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Nanoscale Science, Engineering & Technology

UNIVERSITY OF MINNESOTA

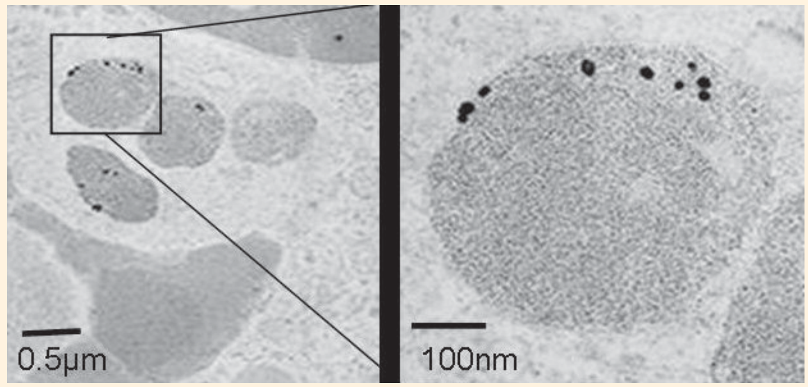
NANOTECHNOLOGY NEWS FROM THE UNIVERSITY OF MINNESOTA

ISSUE EIGHT WINTER 2007

Nano has moved into a new phase at the University with the inception of a new organization, The Center for Nanostructure Applications (CNA). Federal funding in nano has shifted to focus more on the applications of nanotechnology rather than the demonstration of unusual nanostructures or nanostructured materials. The National Science Foundation refers to these applications as "Active Nanostructures". An active nanostructure changes its state during operation in such a way as to carry out a predefined function. This function might be to deliver a drug, detect cancer, convert electrical energy to light (or the inverse), or carry out some mechanical task.

Funded through the Provost's Office and the Office of the Vice President for Research and led by the Dean of the Institute of Technology, the goal of the CNA is to keep the University at the forefront in nano research by encouraging the formation of interdisciplinary teams that will pursue new active nanostructures. This is being done by providing initial device demonstration funding for teams of 3 to 5 faculty who want to work on an application. The funds provide research support for two years so that initial results can be obtained and external proposals can be written. The new Center will also take the lead in promoting nano at the University through workshops, speaker series, and other events. Professor Steve Campbell has been appointed to a 2-year term as the founding Director of the Center for Nanostructure Applications.

WINTER 2007 NANO IMAGE



Gold nanoparticles target prostate cancer. In a collaboration with Cytimmune Sciences, Inc. (Rockville, MD), we have shown that systemically injected gold nanoparticles are selectively taken up and can deliver drugs (i.e. TNF) to LNCaP Pro 5 prostate tumors in mice. Photo courtesy Prof. John Bischof (Mechanical Engineering, Biomedical Engineering).

Reminder: If your work uses CharFac, NFC, or PTL, please add the following in the acknowledgements section of any publication: "Parts of this work were carried out in the Minnesota (Characterization Facility, Nanofabrication Center, or Particle Technology Lab) which receives partial support from NSF through the NNIN program."

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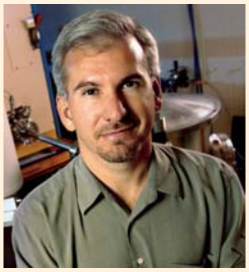
Characterization Facility News

CharFac Director's Message



CharFac Director,
Greg Haugstad

We are excited to announce several developments in training and equipment.

The CharFac has transformed its training course for Introductory Scanning Electron Microscopy (ISEM). (Successful completion of ISEM is required for independent use of CharFac's four field emission SEMs: JEOL 6500 & 6700; Hitachi S-4700 & S-900.) Background content for the course has been developed in the form of a primer which can be accessed through the CharFac website. Trainees read and study the primer prior to training. Hands-on training consists of six hours of group (max 3 students) interaction with the microscope and includes sample loading, system alignment, image capture/saving, and optimizing operational parameters. Each student has an additional one-on-one session with the instructor in which he/she must pass a written test on the ISEM primer as well as a "driving test" on the microscope to demonstrate basic operational competence. Individuals who complete ISEM may arrange for one-on-one training on another SEM or advanced methods: Energy Dispersive Spectroscopy, Electron Backscatter Diffraction, Cathodoluminescence and Cryo-SEM.

