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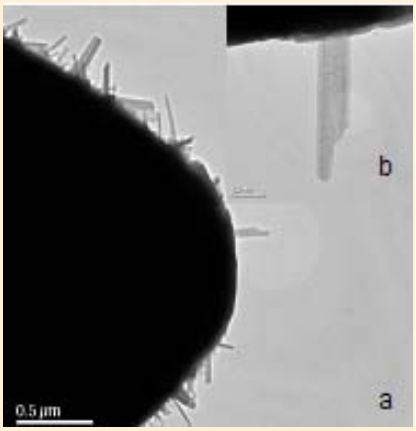
UNIVERSITY OF MINNESOTA

NANOTECHNOLOGY NEWS FROM THE UNIVERSITY OF MINNESOTA

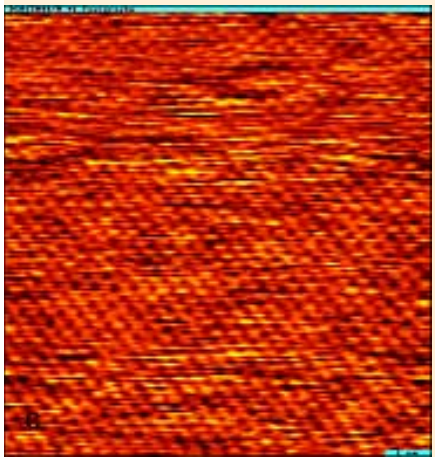
ISSUE FOUR FALL 2005

FALL 2005 NANO IMAGE

Professor Philippe Bühlmann, in the Department of Chemistry at the University of Minnesota, is developing functionalized carbon nanotube tips for Scanning Tunneling Microscopy with chemical selectivity.



Pictured at right are (a) TEM of Ru-plated carbon nanotube tip after use as STM probe. (b) (Inset) Close up of multi-walled nanotube at tip apex.



Pictured at left is an STM image of highly-oriented pyrolytic graphite.

For those of you engaged in teaching or research, welcome to the 2005/2006 academic year. I hope that this proves to be an enjoyable and productive year for you. This fall there is a new University initiative that may impact the long-term direction for our Labs, the Strategic Positioning Task Force process established by President Bruininks. Twenty committees will meet over the coming year and will issue reports designed to help position the University to become one of the top three public research institutions in the country. I have been asked by Vice-President Mulcahy to co-chair the task force most relevant to our labs: Research Infrastructure at the University. Members from across the University will discuss how best to structure our research facilities to provide faculty a competitive advantage over the next few decades. I would encourage you to follow the progress of the process (http://www1.umn.edu/systemwide/strategic_positioning/) and let me know if you have any insights to share on this critically important project.

The University will host two very interesting nano meetings this fall. In September the Center for Science, Technology & Public Policy at the Humphrey Institute will sponsor a workshop to explore and evaluate models for the oversight of nanotechnology, with a focus on nanoparticles that are used in or derived from biological systems. For more information see: www.hhh.umn.edu/centers/stpp/nanotechnology.html. The University is also hosting the 2nd Annual International Symposium on Nanotechnology and Occupational Health (www.cce.umn.edu/conferences/nanotechnology/) in October. As always, consult the nano website: www.nano.umn.edu for the latest information on upcoming events.

Best regards,
Steve Campbell

2 - 3 *Characterization Facility*

4 - 5 *NanoFabrication Center*

6 - 7 *Particle Technology Laboratory*

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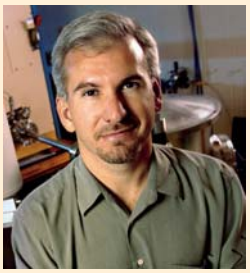
Characterization Facility News

CharFac Director's Message



CharFac Director, Greg Haugstad

Following another busy summer with many graduate students and visitors receiving training, we anticipate new interactions during the school year. There are special educational activities during the fall semester as listed under our Upcoming Events section on the following page. And we are particularly excited to announce *remote control* systems as detailed in our New Capabilities section. These will allow remote operation of scanning electron and scanning probe microscopes. Such usage would more fully book instrument time, especially during the "off season" of November through February, while at the same time fulfill an important NNIN function. Educational roles at core facilities are increasingly important in peer review of grant proposals for instrumentation.

