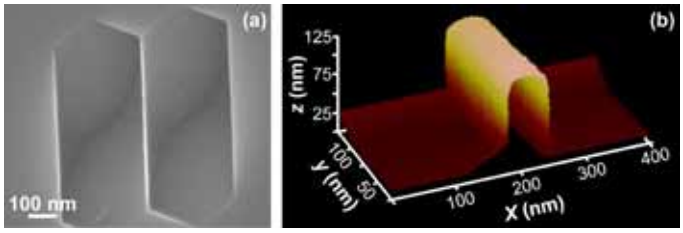
Key Accomplishments:

- Achieved sidewall roughness as low as 0.6 nm (two atoms).
- Fabricated full range of widths from 200 nm down to 5 nm for linearity assessment.

Background: Accurate dimensional metrology is of utmost importance to the continued development of improved

technologies in the semiconductor electronics industry, as device dimensions scale from 32 nm down to 22 nm and beyond. NIST helps to ensure measurement accuracy by providing linewidth standard reference materials that allow traceability of the meter down to these very small scales. Currently, standard uncertainties in the dimensions of these materials are approximately 1 nm. As critical dimensions in the semiconductor industry become smaller, this uncertainty becomes a larger percentage of the measurement. A new generation of linewidth standards is in development to provide uncertainties even smaller than 1 nm.

Approach: The largest component of the measurement uncertainty in the current generation of linewidth standards is caused by non-uniformity in the width of the line along its length. We are therefore targeting uniformity improvements as the most important requirement of the next generation of linewidth standards under development. Since even the highest-resolution electron beam lithography systems do not yield the required uniformity, we instead use electron beam lithography in the CNST NanoFab only as a first step in the production of the linewidth standards. The lithographically-defined pattern is then transferred into a single crystal of silicon through the use of an etchant that selectively removes material to expose certain crystallographic planes which become effectively polished.



(a) SEM image of a next-generation linewidth standard structure. The line in the center of the image is defined by the two adjacent crystallographically-etched hexagonal pits. (b) 3D topography of a linewidth standard structure as measured by critical-dimension atomic force microscopy.

Results and Discussion: To ensure that the crystallographic etchant polishes the linewidth standards without dissolving them away, the linewidth standard structures must be very well-aligned to the facets of the crystal that are unaffected by the etchant. We have developed an auto-alignment method for accomplishing this requirement even when the orientation of the facets is not precisely known. Using this auto-alignment method, we have been able to produce linewidth standard structures having non-uniformity as low as 0.6 nm.

The new generation of linewidth standards is being fabricated with integrated navigation features that will help AFM measurement scientists to precisely position their measurements. These features are expected to further reduce the uncertainty of the measurement by providing greater repeatability. Moreover, the new reference materials, shown in the figure, can now be fabricated with a range of widths extending all the way down to 5 nm. Having a range of widths in the standard will help users of AFMs calibrate the linearity of their instruments in the size ranges required by the semiconductor devices of today and tomorrow.

Recent Publications:

Comparison of measurement techniques for linewidth metrology on advanced photomasks, S. Smith, A. Tsiamis, M. McCallum, A. Hourd, J. T. M. Stevenson, A. J. Walton, R. G. Dixson, R. A. Allen, J. E. Potzick, M. W. Cresswell, and N. G. Orji, *IEEE Transactions On Semiconductor Manufacturing* **22**, 72-79 (2009).

RM 8111: Development of a prototype linewidth standard, M. Cresswell, W. F. Guthrie, R. G. Dixson, R. A. Allen, C. E. Murabito, and J. V. Martinez De Pinillos, *Journal of Research of the National Institute of Standards and Technology* **111**, 187-203 (2006).

Developing Nanocalorimetry Techniques and Material Standards for Measuring Properties of Exothermic Reactions in Multilayer Films

Research Participants: P. Swaminathan,[1,2] D. A. LaVan,[2] and T. P. Weihs[1]

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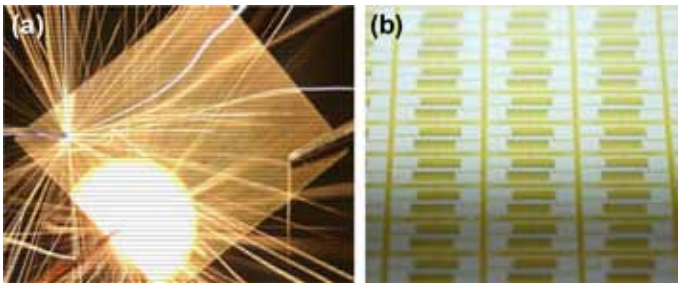
[2]*Material Measurement Laboratory, National Institute of Standards and Technology, Gaithersburg, MD 20899*

Project Goal: To develop nanocalorimetry techniques for studying exothermic reactions in metallic multilayer films and use these as standards for improved nanocalorimeter measurements.

Key Accomplishments:

- Designed a high temperature, fast optical calibration procedure for nanocalorimeters.
- Characterized rapid melting and solidification events in thin metal films and multilayers.

Background: Reactive multilayer materials consist of alternating nanoscale layers of metals and/or alloys that react exothermically, forming stable intermetallics. These exothermic reactions can be exploited to cleanly bond dissimilar metals in sputter targets, and for joining temperature sensitive assemblies such as attaching silicon solar cells to heat sinks. Typical reaction fronts in these systems have velocities of the order of a few meters per second with heating rates as high as 10^6 °C/s and maximum temperatures of 1500 °C (1.5 ms from room temperature to 1500 °C). A wide variety of multilayer systems are being studied; because of the extreme temperatures and rates involved in these reactions, measuring their thermophysical properties presents unique measurement challenges, which are solved using a nanocalorimeter chip fabricated in the CNST NanoFab.



(a) Photograph showing a self propagation reaction in a free standing Ni-Al foil. The reaction is initiated in the lower left corner of the foil and propagates across the foil, with a velocity of a few meters per second. Maximum foil temperatures can reach 1500 °C. The final product is a NiAl intermetallic. (b) An array of nanocalorimeters fabricated in the NanoFab. Each calorimeter consists of a Pt strip on silicon nitride membrane suspended on Si, with electrical connections made to each strip for heating and measuring resistance.

Approach: Nanocalorimetry quantitatively measures thermal and thermophysical properties in materials at the nanoscale using a very sensitive sensor chip with a thermal addendum (the heat capacity of the instrument) comparable to the sample. A typical device consists of a 50 nm platinum strip that is thermally isolated on a silicon nitride membrane suspended on a silicon die. Electrical connections to the die allow for current to be applied to heat the platinum strip, and for voltage and current measurements. The fabrication steps involved are fully implemented at CNST based on a 100 mm wafer that yields approximately 70 nanocalorimeter chips. Individual chips are cleaved from the wafer and then calibrated in the NIST Material Measurement Laboratory. Calibration involves measuring the temperature coefficient of resistance of the platinum by local Joule heating of the strip while simultaneously measuring resistance and temperature. The sample of interest is then evaporated onto the calibrated Pt sensor in the NanoFab and its properties measured.