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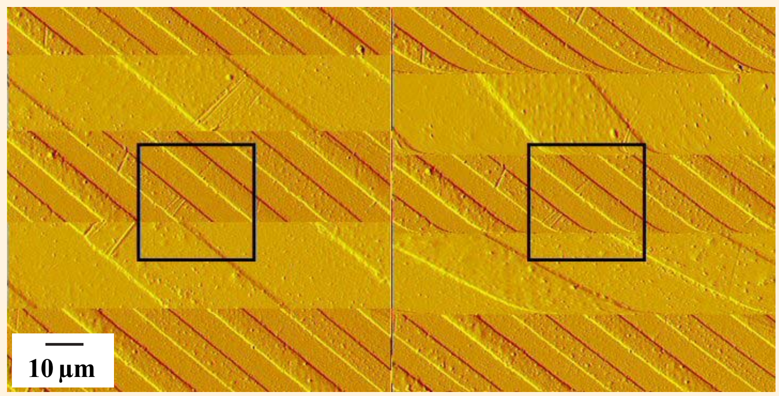
Greg Haugstad, Director
Mike Boucher, Lab Manager

An XYZ closed-loop scanner has been purchased for our MI (now Agilent) PicoPlus AFM system. It uses *inductive sensors* to measure scanner movement while applying voltage biases at the scanner under feedback. Thus creep, nonlinearity and hysteresis are removed. (See figure, left image.) By comparison, conventional scanners *programmatically* vary the biases according to a software calibration with no compensation for scan history. A sudden change of image size (zooming in or out) or location results in strong creep and nonlinearity (right image) until the scanner has fully raster-scanned the new image size for a few minutes. Closed-loop scanning is particularly useful when smaller images are captured following larger survey imaging.



AFM deflection images of metal strips on silicon to compare closed-loop (left) and conventional (right) scanning upon changing back and forth between larger and smaller scans. Upon zooming to the square region or back out, rampant piezoscanner creep is seen when using conventional but not closed-loop scanning.

Analog pulsed force mode (PFM; see Fall 2005 newsletter, CharFac web site) now can be run on our DI (Veeco) AFM systems. A Z sample "shaker" is modulated to provide fast cycling of sample to/from tip. In addition to topography, tip-sample adhesion or interaction stiffness can be imaged at each pixel location within the Nanoscope software. This non-resonant intermittent-contact method improves imaging on many materials compared to contact mode, while providing more meaningful materials contrast compared to phase images acquired in "tapping" mode.

Custom hardware has been developed to cycle the AFM set point during imaging. One application is load ramping in friction force microscopy, to measure friction coefficient from a single image. Custom software averages friction force per scan line then plots versus load. Set point ramping during tapping mode also can be enabling, to identify interaction regimes (attractive, repulsive) and thereby converge quickly on optimal parameter values.

The Nanoindentor XP had been upgraded and repaired. No additional training is needed for experienced users. The Dynamic Contact Module has been repaired allowing extreme low load testing.

CharFac Featured User and Research

Thermally Induced Structural Changes in Nanoporous Silicon Dioxide from X-ray Photoelectron Spectroscopy

Martin T. K. Soh, Fulbright Fellow (2005-2006), University of Minnesota

Silicon dioxide, in particular, its low-temperature evaporated nanoporous polymorphs (np-SiO_2), has been studied extensively for device passivation and optical coatings. The low-packing density and moisture permeability of np-SiO_2 films are well known, leading to alternative deposition schemes to avoid these issues. While these characteristics are usually undesirable, they imply that one might be able to trap adsorbed gases using encapsulation providing a means of controlling stress and perhaps other mechanical properties. This capability would be particularly useful in the design of coatings on membranes in micromechanical devices.

