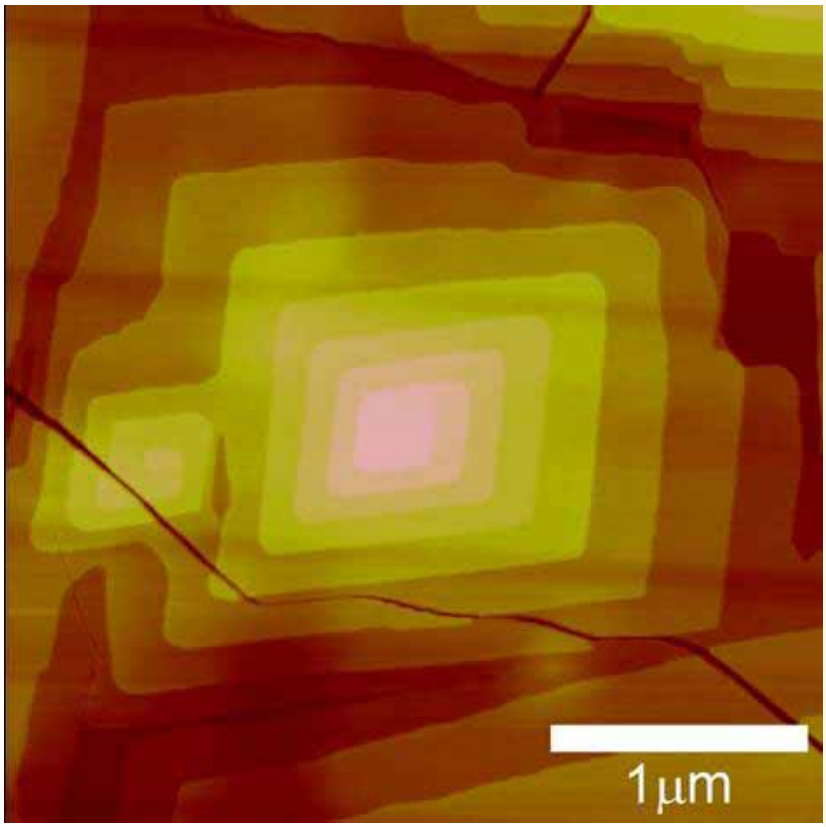
Nanotechnology News from the University of Minnesota

Issue Two Winter 2005

Winter 2005 Nano Image



AFM of terraced, granular, organic semiconductor film with high crystallinity. The long axis of the molecule is nearly perpendicular to the surface. Photo courtesy C. Daniel Frisbie, University of Minnesota, Chemical Engineering and Materials Sciences.

Welcome to the Winter 2005 issue of *Nanotechnology News from the University of Minnesota*. I recently attended the first of our annual NNIN reviews. Although it went very well, NSF is strongly encouraging us to attract more external users. If you know anyone from outside the University who might benefit from our lab services, please encourage them to contact us. I would like to extend my thanks to everyone who helped in providing information on lab usage for this review. We received a great deal of interesting material from our lab users detailing the work they do at our facilities. The information you provided will soon be available on our Nanotechnology Coordinating Office web site (www.nano.umn.edu) in a searchable format. Stop by some time to learn more about your colleagues' research in nanotechnology here at the University.

An exciting research area is emerging at the intersection between the biological and the physical sciences. The National Institute of Health and the National Cancer Institute either just released or will shortly release major new funding initiatives in this new area of nanomedicine that require this type of collaboration. The University of Minnesota is well positioned in this given the close proximity of the Academic Health Center, the Institute of Technology, and the College of Biological Sciences, as well as the strong medical device infrastructure in the region. To kick off interest in this area, the University hosted a Nanotherapy Workshop on November 15, 2004. Over 100 participants from 24 University departments as well as representatives from 16 companies heard talks ranging from bio micromechanical devices (BioMEMS) to gene therapy. A workshop program can be found at www.nano.umn.edu. Many of the talks are posted there as well. A follow up session is planned for the Design of Medical Devices Conference in April 2005 (www.me.umn.edu/dmd/).