Results and Discussion: We have fully optimized the high temperature calibration procedure for the nanocalorimeters, looking at issues of stability and repeatability of temperature and resistance measurements. Optical calibration requires measuring the emissivity of the Pt film as a function of temperature; these measurements have been performed in collaboration with Dr. Hanssen and Dr. Wilthan in NIST's Physical Measurement Laboratory.

As a test of the calibration procedure, we have studied the melting and solidification of a 50 nm-thick aluminum sample at heating and cooling rates of 10^4 °C/s. At the rapid cooling rates achieved with a nanocalorimeter we can directly observe the effect of recalescence (temperature rise in a material due to release of its heat of fusion) in aluminum. Conventional scanning calorimeters operating at slower rates and with larger thermal addenda cannot capture this phenomenon.

Currently, we are measuring the thermophysical changes in Ni-Al bilayer systems with self heating rates around 10^6 °C/s, and plan to combine the nanocalorimeter measurements with post reaction and *in situ* microstructural measurements.

Acknowledgments: This project is supported in part by NIST Grant 70NANB9H9146.

Recent Publications:

Optical calibration for nanocalorimetry measurements, P. Swaminathan, B. G. Burke, A. E. Holness, B. Wilthan, L. Hanssen, T. P. Weihs, and D. A. LaVan, *submitted.*

Dynamics of solidification in Al thin films measured using a nanocalorimeter, P. Swaminathan, R. K. Kummamuru, D. A. LaVan, and T. P. Weihs, *submitted.*

Microfluidic Bumping Chip for Purification of Tumor Cells from Blood

Research Participants: J. B. Haun,[1] L. Chen,[2] M. Cangemi,[2] H. Lee,[1] and R. Weissleder[1]

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[2]*Center for Nanoscale Science and Technology, National Institute of Standards and Technology, Gaithersburg, MD 20899*

Project Goal: To develop a microfluidic device for rapid and selective concentration and separation of epithelial cancer cells from whole blood based on differences in size (12 μm to 20 μm in diameter for tumor cells, < 12 μm in diameter for blood cells). This device will facilitate detection and molecular profiling of circulating tumor cells obtained from "blood biopsies."

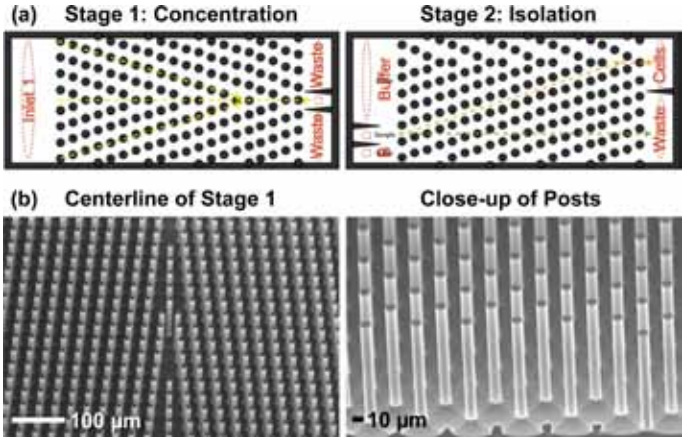
Key Accomplishments:

- Designed and fabricated a microfluidic device for concentration and separation of tumor cells from blood.
- Created micropost structures with extremely high aspect ratio (4x greater than previously demonstrated) with vertical sidewalls.

Background: The ability to detect and molecularly analyze scant and unique cell populations would have far reaching applications in medicine and the life sciences. It would allow rapid detection of rare cells in clinical specimen, enable molecular profiling, and open a new era of medical diagnostics where rational treatment decisions could be based on quantitative molecular phenotypes. Circulating tumor cells (CTCs) are rare cancer cells that have escaped from a primary tumor site and entered the blood stream. They have been identified in the peripheral blood of most patients with metastatic disease and in patients with localized cancers, but at exceedingly low levels (about 1 CTC/mL to 100 CTC/mL of blood, or 1 CTC per million leukocytes). To date, CTCs have been used as a metric for tumor expansion, to predict disease progression, and to monitor treatment response. An emerging application is to use these cells for molecular analysis, thus reducing the need for invasive biopsies. Unfortunately, the ability to molecularly detect and profile CTCs has been largely lacking to date since existing clinical analysis techniques are not sufficiently sensitive.

In prior research, our lab developed a miniaturized NMR (μNMR). This device has been used to detect cells that are specifically tagged with superparamagnetic nanoparticles, thus providing a quantitative read-out of cell number and biomarker expression level. A device capable of rapidly concentrating and separating rare CTCs from blood that could interface directly with the μNMR would be extremely powerful, increasing the detection sensitivity and selectivity of molecular analysis.

Approach: We designed a microfluidic device that utilizes an array of 10 μm-diameter posts that are offset from one column to the next along the direction of flow. The microposts were placed such that larger epithelial tumor cells (12 μm to 25 μm in diameter) are displaced while smaller blood cells (< 10 μm in diameter) pass straight through the device based on a technique termed deterministic lateral displacement. Lateral displacement devices, also known as 'bumping chips,' have previously been used to separate the various components of blood from one another, but to date have not been applied to isolation of tumor cells. We designed two different stages, the first for concentration of tumor cells to the center of the device and the second for high-level purification into buffer. Both stages were designed with four different micropost arrangements to optimize 'bumping' characteristics for tumor cells. To fabricate the bumping chips, standard photolithography techniques were employed at the CNST NanoFab to generate patterns of photoresist corresponding to the micropost structures on silicon wafers. The patterned wafers were subsequently etched using deep-reactive ion etching via the Bosch process. Residual photoresist was removed using plasma treatment.



(a) Schematic of the lateral displacement device ('bumping chip'), including the two separate stages. (b) SEM images of etched micropost structures.

Results and Discussion: To concentrate and separate tumor cells from blood, we have focused on fabricating a microfluidic device with micropost structures that were etched directly in silicon wafers. After optimizing the deep-reactive ion etching process, a protocol was developed that resulted in microposts with high aspect ratio (10 μm diameter, about 80 μm height) and extremely smooth vertical sidewalls. In the fabricated bumping chips, the large aspect ratio obtained is 4 times greater than that of previous versions while still maintaining vertical sidewalls. This improvement should dramatically increase device flow through, thus enabling large blood samples to be processed in a reasonable time frame. Ongoing work in the lab has been focused on developing novel techniques to label tumor cells with magnetic nanoparticles and improving the μNMR device (see recent publications). The performance of the bumping chips will be tested in the near future, modified if necessary, and integrated with the μNMR.

Recent Publications:

Chip-NMR biosensor for detection molecular analysis of cells, H. Lee, E. Sun, D. Ham, and R. Weissleder, *Nature Medicine* **14**, 869-874 (2008).

Rapid detection and profiling of cancer cells in fine-needle aspirates, H. Lee, T. J. Yoon, J. L. Figueiredo, F. Swirski, and R. Weissleder, *Proceedings of the National Academy of Sciences USA* **106**, 12459-12464 (2009).

Bioorthogonal chemistry amplifies nanoparticle binding and enhances the sensitivity of cell detection, J. B. Haun, N. K. Devaraj, S. A. Hilderbrand, H. Lee, and R. Weissleder, *Nature Nanotechnology* **5**, 660-665 (2010).