Martin T. K. Soh
Australian-American Fulbright Fellow

In situ x-ray photoelectron spectroscopy was used to study the peak broadening dependence of Si 2*p* and O 1*s* photoelectron peaks as a function of temperature (RT to 300°C) of electron-beam evaporated np-SiO_2, thermally grown SiO_2, and a-quartz. X-ray photoelectron spectroscopy apparatus used in this study are available in the Institute of Technology Characterization Facility. Available equipment consists of an SSI SSX-100 monochromated automated instrument and a Physical Electronics model 555 non-monochromated instrument equipped with cooling and heating stages. The FWHM of np-SiO_2 during the first thermal cycle in vacuo, as shown in figure 1, for the Si 2p peak (figure 1a), is due to desorbed hydrogen bonded water vapor resulting in subsequent bond angle strain relaxation at the surface (open squares fig. 1b). This is contrasted with the thermal (green crosses) and a-quartz (red circles) samples that do not follow this behavior. That is, the FWHM increases with temperature indicating an increase in bond angle strain. After the initial water desorption process from np-SiO_2 occurs, the FWHM indicates a reproducible decrease in bond angle strain and relaxation (solid blue squares). These data have been shown to be reproducible run to run and for different depositions. Given that the first cycle behavior only extends to T=100 °C, the XPS analysis indicates that the mechanism of bond strain relaxation in np-SiO_2 is through much weaker hydrogen bonding shown by the arrows in the model in figure 1. Re-exposure to ambient strongly suggests that any thermally induced bond reconfiguration in np-SiO_2 is not permanent.

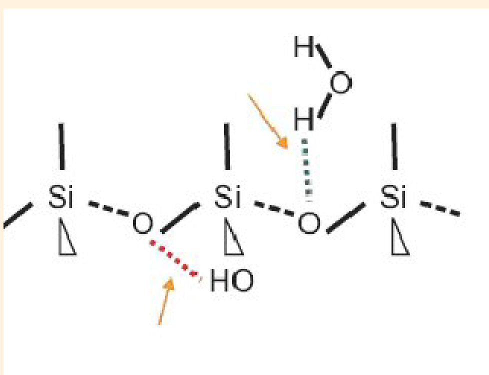
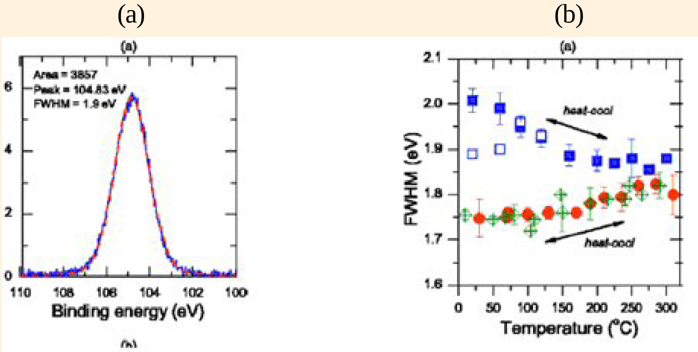


Figure 1

(a) (b)



J. H. Thomas, III, and Joseph J. Talghader, published in J. Vac. Sci. Technol. **A 24** (6), 2147, Nov/Dec 2006.

NANOFABRICATION CENTER NEWS

NFC DIRECTOR'S MESSAGE

NFC Director, Steve Campbell

In this issue I would like to make you aware of a new capability that we will be obtaining for NFC. As a result of a successful Grant-in-Aid proposal led by Professor Beth Stadler (Electrical & Computer Engineering), and using other matching funds obtained from the Dean's office, we are planning to acquire an Atomic Layer Deposition (ALD) system. These systems allow very well controlled growth of extremely thin films, even over highly nonplanar structures such as nanopores, nanowires, and nanoparticles. Typical films are metal oxides such as HfO_2 and Al_2O_3, although metal nitrides and pure metals can also be deposited using this process. For a list of materials that have been demonstrated using ALD go to www.cambridgenanotech.com/papers/al_chemfile_v4_no3.pdf

The process involves the sequential exposure of the substrate to two gasses. The gasses are chosen such that at least one of them saturates the surface at one monolayer of coverage and the process conditions are such that neither gas, by itself, will decompose to form a solid. After exposure to the first gas, the system is flushed, but one monolayer of this gas remains on the substrate where it can react with the second gas to form a

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monolayer of the desired film. The process is repeated until the desired film is grown. For a nice animation of the process, visit http://www.cambridgenanotech.com/animation/.

