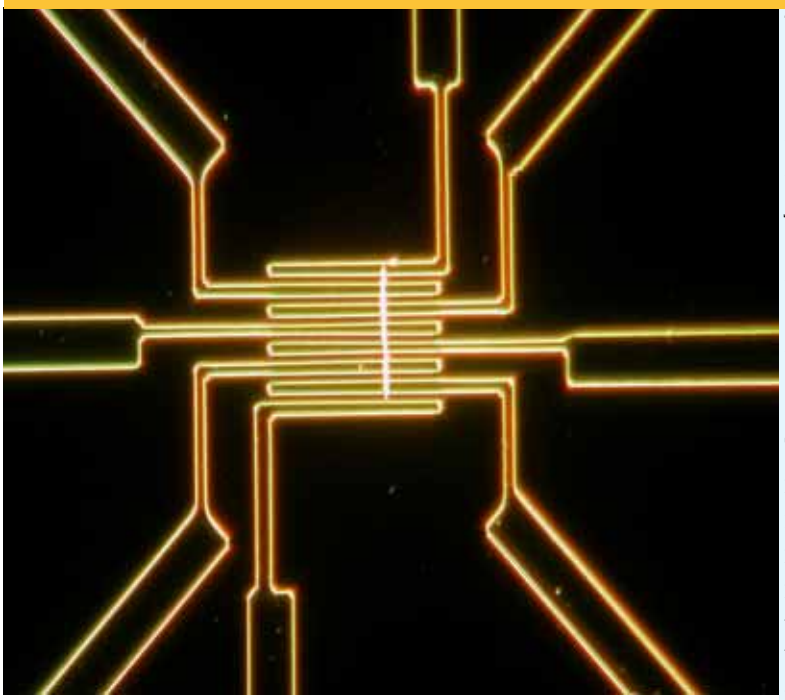
Nanotechnology News from the University of Minnesota

Issue One Fall 2004



Beth Stadler's group in Electrical and Computer Engineering is making magnetoresistive nanowires. This image was taken using dark field optical microscopy, a technique that causes the edges of conductive materials to light up. The nanowire, which has a high surface to volume ratio, is easily resolved as it lies on top of patterned electrodes. Using these electrodes, the resistance of individual nanowires can be measured in a variety of magnetic fields. Although the nanowires are grown in arrays, which will be useful for many applications, individual measurements are critical at the research phase of this project.

Welcome to the first of our planned quarterly nanotechnology newsletters. They will cover news and events from the Characterization Facility (CharFac), the NanoFabrication Center (NFC), and the Particle Technology Lab (PTL). Publication of the newsletter is being supported jointly through the recently established Nano Coordinating Office and the National Nanotechnology Infrastructure Network (NNIN).

NNIN is a network of university laboratories around the country that receive a subsidy from the National Science Foundation to act as open facilities for carrying out nano work. The NSF funding has enabled the labs to have minimal or no rate increases for FY05. We hope to develop the Minnesota node into a regional center of excellence for nano, and will recruit users from other schools in the region. PTL is responding to NNIN by moving toward more of an open lab, with a regular rate structure for equipment and facilities access. We hope that it will serve as a national resource for aerosol research. If you use any of the NNIN subsidized labs for your work, we would ask that you use the following acknowledgement: Partial support of this work was provided by NSF through NNIN (ECS #0335765).

The Nano Coordinating Office was just established by joint funding from Vice President of Research David Hamilton and IT Dean Ted Davis. The office is located in EE/CS room 1-263. The office will develop an improved web presence for nano activities and will organize nano related activities at the University. You can find us at www.nano.umn.edu. An early version should be active by the time that you receive this newsletter. Stop by to learn about an upcoming workshop on nanomedicine that the NCO will be hosting here at the University.

In this issue

2-3 *Characterization Facility*

4-5 *NanoFabrication Center*

6-7 *Particle Technology Lab*

CharFac acquires two new transmission electron microscopes

This spring the CharFac installed new Technai F30 and T12 transmission electron microscopes.