Neural Probes with Waveguides and a Fiber-Coupled Light Emitting Diode

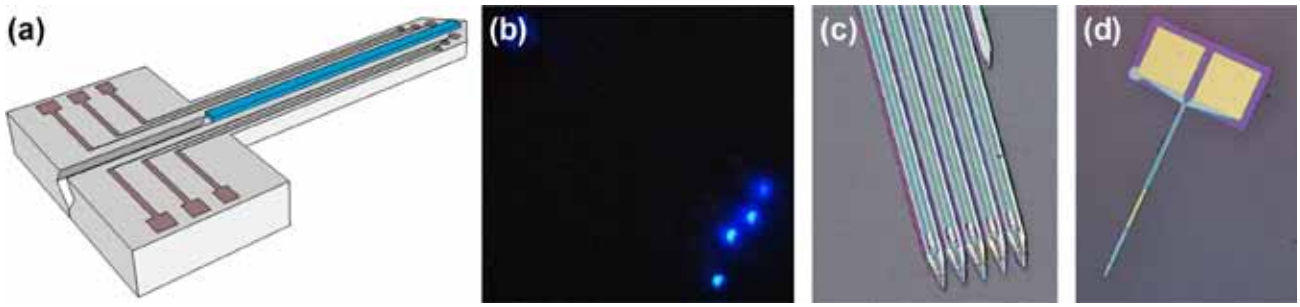
Research Participants: J. N. Mateo and B.G. Jamieson
Scientific Biomedical Microsystems, Columbia, MD 21046

Project Goal: To develop an implantable neural probe system to optically stimulate targeted neurons and record multi-cell neuronal activity by integrating a fiber-coupled light emitting diode (LED) with silicon probes fabricated with SU8 waveguides.

Key Accomplishments:

- Designed, constructed, and tested a fiber-to-LED coupler that couples a single LED to four optical fibers.
- Designed and began fabricating an integrated, extracellular recording array with micromachined fiber alignment grooves, an SU8 waveguide, and stress-compensated penetrating silicon shanks.

Background: Using an implantable, integrated microsystem to modulate neuronal activity with optical stimulation and to simultaneously record single unit action potentials is a potentially groundbreaking approach to systems neurophysiology. SB Microsystems is working with neurophysiologists at the Howard Hughes Medical Institute (HHMI) to produce highly integrated electrodes that can be used in studies of learning, memory, and



(a) Schematic view of integrated probe with SU8 waveguide, an etched fiber alignment groove, and an array of metal microelectrodes for monitoring neuronal activity. A high intensity blue LED is coupled into the waveguide with a 4:1 coupler, not shown in this view, but shown in (b) in operation. (c) and (d) Optical micrographs of fabricated passive neural microprobes currently in use at HHMI.

brain plasticity. When excitable cells with a genetically-expressed protein called Channelrhodopsin (ChR2) are exposed to blue light (480 nm), neuronal action potentials are triggered in these cells. Light is typically delivered using a laser or an LED coupled to an optical fiber, and the electrical signals are probed using an independent microelectrode. Some recent efforts at integrating the light delivery with the microelectrode have been reported; however, these approaches rely on highly labor-intensive "one-off" assembly procedures in which optical fibers are etched and then manually aligned and attached to silicon recording arrays. Others have demonstrated probes with integrated waveguides, but the light sources (i.e. laser or LED) are bulky, are separate from the probe, and connected by a fiber, all of which is problematic for behavioral experiments. Designing a neural probe with integrated optical stimulation allows implanting the probes without a tether, and enables the selective stimulation and monitoring of neuronal activity in freely behaving animals.

Approach: MEMS silicon neural probes with integrated waveguides are being fabricated in the CNST NanoFab with a bulk micromachined silicon-on-insulator process. A top dielectric stack of stress-balanced, oxide-nitride-oxide layers provides hermeticity from the tissue during implantation. Recording sites are patterned from platinum or iridium, which can subsequently be oxidized to reduce site impedance. U-shaped grooves are wet-etched where the optical fiber is to be fixed, and an SU8 waveguide is patterned. Probe boundaries are etched with DRIE, with a second patterned DRIE backside etch used to release the probes from the wafer. An LED coupled to four optical fibers is fixed on a small headstage (printed circuit board), with fiber ends aligned in the grooves to the SU8 waveguides.

Results and Discussion: A coupler that connects a high intensity LED (XRE Cree XLamp) to four optical fibers has been designed, constructed, and tested. The coupler is made of ceramic, stainless steel, and brass, and is fixed onto the LED using an ultraviolet light-curable adhesive. The optical power values measured using a Thorlabs PM100A power meter at the end of each fiber is practically equal (±12 %) across the four fibers.

Passive neural probes (i.e., probes without optical stimulation) fabricated using the bulk micromachined SOI process described above are currently being used in behavioral experiments by our collaborators at HHMI. The integration of optical elements onto these devices represents an opportunity for a system-level approach that will accelerate experimental progress in this area.

More generally, SB Microsystems works on the integration of advanced functionality (CMOS circuitry, optical sources and detectors, acoustic sources and other actuators) into MEMS and microfluidic systems. The development of highly integrated and functional biomedical microsystems is key focus of the research and development work at SB Microsystems, and has been enabling the development of highly miniaturized, low power, and capable systems for biomedical applications.

Acknowledgments: SB Microsystems acknowledges Drs. Tim Harris and Sebastien Royer at HHMI, Dr. Gyorgy Buzskai at Rutgers University, and the team at Neuronexus Technologies, Inc.

Personnel Highlights

The CNST technical staff includes Project Leaders, Postdoctoral and Student Researchers, Visiting Fellows, and Process Engineers and Technicians. The CNST offers a dynamic, multidisciplinary environment for scientists at all career stages—from high school interns, through senior researchers seeking a highly productive sabbatical visit.

NEW PROJECT LEADERS AND NANOFAB STAFF

The CNST has brought together talented, motivated Project Leaders from many disciplines who are open minded about other fields and eager to collaborate on important measurement problems in nanotechnology. NanoFab Process Engineers and Technicians generally have broad nanofabrication and measurement experience, and the desire and ability to train and provide outstanding support to NanoFab researchers. The following staff members have joined the CNST since the start of FY2009 (October 1, 2008), listed alphabetically with the month that they started in parentheses.

Jerry Bowser (January 2010) is a NanoFab Technician with over 13 years of experience in semiconductor manufacturing engineering in the private sector, including five years at Allied-Signal and eight at Covega, where he held both engineering and supervisory positions. He has significant experience with diffusion/oxidation and chemical vapor deposition processes and has worked extensively with indium phosphide and lithium niobate optical devices.

Rachel Cannara (April 2009) is a Project Leader in the Nanofabrication Research Group. She received a B.S. in Physics with Highest Honors from the University of California, Santa Cruz, where her scholarship was recognized with numerous awards. She then received a Ph.D. in Physics from the University of Wisconsin-Madison, where she received the Hirschfelder Award for academic achievement and studied the tribological properties of single crystal diamond. Rachel joined CNST after two years as a Postdoctoral Fellow at the IBM Zurich Research Laboratory. Her current research focuses on the measurement and control of the mechanisms that govern nanoscale frictional energy dissipation, and the use of these discoveries to enable the design and operation of future nanomechanical and nanomanufacturing systems.