The proposal for the system is at the bidding stage as you read this. We hope to have the new system installed and available to our users by early summer, 2007.

SURFACE PROFILING

Process characterization is an essential step in any microfabrication process. Measurements of feature height, width and length, film thickness, and thin film stress are quite commonly done by Nanofabrication Center researchers. Recently we took delivery of a new surface profiler, model P16 from KLA-Tencor. The P16 is the latest model in a long line of Tencor surface profilers, and is a research level instrument that is easy to operate. The vertical scan range is 327um, and the tip force is adjustable from 1 to 50mg. Scans up to 200um can be made, with lateral resolution in the scan direction of 25nm. Vertical resolution is less than 1 angstrom, even for features at the maximum of the vertical range. The computer runs on Windows XP, and has Tencor's recently released Apex 3D analysis software. NFC will be setting up the P16 to allow remote access to the computer via the internet. This will enable a researcher that has sent their sample here for measurement to actually connect to the P16 while the scanning is being done by NFC staff, enabling real-time interaction. The cost for academic users is $0.10/minute, with most scans taking 30 minutes or less. Contact us if you are interested in using this state of the art tool.

SAFETY TRAINING

NFC is offering safety training for new users twice each month. On the first Thursday of every month, the training sessions begin at 1:30PM, and on the third Thursday of the month sessions begin at 10:00AM. The training includes watching our safety video and taking a brief quiz. Also, a NFC staff member provides a tour showing some of the safety related equipment and the gowning process used for the NFC cleanroom. Finally, there is training on using the Coral lab software. The safety training takes about two hours to complete, and must be done before users will be granted access to NFC facilities.

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Steve Campbell, Director
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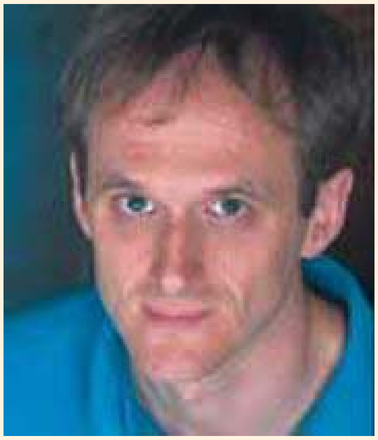
NFC Featured User and Research

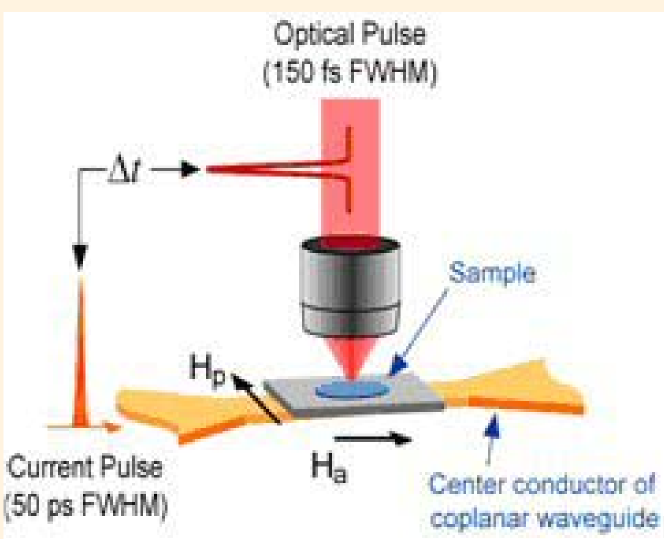
Spin Transport and Dynamics

Mun Chan, David Christle, Robert Compton, Michael Erickson, Eric Garlid, Tsuyoshi Kondo, Xiaohua Lou, Jeff Parker, David Toyli, Jianjie Zhang, and Paul Crowell
University of Minnesota, School of Physics and Astronomy