Stephen A. Campbell
Director, NFC

In this issue

Nanotechnology News from the University of Minnesota is published by the University of Minnesota's Nanotechnology Coordinating Office and made possible by:

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NSF

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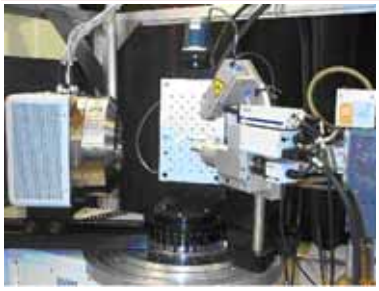
CharFac News



We are pleased to announce the new Bruker AXS Rapid XRD Microdifractometer (pictured). The X-ray beam can be collimated to 50-micron spot size. A multi-wire two-dimensional area detector enables extremely fast data acquisition. Video microscope, laser pointer and XYZ sample stage provide precise positioning and automated surface mapping. Contact Ryan Wold and/or Linda Sauer for further details.

The CCD camera on the new T12 TEM has been integrated into the microscope control software. This means we use the latest version of the camera drivers (faster and more convenient). A stand-alone version of this software is available for offline image processing.

A new digital CCD camera will be installed on the JEOL 1200 TEM in BSBE in January. The camera resides above the specimen viewing chamber, providing a wide field of view while retaining the ability to use the existing plate camera. A significant fraction of basic TEM training is slated for this instrument, making its operation more familiar to general users and freeing time on the JEOL 1210 for advanced work (e.g., cryo).

X-ray photoelectron spectroscopy (XPS or ESCA, electron spectroscopy for chemical analysis) and Auger electron spectroscopy with sputtering are now available as basic capabilities. Contact John Thomas for discussion of particular analytical applications.

The CharFac is now equipped to chemically functionalize AFM tips using thiol/silane chemistry or plasma treatment, and to provide advanced training in chemical force microscopy.

New or upgraded software has been obtained for special data analysis capabilities: SPIP for scanning probe microscopy (e.g., grain size analysis, single-chain force spectroscopy of polymers), and an upgrade to Jade 7 for X-ray diffraction (e.g., whole pattern fitting, 3D visualization of crystal structures). The library from the International Center for Diffraction Data was updated to Release 2004 (163,000 entries).

Characterization Facility at the University of Minnesota

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Greg Haugstad, Director
Mike Boucher, Lab Manager

CharFac Director's Message

The past several months have been extremely active for educational endeavors and outreach at the Characterization Facility. We contributed to two curricular classes: the undergraduate course Structural Characterization Lab (MATS 3801), taught by **Prof. Chris Palmstrom**. Here small teams of students received training from the CharFac staff on lab instruments and went on to conduct lab exercises on specimens tailored for the class. The other course was Physical Principles of Thin Film Technology (EE 5657W), taught by **Prof. Beth Stadler**. The CharFac performed lab demonstrations and provided data on thin film specimens grown by the students.

The CharFac is also joining **Dakota County Technical College** (through a 3-year NSF grant celebrated at DCTC with a speech by **Governor Pawlenty**) to create a course for an associate degree program in Nanoscience Technology. This year, staff member **Stuart McKernan** is developing the fourth-semester lab course content while the first round of 24 students takes the first two semesters of coursework at DCTC. A half day of demonstrations in the CharFac were provided to these students this past fall by several staff members, to help connect current course content with yet-to-be-learned imaging and measurement methods. The capstone fourth semester in the CharFac (along with the NanoFabrication Center) will be provided to two sets of 24 students in spring 2006 and 2007. The content will be similar to that for MATS 3801: training, hands-on exercises, and data acquisition/analysis. This will be only the second tech college "nano" degree program in the nation (along with Penn State), and the first to access the facilities of a research university.

Other recent educational/outreach events included master classes / short courses in small- and wide-angle X-ray scattering organized by staff members **Linda Sauer** and **Ryan Wold** (and former member **Dave Carr**); and proximal probes (e.g., AFM, nanoindentors) organized by **Greg Haugstad, Jinping Dong** and **John Nelson**.

Finally, significant outreach occurred in the form of CharFac overview presentations, tours and/or conference exhibits for several local chapters and one national meeting of professional societies (IEEE, SAE, AIChE, AVS; ASM).