Andrea Centrone (August 2010) is a Project Leader and Visiting Fellow in the Energy Research Group. He received a Laurea degree and a Ph. D. in Materials Engineering from the Polytechnic University of Milan, Italy, where he developed a Raman cell to study the interactions between hydrogen and nanoporous materials for hydrogen storage applications. Andrea then performed postdoctoral work at the Massachusetts Institute of Technology, first as a Rocca Fellow in the Department of Material Science and Engineering, studying the phase separation of molecules self assembled on metal nanoparticles and their effect on the nanoparticles' solubility. He continued his postdoctoral work in the Department of Chemical Engineering, investigating the use of metal-organic framework materials for separating small molecules, and gold nanorods for in vivo cancer detection and treatment. In the CNST, Andrea is developing new measurement methods using vibrational spectroscopy, including the development of an IR-spectrometer with nanoscale spatial resolution.

Justin Dickinson (June 2010) is a NanoFab Process Engineer with a B.S. in Aerospace Engineering from the University of Florida in Gainesville, FL, where he worked on a variety of design, development, and programming projects on the Human Powered Submarine Team and in the Intelligent Machine Design Laboratory. Justin provides training and process support during the NanoFab's evening operating hours (Monday through Friday, from 3 pm to midnight).

Gregg Gallatin (April 2009) is a Project Leader in the Nanofabrication Research Group with a B.S. and Ph.D. in Physics from Penn State. After earning his Ph.D., Gregg spent several years working in academia and then transitioned to industry, where he did research at Perkin-Elmer, SVG Lithography, Bell Labs and IBM. While working in industry, Gregg specialized in analysis, modeling and design of the systems, subsystems and processes associated with the lithographic fabrication of semiconductor devices. He holds 12 U.S. patents and has over 60 publications. Gregg's CNST research is focused on the modeling and analysis of the physics of self-assembly.

Paul Haney (December 2010) is a Project Leader in the Energy Research Group. He received a B.S. in Physics and Mathematics from The Ohio State University, and a Ph.D. in Physics from The University of Texas at Austin. His thesis focused on first-principles calculations of electron and spin transport in magnetic and antiferromagnetic multilayers. Paul spent two years as an NRC Postdoctoral Research Associate in the Electron Physics Group, where he worked with Mark Stiles on the theory of spin transfer torque in magnetic semiconductors and in materials near their Curie temperature. He moved to the Energy Research Group in 2009. Paul's current research includes the effects of disorder in materials used for renewable energy, specifically applied to organic photovoltaics and thermoelectrics.

Chester Knurek (December 2009) is a NanoFab Process Engineer with a B.S. in Electronics Engineering from DeVry University in Chicago, IL. Prior to joining NIST, he worked at AT&T Microelectronics/Lucent Technologies - Bell Labs on characterization and process development for several mask formats, including binary/phase-shift photo masks, point source X-ray, and flood electron beam. He also has experience developing instrumentation to characterize membrane stress uniformity.

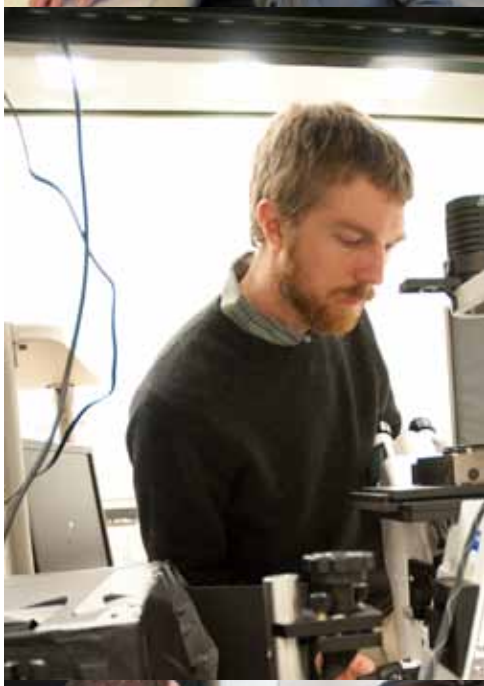
Chester is responsible for training and process support for NanoFab users in various nanofabrication areas, including optical lithography and metrology.

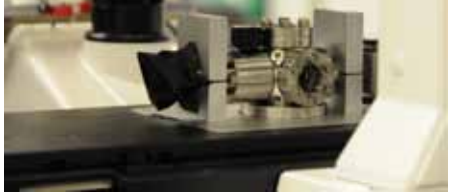
Vincent Luciani (November, 2008) is the CNST's NanoFab Manager, with over 30 years of private industry experience in semiconductor and nanotechnology process development and project management. Vincent began his career at Solarex Corp. producing photovoltaic solar cells. He then joined the Bendix Advanced Technology Center, developing electronic and nanotechnology devices and processes in a variety of semiconductor material systems, including silicon, gallium arsenide, indium phosphide and lithium niobate. When Bendix became part of Allied-Signal, Vincent went on to lead their advanced process development team, and was awarded an Allied-Signal Premier Achievement Award for excellence in Engineering. Prior to joining NIST, he led the process and product engineering teams at Covega Corporation, developing and ramping up the production of novel indium phosphide photonic devices. Vincent is an expert in Project Management, with a Six Sigma Blackbelt, and holds five patents in semiconductor and nanofabrication technology.

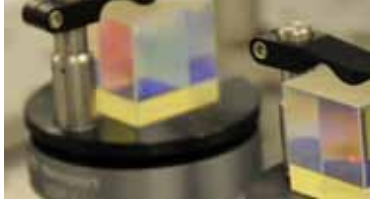
Ann Smith (January 2009) was a NanoFab Process Engineer until July 2010. She had a B.A. in Environmental Studies at the University of Maryland, Baltimore County with a focus in health and conservation, and extensive laboratory experience. In the NanoFab, Ann provided training and process support in various areas, including optical photolithography, metrology, deposition, and oxidation.

Alline Myers (September 2010) is a Senior Process Engineer in the NanoFab, with a B.S. in Physics from North Carolina State University, an M.S. in Physics from Penn State, and a Ph.D. in Materials Science and Engineering from North Carolina State University. Alline has extensive experience in materials and nanostructure characterization using field emission scanning TEM, FIB systems, and SEM. After working at NIST as an NRC Postdoctoral Research Associate in the Chemical Science and Technology Laboratory, Alline spent over 10 years in the semiconductor industry, working at Advanced Micro Devices and Spansion, where she applied analytical electron microscopy techniques to the characterization and failure analysis of logic and flash memory devices. Alline is currently responsible for training and assisting NanoFab users on the Titan TEM, optimizing the performance of the TEM, and helping train users on the dual beam FIB systems.