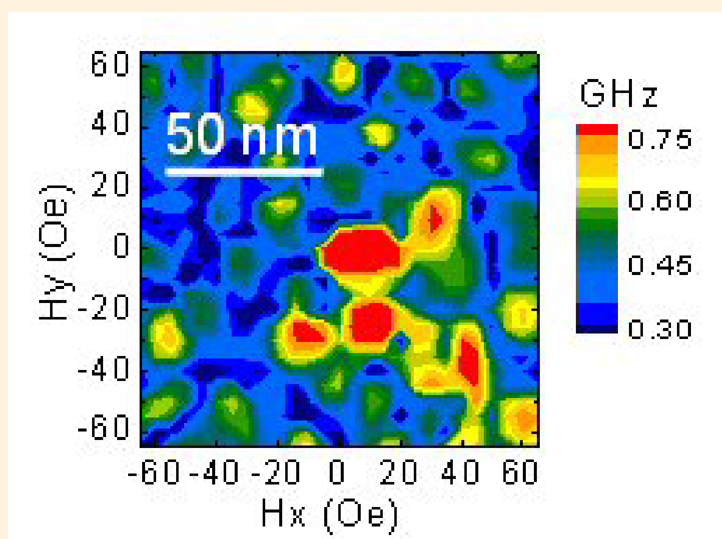


Paul Crowell, Associate Professor of Physics

Magnetism is a familiar aspect of many technologies, including motors, sensors, and storage media. The tools of nanofabrication provide us with the opportunity to study how magnetic properties are influenced by the size of a sample or the presence of interfaces between magnetic and non-magnetic materials. For example, in work carried out in collaboration with Professor Chris Palmstrøm's group, graduate students Xiaohua Lou, Eric Garlid, and Jianjie Zhang, along with undergraduate David Toyli and visiting scientist Tsuyoshi Kondo, have been exploring the properties of new electronic devices in which ferromagnetic materials are integrated with the semiconductor gallium arsenide. It is possible to inject spin-polarized electrons (the carriers of magnetic information) from a source electrode into a semiconductor channel, in which the electron spin can be modulated by a magnetic field or, in future devices, an electrostatic gate. In collaboration with Professor Chris Leighton, postdoctoral associate Jeff Parker and graduate students Mun Chan and Michael Erickson have been investigating how spin-polarized electrons are transported from a ferromagnetic material such as cobalt into a non-magnetic metal such as copper. Graduate student Robert Compton and undergraduate David Christle have been investigating the properties of particles and arrays patterned from thin films of permalloy (an alloy of iron and nickel) using electron beam lithography. In disks with thicknesses on the order of 10 – 50 nanometers and diameters of two microns or less, the magnetic ground state is a vortex, in which the magnetization circulates around the disk. The core of this vortex consists of a region which behaves like a miniature bar magnet with a diameter of approximately 10 nm. In the presence of time-dependent magnetic fields, the vortex core can oscillate with a resonant frequency on the order of several hundred MHz. Using time-resolved optical techniques, the vortex dynamics on time scales of 10 nanoseconds and less can be used to probe material properties on length scales of a few nanometers. In effect, the vortex core itself functions as a scanning probe microscope. See R.L. Compton and P.A. Crowell, *Physical Review Letters* **97**, 137202 (2006).



Schematic of a time-resolved experiment on a magnetic vortex. The sample is patterned by electron beam lithography and sits on the center conductor of a coplanar waveguide.



Map of magnetic vortex dynamics in a thin-film permalloy disk. Note that the vortex frequency fluctuates on length scales on the order of 10 nanometers or less.