CharFac at the University of Minnesota

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Greg Haugstad, Director
Mike Boucher, Lab Manager

New Capabilities

The JEOL 6500 and 6700 scanning electron microscopes have been configured with "WebSEM" remote control software. The 6700 is a cold field-emission gun SEM equipped for secondary electron imaging with an ultimate resolution of 1.0 nm, and a magnification range of 10x to 700,000x. The 6500 has a thermally assisted field-emission gun with an ultimate resolution of 1.5 nm, and modes including secondary and backscattered electron imaging, EDS, EBSD and cathodoluminescence.

WebSEM enables SEM images and major control functions to be displayed on a web browser of a remote client PC. The live image is 640x480 pixels, and high-quality images (1280x1024 pixels) can be saved on the SEM control computer then automatically transferred to the remote PC. The friendly graphical user interface allows access to most microscope control functions through click and drag operations. The software can be downloaded from the CharFac server. Adding free remote desktop software such as "UltraVNC" enables remote users to control other software (e.g., for elemental analysis). WebSEM currently requires Windows NT, 2000 or XP on the remote client PC.

The Molecular Imaging PicoPlus scanning probe microscope is now available for remote operation via Windows XP's Remote Desktop Connection utility. This allows the remote user to take over the SPM control computer. All windows on the control computer can be viewed and manipulated on the remote user's computer desktop. The view from the optical positioning microscope also has a dedicated window and can be captured digitally. Transfer of captured data is fast and simple using the freeware Filezilla. PicoPlus has a 100x100-micron lateral scan range within which the remote user can control all procedures. All conventional AFM imaging modes are available, plus special modes such as digital pulsed force, magnetic AC and current sensing.

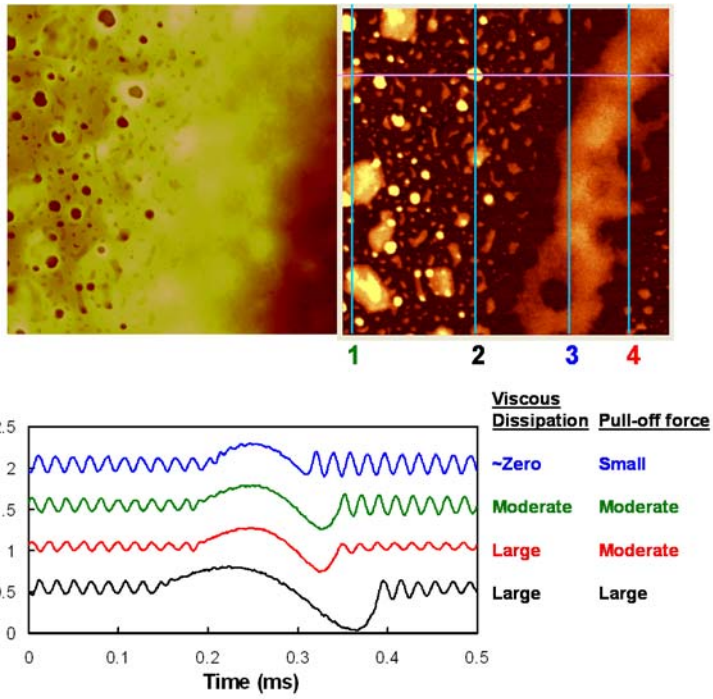
Coarse SPM sample positioning will be performed locally by a staff person, together with instrument setup. Planned additions include (i) a motorized specimen stage controlled from within Windows XP to allow not only coarse positioning but also switching among specimens; (ii) a closed-loop scanner upgrade, to remove the wait time for creep that is unavoidable with conventional (open-loop) AFM piezoscanners following a change of magnification or translation of image center.