Fred Sharifi (April 2009) is a Project Leader in the Energy Research Group. He received a B. S. in Physics (*magna cum laude*) from the University of Illinois, where he continued his graduate education and received a Ph.D. in Condensed Matter Physics. He then performed postdoctoral research at Bell Laboratories and at the University of California San Diego, studying electronic transport in one-dimensional superconductors and tunneling spectroscopy of unconventional superconductors. In 1993, Fred became a faculty member at the University of Florida, where he was awarded an Alfred Sloan Foundation Fellowship, focusing on







electronic transport and tunneling in magnetic systems. He joined TRW in 2001, working on superconducting electronics, and for the past five years was a Principal Scientist at GE Global Research in the Advanced Technology Program. Fred's CNST research is focused on measuring nanoscale energy processes in thermoelectrics and photovoltaics.

Renu Sharma (July 2009) is a Project Leader in the Nanofabrication Research Group. She received a B.S. and B.Ed. in Physics and Chemistry from Panjab University, India, and M.S. and Ph.D. degrees in Solid State Chemistry from the University of Stockholm, Sweden, where she had a Swedish Institute Fellowship. Renu came to the CNST from Arizona State University (ASU), where she most recently served as a Senior Research Scientist in the LeRoy Eyring Center for Solid State Science and as an affiliated faculty member in the School of Materials and Department of Chemical Engineering. Renu has been a pioneer in the development of environment-cell TEM, combining atomic-scale resolution with dynamic chemical analysis of gas-solid reactions. She has applied this powerful implementation of TEM to characterize the atomic-scale mechanisms underlying the synthesis and reactivity of nanoparticles (including catalysts), nanotubes, nanowires, inorganic solids, ceramics, semiconductors, and superconductor materials. Renu has received a Deutscher Akademischer Austauschdienst (DAAD) Faculty Research Fellowship, is a past President of the Arizona Imaging and Microanalysis Society, and has over 140 publications. Renu is establishing the CNST's advanced TEM measurement capabilities for nanoscience research and overseeing the operation of a new TEM tool in the NanoFab.

Eileen Sparks (February 2009) is a NanoFab Process Engineer with over 30 years of experience in semiconductor process development, materials characterization, and contamination control. Eileen has B.S. and M. Ph. degrees in Chemistry from George Washington University. Early in her career, she led surface analysis research at Comsat, characterizing III-V semiconductor-based materials and devices, including failure analysis. She then joined Fusion Systems as a Process Development Engineer, developing, installing, and qualifying downstream microwave and ozone ashing processes for residue removal in OEMs and foundries world-wide. Prior to joining NIST, Eileen worked at ATMI with major chipmakers on wafer cleaning and residue removal for CMOS, MEMS, and MRAM applications. Eileen is currently assisting the NanoFab with base-line process development, FIB training, and industrial outreach.

Veronika Szalai (September 2010) is a Project Leader in the Energy Research Group. She received an A.B. in Chemistry from Bryn Mawr College, and a Ph.D. in Inorganic Chemistry from Yale University. Her thesis work centered on biophysical measurements of water oxidation chemistry in natural photosynthesis. After completing postdoctoral work at the University of North Carolina at Chapel Hill, Veronika spent several years on the faculty in the Department of Chemistry and Biochemistry at the University of Maryland Baltimore County. During that time, she and her group elucidated the biophysical chemistry of copper in Alzheimer's disease fibrils, investigated the effects of DNA condensation on DNA oxidation processes, and developed methods to create quadruplex-based DNA materials. Veronika is leading CNST projects focused on the fabrication of biomimetic nanostructures and on fundamental measurements of nanobiomaterials for artificial photosynthesis applications.

A. Alec Talin (July 2009) is a Project Leader in the Energy Research Group with a B.A. in Chemistry from the University of California at San Diego and a Ph.D. in Materials Science and Engineering from the University of California at Los Angeles. After completing his postdoctoral work at Sandia National Laboratories, Alec spent several years with Motorola Physical Sciences Research Labs, first as a staff scientist and subsequently managing the Materials Characterization Laboratory. In 2002, Alec returned to Sandia to direct and develop programs in nanofabrication, nanoelectronics, photonics, and sensing. Alec is leading CNST projects focused on fundamental measurements of nanostructured materials in electrochemical energy storage.

VISITING FELLOWS

An integral facet of CNST's dynamic, multidisciplinary research environment is the CNST Visiting Fellows program. This program is designed to encourage outstanding, senior researchers from academic, industrial, and government laboratories world-wide to come to NIST to collaborate with CNST's research staff. Researchers selected to be CNST Visiting Fellows may collaborate for extended periods via regular, short-term visits, and/or may work in residence full-time—for example, during a year-long sabbatical. The following researchers have worked at the CNST as Visiting Fellows since the start of FY2009, listed alphabetically along with their home institutions.

- *Vladimir Aksyuk,* University of Maryland, College Park
- *Andrea Centrone,* University of Maryland, College Park
- *Kamil Ekinci*, Boston University
- *John Hartley*, University at Albany, SUNY
- *C. Stephen Hellberg*, Naval Research Laboratory
- *Young Kuk*, Seoul National University, Seoul, Korea
- *Tomohiro Matsui*, University of Tokyo, Tokyo, Japan

- *Jacques Miltat*, Université Paris-Sud XI, Paris, France
- *David R. Penn*, NIST Emeritus
- *Daniel Pierce*, NIST Fellow Emeritus
- *Ben Shapiro*, University of Maryland, College Park
- *Mihaela Tanase*, University of Maryland, College Park
- *John Weiner*, Université Paul Sabatier, Toulouse, France

POSTDOCTORAL RESEARCHERS

The CNST has a variety of mechanisms for participating in postdoctoral research, with postdoctoral researchers comprising approximately two thirds of our research staff. The following researchers have worked at the CNST as Postdoctoral Researchers since the start of FY2009, listed alphabetically along with their doctoral fields and institutions.

- *Maxim Abashin,* Photonics, University of California, San Diego
- *Shaffique Adam,* Theoretical Physics, Cornell University
- *Amit Agrawal,* Electrical Engineering, University of Utah
- *Adam Berro*, Organic Chemistry, University of Texas at Austin
- *Sam Bowden*, Engineering Sciences-Material Science, Dartmouth College
- *Peter Carmichael*, Biochemistry, University of California, San Diego
- *Jungseok Chae,* Physics, Seoul National University, Seoul, Korea
- *Han-Jong Chia*, Physics, University of Texas at Austin
- *Seok-Hwan Chung,* Physics, University of Maryland, College Park
- *Marcelo Davanço*, Electrical and Computer Engineering, University of California, Santa Barbara
- *Zhao Deng*, Analytical Chemistry, University of California, Davis
- *Kan Du, Physics*, University of Massachusetts, Amherst
- *Keith Gilmore*, Physics, Montana State University
- *Behrang Hamadani*, Condensed Matter Physics, Rice University
- *Paul Haney*, Physics, University of Texas at Austin
- *Suyong Jung*, Physics, University of Texas at Austin
- *Raymond Kallaher*, Physics, Florida State University
- *Myung-Gyu Kang*, Electrical Engineering, University of Michigan
- *Nikolai Klimov,* Experimental Condensed Matter Physics, Rutgers University
- *Brenton Knuffman*, Applied Physics, University of Michigan
- *Seung-Hyeon (Sarah) Ko*, Chemistry, Purdue University