The F30 is a scanning TEM with the following special features and capabilities: Schottky field-emission gun, high-resolution and atomic imaging, X-ray energy-dispersive spectroscopy (XEDS), beam scanning, spectrum imaging using XEDS, high-angle annular dark-field (HAADF) or Z-contrast imaging, low-dose imaging, focal–series acquisition, and remote control (stage control, alignment, focus, aperture control, etc...). This instrument represents a significant advance in nanoscale characterization capabilities at the University of Minnesota and will benefit dozens of principal investigators who come from ten different departments. A special control room was constructed in place of a darkroom, to enable the highest performance (removing sources of thermal drift, i.e. human bodies and computer monitors).

The T12 is optimal for training new users, less demanding applications, and "user practice" leading to F30 applications.

A new TEM user group is recommending and implementing protocols designed for optimal usage, oversight and maintenance of our new TEM capabilities.

Staff News

Staff member **Dr. Yongqiang Wang** took a position at Los Alamos National Laboratory in ion beam analysis (IBA). Ongoing IBA staffing in the CharFac is being provided by a new combination of staff members with complementary expertise: **John Kadlec**, **Greg Haugstad** and **John Thomas** as well as part-time students. Dr. Thomas is a 3M retiree who is helping to upgrade older systems in several laboratories. Additional technicians will soon be added in electron microscopy areas in both our Shepherd Labs and BSBE facilities, funded in part by the NNIN. In X-ray scattering, **David Carr** spent an interval working in the CharFac at the end of his Ph.D. work (see feature, page 3) but took a position at the local analytical company Evans Phi. More recently **Ryan Wold** finished his M.S. program and joined the CharFac in wide-angle X-ray scattering.

Characterization Facility at the University of Minnesota

12 Shepherd Labs
100 Union Street SE
Minneapolis, MN 55455

www.charfac.umn.edu
Main telephone: 612-626-7594
Fax: 612-625-5368

Greg Haugstad, Director
Mike Boucher, Lab Manager

Welcome to the CharFac area of the newsletter. In FY04 the CharFac evolved considerably in the areas of training, staffing, rates and advisory groups.

Major new instrumentation was added in wide-angle X-ray scattering and transmission electron microscopy (TEM), exceeding $2M in value. A new Pananalytical X'pert high angular resolution diffractometer is now available and serving numerous users, including advanced applications in reflectometry and reciprocal space mapping (see feature on page 3). Another new X-ray diffractometer is being optimized to provide high brightness and high-speed sampling that can be programmatically acquired over a two dimensional spatial domain. Two new TEMs (see adjacent article) and ancillary equipment (ion thinner for specimen prep, plasma cleaner for specimen holders) make the CharFac state-of-the-art in many respects. A new sequence of one-credit graduate classes in electron microscopy has been developed by staff member Dr. Stuart McKernan, offered through the Department of Chemical Engineering and Materials Science. Classes consist of lecture and lab components including hands-on training.

Discounted rates on select instruments have been developed for qualified academic users, for use during evenings and weekends (upon qualification) and for sessions exceeding two hours in length. The motivation is to encourage more challenging, tedious and advanced applications. The discounts now apply to the TEMs, field emission SEMs, microtomy, small-angle X-ray scattering, and select wide-angle X-ray scattering systems. The evaluation of the discounts is ongoing and will likely result in some tweaking of the rate scheme.

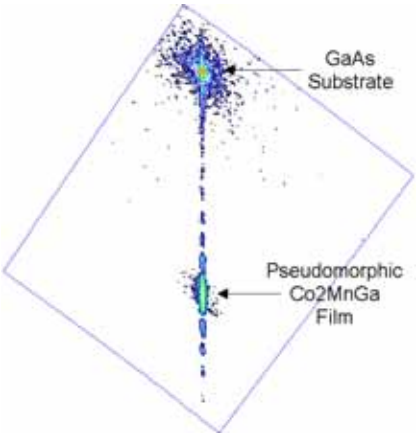
Many of the above developments in instrument acquisition and rate discounts have been in part driven by new advisory committees to the CharFac in electron microscopy and X-ray scattering. We will soon add a committee for surface/thin-film analysis to advise on IBA and electron spectroscopy.