CharFac Featured User and Research

Coatings for Drug Delivery: New AFM Characterization Methods

Greg Haugstad, Characterization Facility, University of Minnesota, Minneapolis, MN
Klaus Wormuth, Surmodics, Eden Prairie, MN

Combining the physical action of medical stents with the chemical action of drug eluting coatings is advancing the treatment of cardiovascular disease. A stent is a cylindrical mesh inserted into clogged coronary arteries and expanded via balloon angioplasty. An elastomeric coating encases the metal struts of the stent and includes drug distributed for controlled release. Imaging polymer/drug morphology, down to the nanometer scale, is essential to coating development. Atomic force microscopy (AFM) can contrast copolymers and crystalline organic phases via the interaction with the AFM tip. A special method now available on the CharFac's PicoPlus AFM, Digital Pulsed Force Mode, can isolate sources of material contrast. Tip-sample interaction force is measured during approach-retract cycles (one cycle per pixel location, e.g. 2.5 GB data file) via the deflection of the cantilever to which the tip is attached. The contrast of deflection data from different surface locations, as derived from *specific time intervals* within each cycle, can reflect differences in known properties: modulus, surface energy, viscous character, etc. Figure 1 compares tip-sample adhesion in terms of (i) the largest pulling force sensed during retraction and (ii) the amount of viscous dissipation during the break of tip-sample contact as gauged by the amplitude of the high-frequency rattling of the "diving-board" cantilever compared to its maximum downward deflection. In a larger body of work, known differences in the above properties, along with control studies on drugless coatings, enabled the identification of components.



(Top left) Surface topography image (5x5 micron, brighter = higher); **(Top right)** Corresponding pull-off force image. Brighter regions are exposed patches of different phases of poly (butyl methacrylate)-co-poly (laurel methacrylate); dark regions are the crystalline drug dexamethasone. **(Bottom)** Approach-retract cycles (time domain, shifted vertically for clarity) at four representative locations (crossing points of horizontal and vertical lines in top right image). Resonant oscillations of the cantilever are excited following (i) the break of a previous contact (negative time) and (ii) the displayed contact interval (0.15-0.35 ms). Curvature of data trends during contact is due to *sinusoidal* cycling of distance at 2 kHz. Maximum deflection relative to baseline (averaged over 0.1 ms) is the control parameter for imaging.

This research project resides within the Nanostructural Materials and Processes program of the University of Minnesota's Industrial Partnership for Research in Interfacial and Materials Engineering (IPRIME). IPRIME creates opportunities for professionals in industry to collaborate with researchers at the University.

Upcoming Events

Structural Characterization Lab, a course for materials science juniors, will make use of several CharFac labs. Students are trained to use each instrument during a 3-hour session, and then perform independent lab exercises during another session. Physical Principles of Thin Film Technology, a graduate course in electrical engineering, includes demos and data acquisition on student-grown films. Together these two courses have used X-ray diffraction, FTIR spectroscopy, scanning electron microscopy, transmission electron microscopy, scanning probe microscopy, nanoindentation, stylus profilometry and light microscopy.

A late-autumn **workshop on advanced scanning probe microscopy** (SPM), co-sponsored by SPM vendor Molecular Imaging, will be held in our Nils Hasselmo Hall facility, the second workshop of 2005 (and recurring twice annually). There will be particular emphasis on biological and biomedical soft materials, along with special SPM methods for such systems. Molecular Imaging participates as a member company of IPRIME. For more information contact Bill Domansky (bdomansky@btsbio.com) or Greg Haugstad (haugs001@umn.edu).

NanoFabrication Center News

Process Equipment Improvements

Deposition of high quality thin films is an important aspect of technologies such as MEMS. One of the best techniques for deposition of silicon nitride thin films is low pressure chemical vapor deposition (LPCVD). Our current LPCVD system has the capability to deposit silicon nitride, polysilicon, and low temperature silicon dioxide on substrates up to 4 inches in diameter. We routinely deposit two types of silicon nitride, standard stoichiometric silicon nitride, and low stress (silicon-rich) silicon nitride. The low stress film is particularly desirable for MEMS applications requiring free standing beams or membranes. The system we have been using for the past 15 years is now approximately 25 years old, and a major upgrade is in progress. We are improving the computer control system by moving to a Sun Microsystems-based workstation software package from SEMY Engineering. Also, we are bringing up a fourth deposition tube in the system, this one set up to deposit silicon nitride on substrates up to 6 inches in diameter. This new tube gives us two nitride deposition tubes, a significant increase in redundancy for these maintenance-intensive systems. We are upgrading the vacuum systems on the LPCVD to more reliable dry pumps. We expect to have the upgraded LPCVD back in operation in September.

NFC has also recently installed a YES 310 image reversal oven. This oven uses an ammonia atmosphere to change the property of positive resist such that instead of being removed during development, the resist remains. This well known technique of image reversal can be a valuable tool for lithographic processing.