- *Stephan Koev,* Electrical Engineering, University of Maryland, College Park
- *Niv Levy*, Physics, University of California at Berkeley
- *Pramod Mathai*, Aerospace Engineering, University of Maryland, College Park
- *Stefano Mazzucco*, Physics, Université Paris-Sud XI, Paris, France
- *Matthew McMahon*, Physics, Vanderbilt University
- *Ben McMorran*, Physics, University of Arizona
- *Houxun Miao*, Electrical Engineering, Purdue University
- *Hongki Min*, Physics, University of Texas at Austin
- *Sander Otte*, Physics, Leiden University, Leiden, The Netherlands
- *Sukumar Rajauria,* Physics, University of Joseph Fourier/Neel, Grenoble, France
- *Matthew Rakher*, Physics, University of California, Santa Barbara
- *Gregory Rutter*, Physics, Georgia Institute of Technology
- *Dmitry Ruzmetov*, Physics, Indiana University
- *Young Jae Song*, Physics, Seoul National University, Seoul, Korea
- *Adam Steele*, Physics, Georgia Institute of Technology
- *Gila Stein*, Chemical Engineering, University of California, Santa Barbara
- *Ceren Susut*, Chemistry, Georgetown University
- *Hua Xu*, Applied Physics, University of Maryland, College Park
- *Tong Zhang*, Physics, Institute of Physics at the Chinese Academy of Sciences
- *Meng Zhu,* Experimental Condensed Matter Physics, Penn State University

STUDENT RESEARCHERS ANDSUMMER UNDERGRADUATE RESEARCH FELLOWS

The CNST's collaborative approach to research creates a variety of opportunities for students to participate in hands-on research under the mentorship of a CNST scientist in our world-class laboratories, including the NanoFab and its cleanroom. We especially encourage undergraduate science and engineering majors interested in nanotechnology to participate in the Summer Undergraduate Research Fellowship (SURF) program at NIST, which offers the opportunity for a student to spend 11 weeks during the summer participating in full-time research at NIST. The following students have worked at the CNST since the start of FY2009, listed alphabetically along with their home institutions, with the SURF participants listed subsequently.

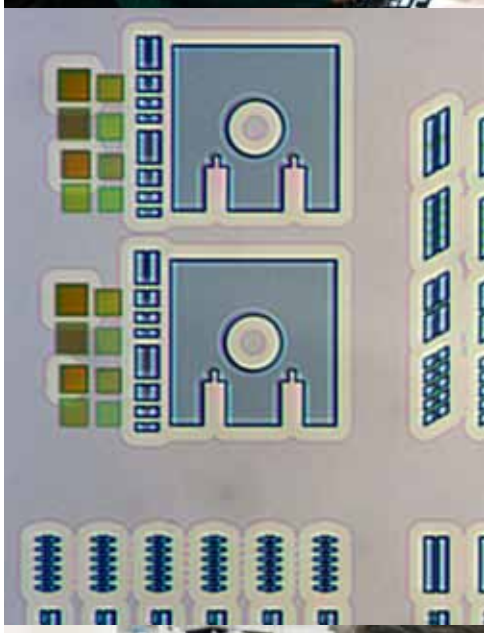
- *Katherine Freeman,* high school intern, Poolesville High School Magnet Program
- *Elizabeth Golovatski,* graduate student, Department of Physics and Astronomy, University of Iowa
- *Wesley Hillard*, high school intern, Poolesville High School Magnet Program
- *Parakh Jain,* high school intern, Poolesville High School Magnet Program
- *Jaime Javier Juarez*, graduate student, Department of Chemical and Biomolecular Engineering, Johns Hopkins University
- *Timothy Lee,* undergraduate, University of Maryland, College Park
- *Isaac Moreyra*, undergraduate, University of Maryland, College Park
- *Michael Rahimi*, undergraduate, University of Maryland, Baltimore County
- *Pedram Roushan*, graduate student, Physics Department, Princeton University
- *Jungpil Seo*, graduate student, Physics Department, Princeton University
- *Rebecca Thomas,* graduate student, Department of Electrical and Computer Engineering, North Carolina State University
- *Charles Yeh*, high school intern, Poolesville High School Magnet Program
- *Chenyu Zhao,* high school intern, Blair High School Magnet Program

2009 SURF

- *Jason Bylsma*, University of South Florida
- *Suehyun Cho*, University of Maryland, College Park
- *Aaron Cochran*, University of Wisconsin-Stout
- *Timothy Enright*, University of the Sciences in Philadelphia
- *Christopher Hong*, Pennsylvania State University
- *Robert Hoyt*, Harvey Mudd College
- *Joshua Leibowitz*, University of Connecticut
- *Nicole Messier*, George Washington University
- *Gregory Meyer*, University of Maryland, College Park
- *Karthik Prakhya*, University of Massachusetts

2010 SURF

- *Philip Campbell*, University of Texas at Dallas
- *Olivia Hoff*, University of Southern Mississippi
- *Robert Hoyt*, Harvey Mudd College
- *Joshua Leibowitz*, University of Connecticut
- *Ramsey Noah*, California State University Long Beach
- *Brian Soe*, Harvey Mudd College
- *Mathew Swisher*, Carnegie Mellon University
- *Martin Vilarino*, University of Maryland
- *Richard Webb*, University of Texas at Austin



CNST Staff Honors and Awards

Honors and awards received by CNST staff members, including postdoctoral researchers and students, during FY2009, FY2010, and the first quarter of FY2011 (December 31, 2010) are listed below in reverse chronological order. Additional information is provided for the most significant awards.

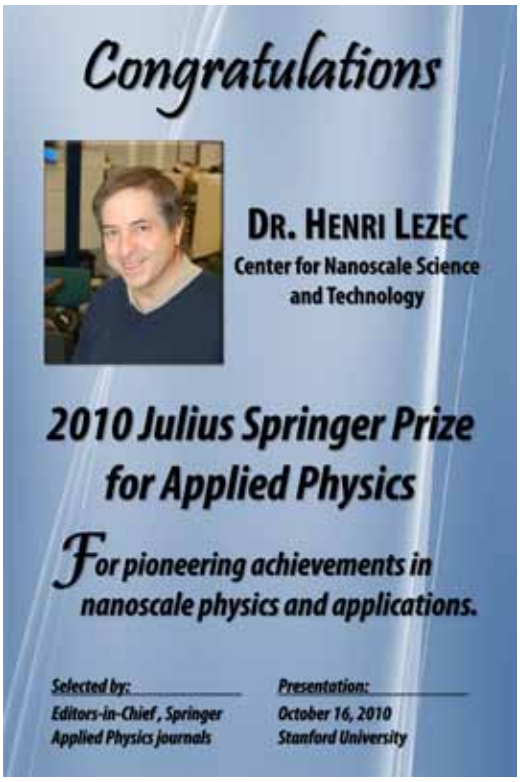
Joseph Stroscio, Fellow, American Association for the Advancement of Science (AAAS), 2010. For 2010, 503 AAAS members were chosen for this honor by their peers in recognition of scientifically or socially distinguished efforts to advance science or its applications. Stroscio was elected as an AAAS Fellow as part of the Physics Section for his "distinguished contributions to the fields of surface and condensed matter physics, particularly for the development and application of scanning tunneling microscopy."