CharFac Director, Greg Haugstad

CharFac Featured User and Research

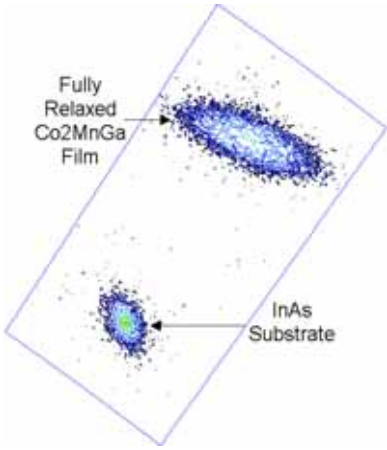
Reciprocal Space Mapping of Epitaxial Magnetic Metal Films on III-V Semiconductors
David Carr, Ph.D. July 2004, Department of Chemical Engineering and Materials Science
Advisor: Dr. Chris Palmstrøm

In the rapidly expanding world of nanotechnology, epitaxial magnetic metal films are growing in importance. The epitaxial growth process induces strain in the films which can dramatically alter the magnetic anisotropy. This research investigated the ability to control the magnetic anisotropy of 300 Å thick Co_2MnGa films grown on III-V semiconductors. Co_2MnGa belongs to a class of materials called Heusler alloys. These alloys have exhibited a remarkable ability to grow pseudomorphically (i.e. grow with the same in-plane lattice parameter as the substrate) to thicknesses much greater than normally found for other metals. By growing films on substrates ranging from GaP to InAs, the limits of the pseudomorphic growth and induced magnetic anisotropy were tested with strains varying between ± 5%.



The strain states in the films were examined using an advanced x-ray technique called Reciprocal Space Mapping (RSM) on the Panalytical X'Pert high resolution x-ray diffractometer in the Characterization Facility. A RSM can be used to determine if the film is fully strained (pseudomorphic), partially strained, or fully relaxed. These results were correlated with the magnetic properties to understand the epitaxial strain's effect on magnetic anisotropy. Example RSMs for the (224) lattice points of a pseudomorphic film on GaAs and a fully relaxed film on InAs are shown at right. The horizontal and vertical positions of the lattice points yield information about the in-plane and out-of-plane lattice parameters, respectively. The narrow width of the lattice point vertically aligned with the GaAs substrate shows a high quality pseudomorphic film. The vertical elongation and interference fringes are due to the small finite film thickness. This contrasts the broad lattice point for the fully relaxed film grown on InAs which shows a different in-plane lattice parameter from the substrate and a structurally less uniform film.

David Carr, Ph. D. of CharFac



The Co_2MnGa films were successfully grown pseudomorphically on GaAs and InP, with large perpendicular anisotropies induced by the epitaxial strains of ± 2%. For the film on GaAs, a perpendicular field of only 3000 Oe was needed to saturate the film, while 17,000 Oe were needed for the film grown on InP. These results clearly demonstrate the ability to engineer the magnetic anisotropy using epitaxial strain and Heusler alloys.

Upcoming CharFac Events

A short course on Electron Backscatter Diffraction (EBSD) will be held September 7–8, including morning lecture sessions and supervised hands-on learning sessions. The lectures will cover basics of the techniques as well as applications to specific materials science problems. The tentative outline includes the following topics: introduction, texture mapping, specimen preparation, texture measurement, phase identification, image processing, phase discrimination and data analysis. The hands-on labs will demonstrate the techniques covered in the lectures using both the Characterization Facility's SEMs and stand-alone computer simulators. The instructor is Dr. Scott Sitzman of HKL Technology. Enrollment is limited, so please respond as soon as possible to guarantee a space. To register for the workshop call 612-626-8953 or e-mail cbcadmin@cems.umn.edu. A fee of $200 will be charged to cover expenses.

Fall semester course Structural Characterization Lab (MatS 3801, typically juniors) will again make use of several CharFac labs including scanning electron and atomic force microscopy, X-ray diffraction and infrared spectroscopy.