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Steve Campbell, Director
Greg Cibuzar, Lab Manager

NFC Director's Message

NFC Director, Steve Campbell

This fall sees the addition of several new groups using the NanoFabrication Center. Several out of state companies have been drawn to NFC recently for carrying out their product development. One routinely commutes from the west coast to use the facilities. All of the new companies are engaged in the microfabrication of micro mechanical devices of one sort or another. New faculty this fall for the lab include Professor Daniel Bond from the Biotechnology Institute on the St. Paul campus. Dan's exciting research work is profiled on the following page. Also joining us is Professor Eusik Yoon from the ECE Department. Eusik, who previously held a faculty position in Korea at KAIST, works in the fields of biomedical and RF MEMS. Look for his research to be featured in an upcoming issue. Professor Sang-Hyun Oh, who is also active in biomedical MEMS will join the ECE Department next spring.

The addition of faculty in this area is no coincidence. Clearly there are tremendous opportunities on the horizon provided by the combination of biological or biomedical sciences and micro and nano engineering. You can read more about this at the NIH website: http://nihroadmap.nih.gov/nanomedicine/. Over the long term, we expect more activity in this area as other new faculty and companies arrive and as existing users discover research opportunities in this area. The challenges for the Lab include 1) how to continue to support our users who are active in more traditional areas while we facilitate this exciting new research topic, and 2) how to help people make connections across this technical divide.

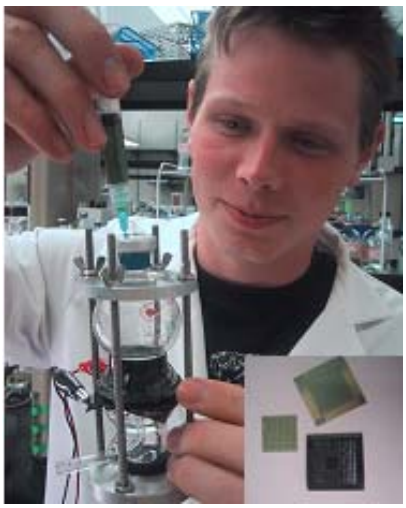
NFC Featured User and Research

Miniaturization of Microbial Fuel Cells

Professor Daniel R. Bond, Biotechnology Institute and Department of Microbiology
University of Minnesota, Room 140 Gortner Labs, Saint Paul Campus

Bacteria have been recently discovered with the ability to use electrodes as electron acceptors. The process is analogous to a traditional fuel cell, with bacteria acting as catalysts for oxidation of a fuel. No device exists that allows multiple microbial electrode growth experiments to be conducted in parallel, or with a significant degree of control over the local environment. In addition, no equipment exists to conduct experiments using materials able to utilize this phenomenon in a biosensor. We have therefore initiated the use of microfabrication as a method for constructing devices better able to study this process.

SEM of *Geobacter metallireducens* producing electricity on an electrode surface, using benzoate as fuel



Undergraduate Benjamin Hunt has been responsible for all design and microfabrication.

We use gold-coated, etched wafers allowing thousands of micron-scale features for microbial attachment, allowing current microbial fuel cell designs to be two orders of magnitude smaller than previous instruments. The final designs should allow us to mimic multi-well format of microtiter plates (e.g., 96-well plates) with microfabricated electrodes at the base of each well. The ultimate goal of this line of investigation is the ability to monitor current production by microcolonies, or even individual cells. Wafers etched with microchannels are also being investigated as electrodes for a flow-through sensor able to sense metabolite levels in biotechnology fermentations. These devices represent the first demonstrations of bacteria interfaced with microelectrical devices, without use of soluble mediator compounds.

Upcoming Events

CMP Training

The Strasbaugh 6EC chemical mechanical polishing system for oxide materials is now operational. Contact us at nfc@umn.edu for more information.

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 at 10AM. The training includes watching our safety video and taking a brief quiz. Also, an NFC staff member provides a tour showing some 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 2 hours to complete, and must be done before users will be granted access to NFC facilities.

PARTICLE TECHNOLOGY LAB NEWS

PARTICLE SIZE SPECTROMETRY IN LIQUID

In recent months a number of potential clients have expressed an interest in measuring particles in liquid suspensions. In response to this the Particle Technology Laboratory is in the process of enhancing its capability for detection and sizing of liquidborne particles.