Parakh Jain, Semifinalist, Intel Science Talent Search, December 2010. Jain, a senior in the Math, Science, and Computer Science Magnet Program at Poolesville High School and student intern in the Electron Physics Group, was one of 300 semifinalists selected from 1744 applicants. Jain's application described calculations of the transport properties of multilayer graphene performed in collaboration with NRC Postdoctoral Research Associate Shaffique Adam and NIST Fellow Mark Stiles.

Henri Lezec, Julius Springer Prize for Applied Physics, October 2010. 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 prize recognizes researchers who have made an outstanding and innovative contribution to the fields of applied physics, and recognized Lezec for his "pioneering achievements in nanoscale physics and applications."

Mark Stiles, "Outstanding Referee" for Physical Review, March 2010. This lifetime APS award was initiated in 2008 "to recognize scientists who have been exceptionally helpful in assessing manuscripts for publication in the APS journals." The status 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.

Shaffique Adam, NIST Sigma Xi Postdoctoral Best Poster Award, March 2010.



Joe Stroscio, AVS Nanometer-Scale Science and Technology Division Nanotechnology Recognition Award, November 2009. This award recognizes outstanding scientific and technical contributions in the science of nanometer-scale structures, technology transfer involving nanometer-scale structures, and/or the promotion and dissemination of knowledge and development in these areas. Stroscio was recognized "for his pioneering development of instrumentation to create and characterize nanostructures enabling fundamental insights into the mechanisms of atom manipulation and the magneto-electric properties of low-dimensional structures."

Robert McMichael, Fellow, American Physical Society, November 2009, awarded for "broad contributions to the measurement, modeling, interpretation, and understanding of magnetization dynamics."

Robert McMichael, Department of Commerce, Silver Medal Award, November 2009.

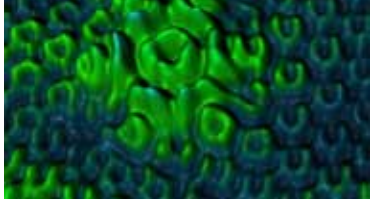
Mark Stiles, NIST Fellow, June 2009.

Alan Band, NIST Sigma Xi Award for Outstanding Services to NIST Research Scientists, May 2009. The NIST Chapter of Sigma Xi recognized Band for instrumentation solutions that made many programs productive throughout NIST and, on occasion, at other federal organizations, including contributions to a science display at the Smithsonian American History Museum.

Greg Rutter, Best Ph.D. Thesis Award by the Georgia Institute of Technology Chapter of Sigma Xi, April 2009. This award recognized Rutter's thesis, "Atomic Scale Properties of Epitaxial Graphene Grown on SiC(0001)," which was completed under the direction of Professor Phillip First in the School of Physics, with the majority of the research performed at the CNST under the guidance of Joseph Stroscio.

Dan Pierce, "Outstanding Referee" for Physical Review, March 2009. See award description above for Stiles.

Gregory Rutter, AVS Dorothy M. and Earl S. Hoffman Award, October 2008. The AVS recognized Rutter for continuing excellence in graduate studies in the sciences and technologies of interest to AVS for his doctoral research done at the CNST in collaboration with the Georgia Institute of Technology.



Research Participant Publications

One measure of the impact of CNST as a national user facility is the publications by CNST Research Participants; those working directly on CNST staff projects, NanoFab projects, or through a grant from the CNST. The publications associated with NanoFab projects and grant research are listed for FY2009 and FY2010. The publications authored by CNST staff members are first listed separately, and include those published and in press through the first quarter of FY2011 (December 31, 2010). Published papers are listed in reverse chronological order.

CNST STAFF PUBLICATIONS (FY2009–FY2011:Q1)

1. Atomic-level stick-slip, R. W. Carpick and R. J. Cannara, in *Encyclopedia of Tribology*, *in press*.
2. Effect of alternating Ar and SF_6/C_4F_8 gas flow in Si nano-structure plasma etching, L. Chen, V. Luciani, and H. Miao, *Microelectronic Engineering*, *in press*.
3. Electronic structure of multilayer graphene, H. Min, in *Graphene Nanoelectronics: Metrology, Synthesis, Properties and Applications*, edited by H. Raza (Springer, 2011), *in press*.
4. Electron transport in two dimensional graphene, S. Das Sarma, S. Adam, E. Hwang and E. Rossi, *Review of Modern Physics, in press*.
5. Feedback control of micro-flows, M. Armani, Z. Cummins, J. Gong, P. Mathai, R. Probst, C. Ropp, E. Waks, S. Walker, and B. Shapiro, in *Feedback Control Systems for Micro- and Nano-Scales: MEMS to Atoms*, edited by J. Gorman and B. Shapiro (Springer, 2011), *in press*.
6. First-principles study of defect migration in RE-doped ceria (RE = Pr, Gd), R. Sharma, P. Dholabhai, J. B. Adams, and P. Crozier, in Next-Generataion Fuel Cells—New Materials and Concepts, Proceedings of the Material Research Society, Vol. 1311 (Boston, MA), *in press*.
7. Graphene carrier transport theory, S. Adam, in *Graphene Nanoelectronics: Metrology, Synthesis, Properties and Applications*, edited by H. Raza (Springer, 2011), *in press*.
8. Micro-optical techniques, K. Srinivasan, M. T. Rakher, and M. Davanco, in *Optical Techniques for Materials Characterization* (Taylor and Francis), *in press*.
9. Nanoscale friction: measurement and analysis, R. J. Cannara, in *Micro- and Nano Scale Phenomena in Tribology*, *in press*.
10. Perspective on probing metallic ferromagnetism with electrons, D. T. Pierce, *Journal of Applied Physics, in press*.
11. Plasma etching uniformity control for making large and thick dual-focus zone plates, L. Chen, Q. Wang, and U. Griesmann, *Microelectronic Engineering*, *in press*.
12. Reversal of patterned Co/Pd Multilayers with graded magnetic anisotropy, J. E. Davies, P. Morrow, C. L. Dennis, J. W. Lau, B. McMorran, A. Cochran, J. Unguris, R. K. Dumas, P. Greene and Kai Liu, *Journal of Applied Physics* **109** (2011), *in press*.
13. Electron-beam-induced growth of TiO_2 nanostructures, S. W. Chee, S. Sivaramakrishnan, R. Sharma, and J. Zuo, *Microscopy and Microanalysis* **17**, 274-278 (2011).
14. Metal–insulator–metal surface plasmon polariton waveguide filters with cascaded transverse cavities, L. O. Diniz, F. D. Nunes, E. Marega, J. Weiner, and B. V. Borges, *Journal of Lightwave Technology* **29**, 714-720 (2011).
15. Evolution of microscopic localization in graphene in a magnetic field: from scattering resonances to quantum dots, S. Jung, G. M. Rutter, N. N. Klimov, D. B. Newell, I. Calizo, A. R. Hight-Walker, N. B. Zhitenev, and J. A. Stroscio, *Nature Physics* **7**, 245-251 (2011).
16. Effect of interactions on edge property measurements in magnetic multilayers, M. Zhu and R. D. McMichael, *Journal of Applied Physics* **109**, 04PDF3904 (2011).
17. Ordered metal nanostructure self-assembly using metal–organic frameworks as templates, B. W. Jacobs, R. J. T. Houk, M. R. Anstey, S. D. House, I. M. Robertson, A. A. Talin, and M. D. Allendorf, *Chemical Science* **2**, 411-416 (2011).
18. Enhanced magnetization drift velocity and current polarization in CoFe1–xGex alloys, M. Zhu, B. D. Soe, R. D. McMichael, M. J. Carey, S. Maat, and J. R. Childress, *Applied Physics Letters* **98**, 072510 (2011).
19. Effects of disorder on magnetic vortex gyration, H. Min, R. D. McMichael, J. Miltat, and M. D. Stiles, *Physical Review B* **83**, 064411 (2011).
20. Optomechanical transduction of an integrated silicon cantilever probe using a microdisk resonator, K. Srinivasan, H. Miao, M. T. Rakher, M. Davanco, and V. Aksyuk, *Nano Letters* **11**, 791-797 (2011).
21. 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).
22. Fiber-based cryogenic and time-resolved spectroscopy of PbS quantum dots, M. T. Rakher, R. Bose, C. W. Wong, and K. Srinivasan, *Optics Express* **19**, 1786-1793 (2011).
23. Electron vortex beams with high quanta of orbital angular momentum, B. J. McMorran, A. Agrawal, I. M. Anderson, A. A. Herzing, H. J. Lezec, J. J. McClelland, and J. Unguris, *Science* **331**, 192 -195 (2011).
24. 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* **23**, 414-420 (2011).
25. A 10 mK scanning probe microscopy facility, Y. J. Song, A. F. Otte, V. Shvarts, Z. Zhao, Y. Kuk, S. R. Blankenship, A. Band, F. M. Hess, and J. A. Stroscio, *Review of Scientific Instruments* **81**, 121101 (2011).
26. Nanofabrication techniques for controlled drug released devices, L. Chen, G. Henein, and V. Luciani, *Nanomedicine* **6**, 1-6 (2011).
27. Efficiency enhancement of copper contaminated radial p-n junction solar cells, A. Boukai, P. Haney, A. Katzenmeyer, G. M. Gallatin, A. A. Talin, and P. Yang, *Chemical Physics Letters* **501**, 153-158 (2011).