Training courses in all techniques are scheduled ad hoc year-round, requiring a research account (internal users) or purchase order (external users). Call 612-626-7594 or e-mail charfac@umn.edu.

NFC Implements New Lab Automation Software

In July, NFC rolled out a new laboratory automation system called Coral. This is the system with which users of NFC facilities interface to perform tasks such as turning equipment on and off, making equipment usage reservations, checking on the status of processing equipment, and reporting equipment or processing issues. Administrative reports of equipment usage and charging are also available. Coral has been under development at Stanford and MIT for several years, and NFC is one of the first new installations of the software. The implementation of Coral at NFC has been a two year process involving personnel from NFC, Stanford, MIT and system administrators from the University of Minnesota's Office of Information Technology.

Compared to our old lab automation system, Coral has several advantages:

- More robust database (Oracle)
- Ability to hardware lock equipment to prevent unauthorized usage
- Supported by programmers at Stanford and MIT
- Improved performance and reliability
- Integrated reporting functions

Coral is used at the Stanford Nanofabrication Facility (SNF), and is also scheduled to be implemented at the Cornell Nanofabrication Facility (CNF) in the future.

Also...

NFC now has a facilities write up that you can use or adapt for your proposals. Download it as a Word or PDF file from www.nfc.umn.edu.

NanoFabrication Center at the University of Minnesota

1-165 Electrical Engineering/Computer Science
200 Union Street SE
Minneapolis, MN 55455

www.nfc.umn.edu
Main telephone: 612-624-8005
Fax: 612-625-5012

Steve Campbell, Director
Greg Cibuzar, Lab Manager

Welcome to the NFC area of the newsletter. NFC'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. I hope to use this section to keep you informed of long term trends and changes in the lab. Please contact me if you have any questions or concerns about the operation of NFC.

In FY04 the NanoFabrication Center showed a small increase in usage as compared to FY03. Staffing has remained level. We currently have one opening in the maintenance area. Equipment brought into the facility over the last couple of years all continue to be popular. Direct write e-beam usage remains fairly steady at a level sufficient to cover most of the cost of the system. Some students have demonstrated sub 30 nm features with the system. The new AJA deposition system is also quite popular. The load lock feature can allow faster runs than the old system, particularly for many wafer batches. A new AFM system, which allows full 6" wafer capability, came up this summer. We had more than 20 people sign up for the first short course. We have been working since spring to bring up a chemical mechanical polishing system and hope to have this machine ready for use by early fall. The system will be available for polishing SiO_2, Si_3N_3, PSG, and other dielectrics. This year we also plan to upgrade the electronics on the LPCVD system, which is 1970's vintage.

We are also introducing a "frequent user" program for academic researchers in NFC. If you spend a total of $10,000 or more at NFC in FY05, we will set up a research incentive account for 15% of what you spent. You will be able to use this account in FY06 for processing costs on any project. We hope that you can use this to develop new proof-of-concept results to strengthen future proposals.