Three different liquidborne particle counters will soon be available for use at the UM Particle Technology Laboratory: a Sub-Micron Particle Analyzer (SMPA, Coulter Model N4SD), a Laser Liquid Particle Spectrometer (LLPS, PMS Model LLPS-X) and a Liquidborne Particle Detection System (LPDS, TSI Model 7750).

The SMPA can size particles in liquid suspension inside a cuvette in a size range from 3 nm to 3 µm. The measurement principle is based on the size dependent Brownian motion of the particles within the liquid suspension. Changes in the patterns of scattered light from the particles are analyzed by the SMPA to determine the size distribution of the particles in the suspension.

The LLPS uses Mie scattering of particles in a liquid flow to determine size and concentration of suspended particles in the 0.1 µm to 100 µm size range. The particle size is measured based on the intensity of light scattered by a particle in a laser beam at a certain angle.

The LPDS measures particles in a size range from 0.19 µm to 0.48 µm (50% detection limit at 0.2 µm) in a liquid flow. Particles are distinguished from micro bubbles in the fluid using a dual-beam interferometer. See www.me.umn.edu/labs/ptl/equipment.html for more information.

PARTICLE TECHNOLOGY LAB AT THE UNIVERSITY OF MINNESOTA

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

PTL DIRECTOR'S MESSAGE

Distinguished McKnight University Professor, David Y.H. Pui

We just completed another successful Aerosol and Particle Measurement Short Course. This year, the 30th offering of the Short Course attracted an all-time high of 78 registrants from industry, government and academia. Most of them indicated an interest in our newly added curricula on Nanoparticle Technology, and Bioaerosol Sampling and Measurement. The Short Course has had more than 1,700 registrants in 30 offerings. It demonstrates that aerosol is a discipline with wide practical applications both in industrial and environmental fields, i.e. an enabling discipline on which many other fields depend.

In the next few weeks, two more events, a workshop and a symposium, will be held at the University of Minnesota to address nanotechnology issues. The first is a Workshop to be held September 15 on the topic of "The Nanotechnology-Biology Interface: Exploring Models for Oversight," (www.hhh.umn.edu/centers/stpp/nanotechnology.html). The workshop will address the emerging concerns about the health and environmental safety of nanoparticles applied to, or derived from, biological systems. The second is the Nanotechnology and Occupational Health Symposium (www.cce.umn.edu/nanotechnology) that will be held October 3-6. It will provide a multi-stakeholder forum for presenting, assimilating and discussing the latest breakthroughs and activities in addressing nanotechnology and worker safety and health. We have already received a large number of registrations for both events. Please join us in this unique opportunity to be part of the global effort working towards responsible nanotechnology.

PTL Featured User and Research

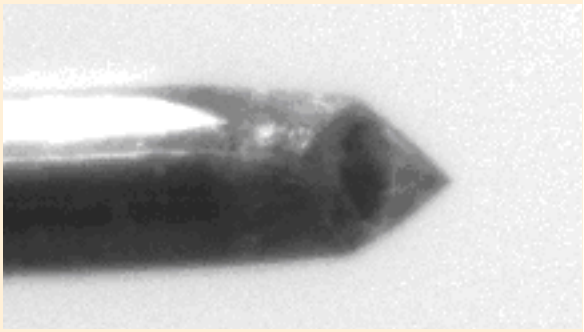
Development of Airborne Nanoparticles for Targeted Drug Delivery

Powerscope, Inc., Minneapolis, Minnesota

Drug nanoparticles are promising in targeted delivery of drugs to body parts that are not accessible with the conventional means of drug delivery. These include the interior of the eye, the back of the eye and the deep lung. Certain drugs that are currently not approved by the FDA due to their toxic side effects may become acceptable when delivered to their specific targets in small amounts in the form of nanoparticles. Mitomycin C is one such drug. In-vitro experiments show that this drug is very potent in inhibiting the growth of scar tissues in the human retina and hence very desirable for patients who undergo retinal reattachment surgery and have a tendency to grow scar tissues. However, benefits of Mitomycin C are plagued by its side-effects when delivered in a conventional manner. Nanoparticles of this drug offer a hope to eliminate the undesirable side effects. Drug nanoparticles also appear promising in treating cancer of the deep lung using an inhalation therapy.