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

29. High-frequency nanofluidics: a universal formulation of the fluid dynamics of MEMS and NEMS, K. L. Ekinci, V. Yakhot, S. Rajauria, C. Colosqui, and D. M. Karabacak, *Lab on a Chip* **10**, 3013-3025 (2010).

30. Focused chromium ion beam, A. V. Steele, B. Knuffman, J. J. McClelland, and J. Orloff, *Journal of Vacuum Science and Technology B* **28**, C6F1-C6F5 (2010).

31. 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).

32. Contact resistance of flexible, transparent carbon nanotube films with metals, H. Xu, L. Chen, L. Hu, and N. Zhitenev, *Applied Physics Letters* **97**, 143116 (2010).

33. Atomistic factors governing adhesion between diamond, amorphous carbon, and model diamond nanocomposite surfaces, P. L. Piotrowski,, R. J. Cannara, G. Gao, J. J. Urban, R. W. Carpick, and J. A. Harrison, *Journal of Adhesion Science and Technology* **24**, 2471-2498 (2010).

34. Epitaxial graphene electronic structure and transport, W. A. de Heer, C. Berger, X. Wu, M. Sprinkle, Y. Hu, M. Ruan, J. A. Stroscio, P. N. First, R. Haddon, B. Piot, C. Faugeras, M. Potemski, and J. Moon, *Journal of Physics D: Applied Physics* **43**, 374007 (2010).

35. Spatial coherence in electron-beam patterning, G. M. Perera, G. E. Stein, and J. A. Liddle, *Journal of Vacuum Science & Technology B* **28**, 1048 (2010).

36. High-resolution tunnelling 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).

37. 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).

38. Quantum dots precisely placed by controlled flow, J. A. Liddle, *InterNano*, August 24, 2010.

39. A spray drying system for synthesis of rare-earth doped cerium oxide nanoparticles, V. Sharma, K. M. Eberhardt, R. Sharma, J. B. Adams, and P. A. Crozier, *Chemical Physics Letters* **495**, 280-286 (2010).

40. Depth profiling magnetic structure using scanning electron microscopy with polarization analysis (SEMPA), B. McMorran, D. Pierce, and J. Unguris, *Microscopy and Microanalysis* **16**, 1862-1863 (2010).

41. Temperature dependence of the diffusive conductivity of bilayer graphene, S. Adam and M. D. Stiles, *Physical Review B* **82**, 075423 (2010).

42. Polymer-assisted conformal coating of TiO_2 thin films, E. S. Gillman, D. Costello, M. Moreno, A. Raspopin, R. Kasica, and L. Chen, J*ournal of Applied Physics* **108,** 044310 (2010).

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

44. Statistics of camera-based single-particle tracking, A. J. Berglund, *Physical Review E* **82**, 011917 (2010).

45. The NIST Center for Nanoscale Science and Technology: supporting US innovation in nanotechnology, R. Celotta, in *Micro/Nano Symposium (UGIM), 2010 18th Biennial University/Government/Industry* (2010), p. 1-3.

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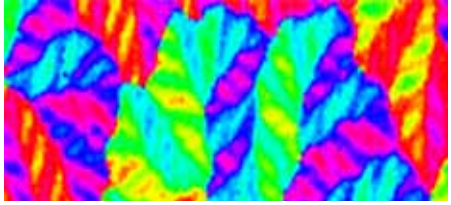

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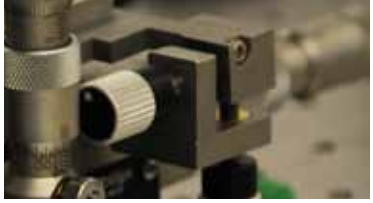

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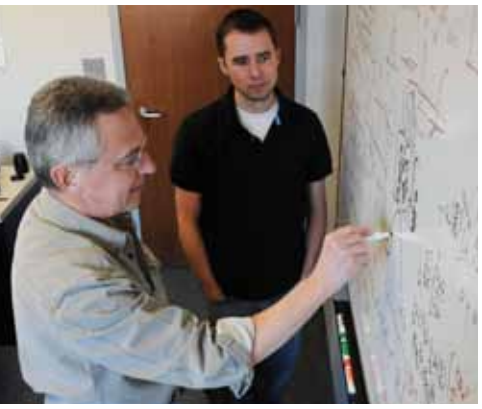

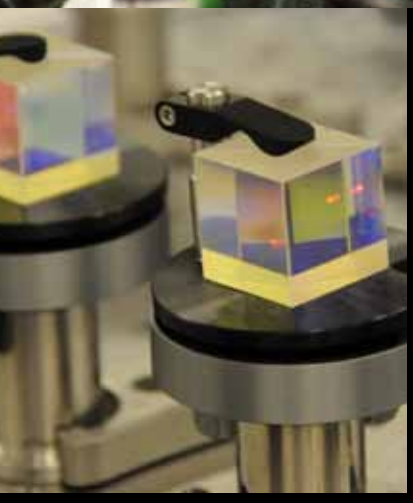

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