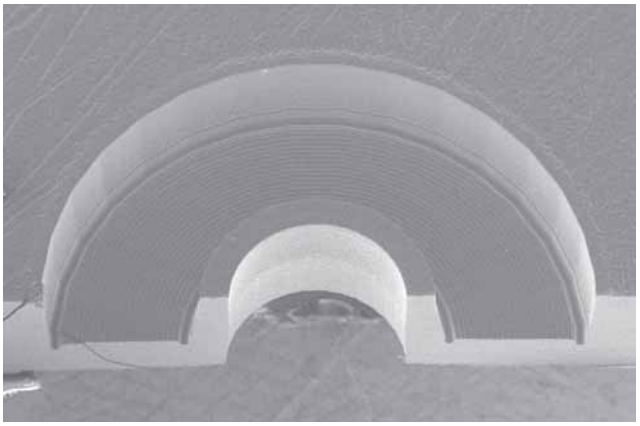
NFC Director, Steve Campbell

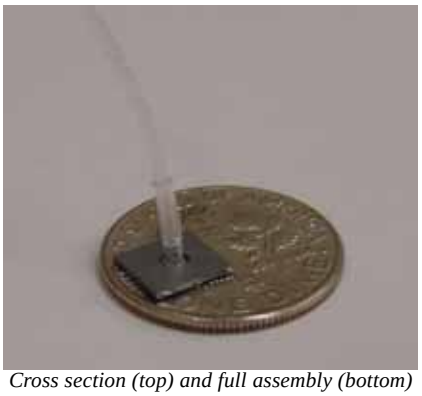
NFC Featured User and Research

Biomedical Microsystems Laboratory at the University of Minnesota
Research Directed by Professor Babak Ziaie
Graduate students: Tingrui Pan, Woohyek Choi, Ming Lei, Amani Salim, Jayant Parthasarathy, Chulwoo Son, and Hyunjoong Kim

Biomedical Microsystems Laboratory is part of the Electrical and Computer Engineering Department of the University of Minnesota. Research in our laboratory is mostly geared towards the design, fabrication, and test of biomedical microsystems with an emphasis on implantable wireless microsystems, biomedical microsensors/ microactuators, MEMS packaging technology for implantable microsystems, micromachined drug delivery systems, biomimetics, and advanced microinstrumentation techniques for high precision biological measurements. Most of the work in our laboratory is collaborative across several departments and disciplines including Pharmaceutics, Biomedical Engineering, Neuroscience, Ophthalmology, Mechanical Engineering, and Biotechnology Institute. In collaboration with the Pharmaceutics Department (R. A. Siegel), we are developing several microfluidic platforms for smart sensing and active flow control. These microdevices use environmentally sensitive hydrogels integrated inside MEMS components in order to fabricate wireless glucose transponders and smart micovalves for drug delivery applications. In collaboration with the Ophthalmology Department (J. D. Brown), we are developing a wireless microtransponder of intraocular pressure measurement. In addition, we are also working on several wireless microvalves for the treatment of glaucoma. With our colleagues in Neuroscience (A. D. Redish) and Mechanical Engineering (A. Erdman), we are currently developing a wireless platform for multichannel neural recordings in rat hippocampus. Miniature motors are used to drive an array of electrodes in the brain while simultaneously recording neural activities. We are also collaborating with Biomedical Engineering Department (V. A. Barocas) to design and fabricate microflow sensors for solutions containing proteins and other charged macromolecules.

University of Minnesota Electrical & Computer Engineering Professor Babak Ziaie, Ph. D.





Cross section (top) and full assembly (bottom) of an adhesive-free interconnect for integrated microfluidic systems.

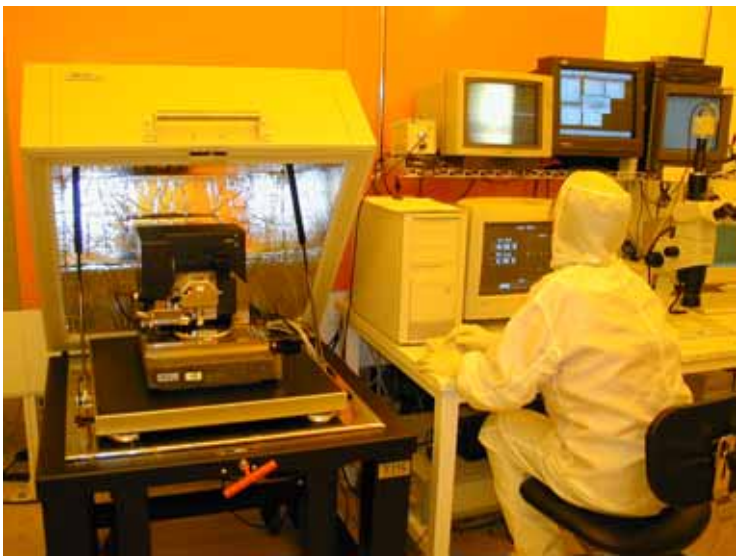
Upcoming NFC Events

Digital Instruments 3100 Atomic Force Microscope Training

The NFC is arranging training classes for our new Digital Instruments 3100 Atomic Force Microscope. This AFM is located inside the NFC cleanroom, making it convenient for examining samples during processing. For more information contact the NFC.