PARTICLE TECHNOLOGY LAB NEWS

PTL DIRECTOR'S MESSAGE

Distinguished McKnight University Professor, David Y.H. Pui

One current issue of concern to the Nanoparticles/Nanomaterials community is managing environmental, health and safety risks of the nano-products. The challenges include the lack of information on the risks and the need on oversight. In October 2005, we hosted the 2nd International Symposium on Occupational Health in Minneapolis. I am pleased to announce that Springer will publish a book this month on Nanoparticles and Occupational Health (Andrew Maynard and David Y. H. Pui, Editors), see : http://www.springer.com/978-1-4020-5858-5. The on-line version of the book, also a Special Issue of the Journal of Nanoparticle Research, is currently available free of charge. You can view the contents of the book and download a pdf copy by visiting the following website: http://www.springerlink.com/content/p13817kll818/. We invited about sixteen papers presented at the 2nd International Symposium to publish a coherent book on the topics. The book includes topics on the measurement of airborne nanostructured particles and exposure control, and provides new perspectives on nanotechnology and its potential impact within society.

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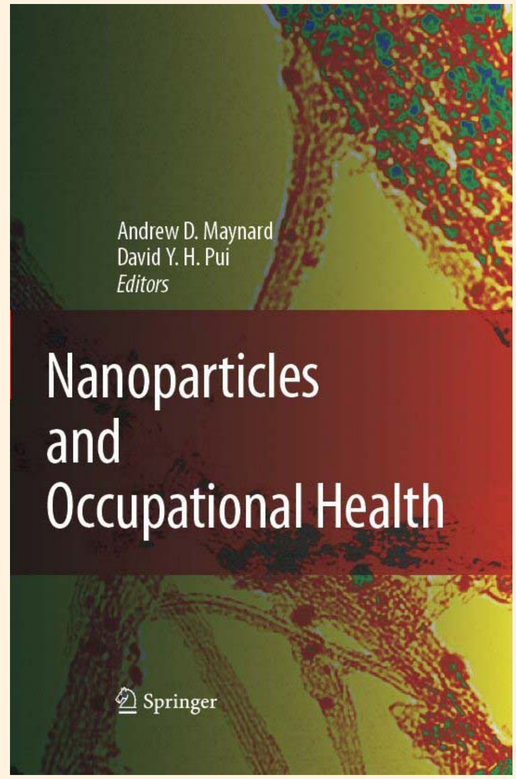

"Workers are society's canaries-in-the-coal mines when it comes to the environmental, health and safety effects of new materials—and nanoscale materials are no different," said Andrew Maynard, chief scientist of the Woodrow Wilson Center's Project on Emerging Nanotechnologies.

"The good news is that international concern over how to ensure safe nanotech workplaces has resulted in some progress. The bad news is that critical questions about worker safety—and about broader environmental, human health and safety issues—remain unanswered," stated Maynard.

The 1st International Symposium was held in the U.K., and the 2nd Symposium was held in the U.S. It is very appropriate that the 3rd International Symposium will be held in Taipei, Taiwan, August 29 to September 1, 2007. The Symposium will be hosted by Taiwan's National Science and Technology Program for Nanoscience and Nanotechnology and the National Chiao Tung University (see: http://nano-taiwan.sinica.edu.tw/EHS2007/index.htm). The organizers expect the Symposium will draw 500 registrants, many from Taiwan, Korea, Japan and China. A Call for Abstracts announcement was sent with a due date for abstracts of March 15, 2007. We invite NNIN users from academia and industry to attend this important symposium. For sponsorship information, please contact Symposium Co-Chair Professor Chuen-Jinn Tsai at cjtsai@mail.nctu.edu.tw.