Amir Naqwi, Chris Fandry and Gerry Kauma at Powerscope Incorporated, a company based in Dinkytown, are developing technologies to generate airborne drug nanoparticles for the above applications. They are working in collaboration with Profs. Tim Wiedmann and Tim Olsen of the Pharmaceutics and Ophthalmology Departments, respectively, at the University of Minnesota. Powerscope is investigating the use of electrospraying and high-frequency ultrasonics to generate particles smaller than 100 nm.

PTL's electromobility based nanoparticle sizer is ideally suited for characterization of these airborne particles. Particle size measurements have helped to optimize the liquid conductivity in electrosprays of interest. Electrospraying technique uses an intense electric field to form a liquid cone at the tip of a capillary (shown at right) through which a conductive solution is pumped. An extremely fine liquid jet issuing from the vertex of the cone breaks into tiny drops which dry to form nanoparticles.



With the support from PTL, Powerscope and its university partners are proceeding with animal experiments to examine the performance of drug nanoparticles.

Powerscope also plans to examine the effectiveness of its particle generators in producing airborne suspensions of macromolecules.

Upcoming Events

September 15, 2005
The Nanotechnology-Biology Interface: Exploring Models for Oversight
A Workshop at the U of M Humphrey Institute
Minneapolis, Minnesota
www.hhh.umn.edu/centers/stpp/nanotechnology.html

October 3-6, 2005
Second International Symposium on Nanotechnology & Occupational Health
Radisson Hotel Metrodome
Minneapolis, Minnesota
www.cce.umn.edu/nanotechnology/

October 17-21, 2005
24th Annual AAAR Conference
Hilton Austin Hotel
Austin, Texas
www.aaar.org/05AnnualConf/index.htm

October 20-21, 2005
PCIH 2005-The Summit for New Opportunities
20th Annual Professional Conference on Industrial Hygiene
Denver Marriott City Center
Denver, Colorado
www.aiha.org/pcih/htm

October 24-25, 2005
Frontiers in Aerosol Dosimetry Research
The Beckman Center of the National Academies
Irvine, California
For more information please e-mail: rfphalen@uci.edu

Dec 13-16, 2005
4th Asian Aerosol Conference
Grand Hyatt
Bombay, India
www.iasta-aac.org

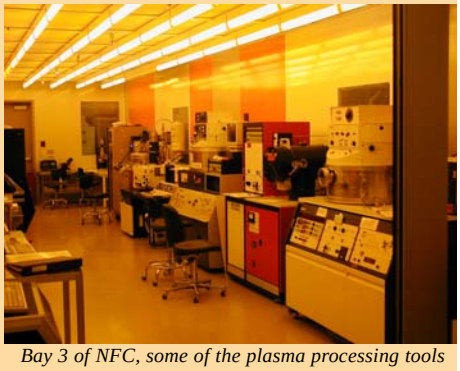
The IT Characterization Facility mission relates directly to the core teaching, research and outreach missions of the University

- Provide centrally accessible materials characterization instrumentation for University 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 the above 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 JEOL 6500 FEG-SEM at CharFac

The NanoFabrication Center's goal is to provide reliable access to tools that enable the research needs of its user base at as low a cost as possible.

The NanoFabrication Center (NFC), a research lab on the Minneapolis campus of the University of Minnesota, is an interdisciplinary facility that supports faculty and industrial research within the Institute of Technology to support education, research and industrial collaboration in microelectronics and other related research involving nanofabrication.



Bay 3 of NFC, some of the plasma processing tools in the cleanroom

The Particle Technology Laboratory mission is to foster research and educate students and the greater community in the following areas:

- Fundamental Aerosol Research and Instrumentation
- Engineered and Environmental Nanoparticles
- Air, Gas and Liquid Filtration
- Cleanrooms and Microcontamination Control
- Air Pollution and Environmental Studies
- Ventilation and Bioaerosols Studies
- Materials Synthesis in Reacting Flows

Sampling platform for jet engine exhaust aerosol characterization experiment

Nanotechnology News from the University of Minnesota
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and the National Nanotechnology Infrastructure Network.
Comments and suggestions are welcome! Would you like to be added to or removed from our distribution?
Contact: Becky Von Dissen at vondi001@umn.edu or 612-625-3069

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