Safety Training

The second Thursday of every month, the NFC has safety training for new lab users. The training includes watching our safety video and taking a brief quiz on the video. Also, a 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 (see feature, page 4). The safety training takes about 2 hours to complete, and must be done before users will be granted access to NFC facilities.



PTL News

PTL Capabilities

To help introduce potential clients to the Particle Technology Laboratory, here is a short list of a few of the measurements we can provide:

- Measure particle size distributions from 3 nm to over 10 mm with high resolution in real time
- Aerosolize a broad range of materials in different phases
- Characterize particle shape
- Select mono-sized particles from a broad distribution for further characterization, conditioning and/or deposition or detection
- Provide real-time size and composition characterization of individual sub-micron particles

The combined expertise of our staff covers a broad range of topics in aerosols. Here is just a small sampling of the types of services we can provide:

- Measure airborne contaminant levels at client workplace
- Characterize powders by size
- Measure non-volatile residue in solutions
- Deposit size-classified particles on wafers, electron microscopy grids or stubs or other surfaces
- Consult on filtration or cleanroom issues

These are just a few examples of the services that we provide. Please contact us with any specific questions about your particle related research projects.

It is with great pleasure that I welcome you to the first edition of the Particle Technology Lab section of the University of Minnesota nanotechnology newsletter. I feel that the opportunities for interaction and collaboration offered by the NNIN program stand to benefit both our clients and our program.

The success of our interactive projects is important to us. We offer access to one of the leading aerosol laboratories in the world and the expertise borne of several decades experience. The combination of knowledge and cutting edge technology offered through this program will provide our clients unique research opportunities while exposure to "real-world" problems will be advantageous to our researchers as well. Many of our measurements can be made *in-situ*, at a client's facility, or in our laboratory.

Our program's special emphasis is on gas-borne nanoparticles. Engineered nanoparticles are important building blocks for nanoscale materials and devices. Gas-phase synthesis and processing techniques produce a million tons of nanoparticles annually and are used in various industrial applications. With over twenty widely used devices developed here, our program has earned a reputation for innovative nanoparticle tool development and application.

Our goal is to perform and facilitate world class aerosol measurements and research, and we welcome any inquires, suggestions, or feedback. Please feel free to contact me at dypui@umn.edu or contact our Lab Manager, Dr. Mark Stolzenburg at mstolz@me.umn.edu, (612) 625-8354.

We look forward to serving you as a partner in aerosol research and measurement.

Particle Technology Lab at the University of Minnesota

Mechanical Engineering 3101
111 Church Street SE
Minneapolis, MN 55455

www.me.umn.edu/divisions/environmental/ptl/
Main telephone: 612-626-7594
Fax: 612-625-5368

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

Distinguished McKnight University Professor, David Y.H. Pui

PTL Featured User and Research

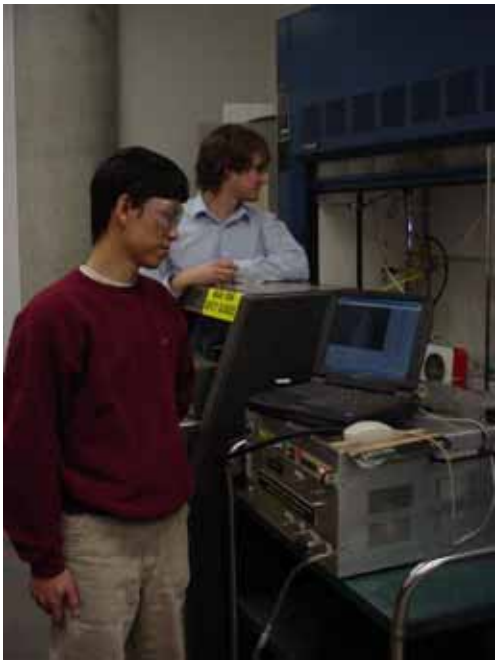
InnovaLight, Inc. Austin, Texas
PTL Instruments Help Optimize Plasma Synthesis of Silicon Quantum Dots