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David Y.H. Pui, Director
Mark Stolzenburg, Lab Manager

PTL Featured User and Research

Vacuum Test Facility to study Nanoparticle Behavior below 20 mTorr

Under Intel sponsorship the last three years, the Particle Technology Laboratory has developed a vacuum test chamber capable of investigating particle behavior at low pressure down to 10 mTorr. The chamber is also equipped with a particle injection system that will allow e.g. NIST traceable nanoparticles in the range of 10 nm to 300 nm to be introduced into the chamber with known speed. Particles can be injected either horizontally or vertically. A witness wafer can be installed inside the chamber. After experiments, the wafer can be checked for number and pattern of deposited particles, using a wafer surface scanner. The facility has been documented in several papers (e.g. Kim, J. H.et al. (2006) *J. Vac. Sci. Technol. A* **24(2):**229-234.; Asbach, C.et al. (2005) *Appl. Phys. Let.* **87:**234111). The vacuum chamber (see picture) is installed in the Nanofabrication Center in the NNIN facility side by side with a wafer surface scanner. We welcome potential clients from industry and academia to make use of this facility.

We are pleased that two well known groups will be using this facility in the coming months. IUTA (Institute of Energy and Environmental Technology) in Duisburg, Germany has received a grant from the German Science Foundation (DFG) to study diffusional deposition of nanoparticles under low pressure. The project PI is Dr. Christof Asbach and the project title is "Analytical and statistical modeling of nanoparticle contamination of critical surfaces under low pressure conditions". The objective of the project is to analytically model the diffusional particle deposition on critical surfaces at low pressure under the influence of gravity and thermophoresis. The goal is to predict the risk of nanoparticle contamination of sensitive equipment as e.g. used in semiconductor manufacturing.

Sandia National Laboratory is working to develop a statistical, Langevin particle tracking algorithm that can predict nanoparticle transport (including inertia, thermophoretic, gravitational and diffusional effects) under low-pressure conditions. Ultimately, this particle-tracking algorithm will be coupled to the low-pressure, noncontinuum gas flow fields calculated by the Direct Simulation Monte Carlo (DSMC) method. The project PI is Dr. Michael Gallis, a well known expert in the application of the DSMC method to microsystem and low-pressure flows. The objective of the project is to provide accurate numerical predictions of nanoparticle transport and deposition in low-pressure systems which can be used to verify analytic models.

Both IUTA and Sandia groups will make use of the PTL Vacuum Test Facility to provide definitive experimental results to validate their models. They will inject particles with known speed, size, and material. This will allow them to precisely predict the particle stopping distance and thus the point where the inertial particle motion will come to rest and diffusional will become the dominant source of motion.

The PTL Vacuum Test Facility is set up inside the cleanroom of the Nanofabrication Center. PSL and SiO2 nanoparticles are injected into the Vacuum Chamber at below 20 mTorr with known speed and known particle size. Various protection schemes are evaluated to prevent nanoparticle contamination in EUVL (Extreme Ultraviolet Lithography) Systems.

The IT Characterization Facility mission relates directly to the core teaching, research and outreach missions of the University.

· Provide centrally accessible materials characterization instrumentation for researchers, maintained and upgraded by experts.
· Build, preserve and upgrade the knowledge and skills required for the optimal operation and research capability of the instrumentation.
· Teach University researchers to apply this instrumentation, knowledge and skills most fruitfully.
· Make the instrumentation, knowledge, skills and training available to entities external to the University of Minnesota, to a degree that does not detract from the preceding mission clauses.

The Nanofabrication Center (NFC) is an interdisciplinary facility supporting faculty and industrial research within the Institute of Technology to foster education, research and industrial collaboration in microelectronics and other related research involving nanofabrication. Capabilities include:

- E-Beam, Ion Beam & Mask Making
- Optical Photolithography
- Chemical Vapor Deposition
- Metalization & Sputtering
- Dry Etching
- Annealing, Oxidation & Doping
- Wafer Bonding, Polishing & Sawing

The Particle Technology Laboratory mission is to foster research and educate students and the greater community in the areas of aerosol and small particle research and instrumentation. Major facilities of the laboratory include:

- Cleanroom, wind tunnel, filter testing, plasma and vacuum facilities.
- Instruments for particle generation, measurement, sampling and analysis in the 0.002 to 100 um diameter range.
- Gas-, liquid- and surface-borne particle measurement instrumentation
- Single-particle mass spectrometer
- Bioaerosol sampling and measurement capabilities

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