InnovaLight Inc., based in Austin, TX, is a very early stage technology company that was founded in 2001 to exploit the unique optoelectronic properties of silicon nanocrystals in solid-state lighting devices. Silicon nanoparticle-based lighting devices are expected to yield significantly enhanced energy efficiencies as compared to traditional light sources that will also benefit from silicon's reputation as an environmentally benign material. InnovaLight is funded by a top tier investor syndicate that includes ARCH Venture Partners, Sevin Rosen Funds, and Apax Partners. InnovaLight's vision is to become the leading global supplier of solid-state lighting solutions. The company has built strong collaborative relations with research groups at several major research institutions.

InnovaLight collaborates with the group of Professor Uwe Kortshagen in the Department of Mechanical Engineering at the University of Minnesota on the development of a plasma process for the synthesis of silicon nanocrystals in the size range of 2-10 nm. The process involves the use of a nonthermal plasma that is generated at pressures of several mbar. Silicon particles are formed via dissociation of silane (SiH_4) through the energetic plasma electrons. Particles grow in the supersaturated silicon vapor and have a mostly hydrogen terminated surface. The advantage of using a nonthermal plasma lies in the fact that the majority of the silicon particles in the plasma is negatively charged, which strongly suppresses deleterious particle agglomeration compared to other gas phase based processes.

For the *in situ* characterization of the particles produced in the plasma process, InnovaLight and Kortshagen's group use a nanometer Aerosol Size Analyzer (nASA) [1], a device that was developed and made available to InnovaLight by the Particle Technology Laboratory. The nASA enables a fast, online measurement of the particle size distribution. Particles are extracted from the plasma process and the product aerosol is brought to atmospheric pressure through a device called a diluter, in which the aerosol flow is mixed with a high-flow rate nitrogen stream. The nASA allows measurements of the particle size distribution in only a few seconds, which enables online control and adjustment of the plasma process parameters. The use of the nASA has significantly reduced the development time for the plasma process, because the online measurement of the particle size distribution is significantly less time-consuming and labor-intensive than other methods such as electron microscopy.

1. Han, H.-S., D.-R. Chen, D.Y.H. Pui, and B.E. Anderson, *A Nanometer Aerosol Size Analyzer (nASA) for Rapid Measurement of High-concentration Size Distributions.* Journal of Nanoparticle Research, 2000. **2**(1): p. 43-52.



He-Shu Han and Elijah Thimsen measure the particle size distribution of silicon particles synthesized in a nonthermal plasma with the nASA

Upcoming PTL Events

European Aerosol Conference, September 6-10, 2004, Budapest, Hungary, see http://www.aeroszol.hu/conference/ for more information

American Association for Aerosol Research 2004 Annual Conference, October 4-8, 2004, Hyatt Regency Atlanta, Atlanta Georgia, see http://www.aaar.org for more information

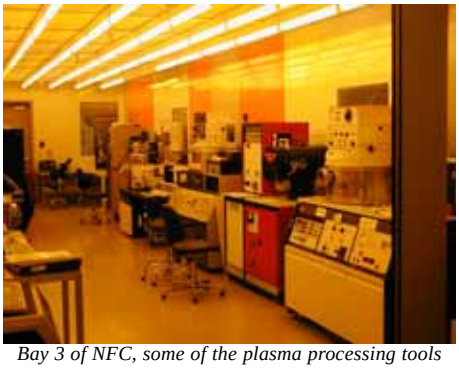
The **IT Characterization Facility mission** relates directly to the core teaching, research and outreach missions of the University

- 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 6700 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
Published quarterly by the University of Minnesota's Nanotechnology Coordinating Office
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

Nanotechnology News from the University of Minnesota
1-165 Electrical Engineering & Computer Science Bldg.
200 Union Street SE
Minneapolis, MN 55455