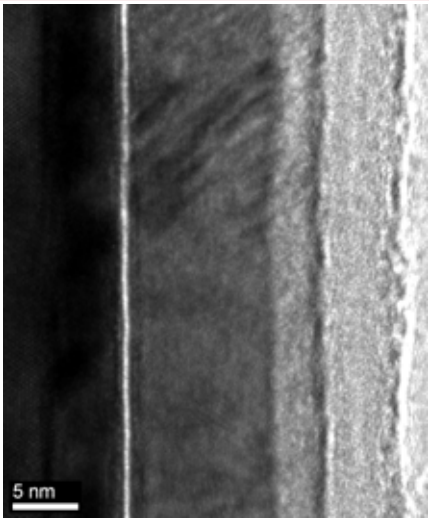
CharFac Director, Greg Haugstad

CharFac Featured User and Research

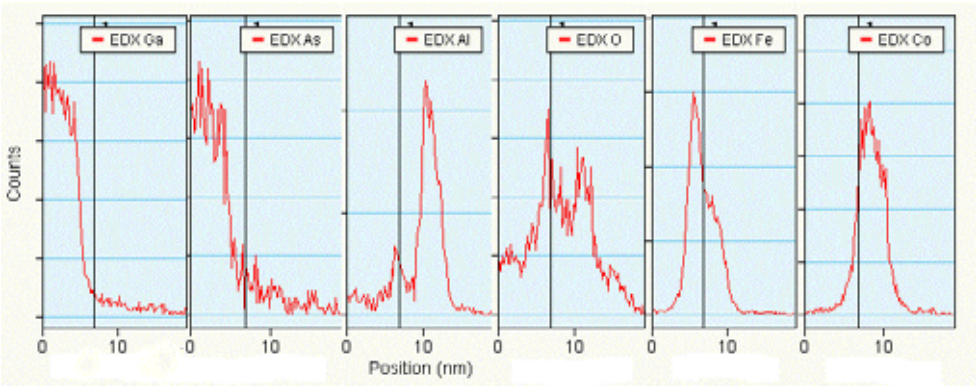
Elemental Mapping of a Magnetic Tunnel Junction Structure

Stuart McKernan, Chang Sun Kong, Xuying Dong, Advisor: Dr. Chris Palmstrøm
Department of Chemical Engineering and Materials Science

In the ever-expanding world of nanotechnology, epitaxial metal films are growing increasingly in importance. It is important to be able to carefully characterize both the growth process of the films and the structure of the entire multilayer after processing to ensure that the desired structure has been successfully fabricated. This research investigated the structure of a magnetic tunnel junction structure consisting of ~200 Å thick $Fe_{0.5}Co_{0.5}$/Al_2O_3 (8 Å)/~50 Å thick single crystal Fe films grown on III-V semiconductors. The thin layer of aluminum was deposited on top of the first single crystal layer of Fe and oxidized in-situ to form an alumina tunneling barrier about 8 Å wide. This barrier layer needs to be very uniform and pinhole-free in order to function as an effective tunneling barrier. After the barrier layer is produced the polycrystalline $Fe_{0.5}Co_{0.5}$ layers are deposited, and the structure is capped with another protective aluminum layer.



To characterize these layers the new field-emission gun transmission electron microscope was used. In STEM mode an electron probe about 0.1 nm can be produced and scanned over the area of interest in the sample. The sample has to be thin enough to be electron transparent – typically less than 20 nm for high-resolution imaging – and this is usually done by mechanical grinding and a final clean-up in an Ar ion mill. This process may cause some damage in the thinnest areas, but careful preparation produces good, clean areas of sample to analyze.



The TEM image shown above depicts the multilayer cross-section, with the GaAs substrate on the left, followed by a 50 Å thick single crystal Fe layer, with the thin, light-colored alumina barrier layer separating that from the $Fe_{0.5}Co_{0.5}$ and Al metal overlayers. Using the small-probe capability of the instrument we generate a linescan across these layers, and the results are shown in the composition profiles here. The vertical marker is in the same position in each image, showing the abrupt transition from the pure iron layer to the $Fe_{0.5}Co_{0.5}$ layer. The Alumina tunneling junction is seen to be fully oxidized and less than 2 nm in width.

Staff News

Ryan Wold has joined the CharFac staff as Scientist in the X-ray diffraction area of the Charfac. Ryan contributes in many aspects of X-ray including design of experiments and data analysis for industrial users; training and assisting new users; and calibration/maintenance of lab equipment. Ryan became active in the field of X-ray diffraction during his recent research on single crystal ferromagnetic shape memory alloys, leading to his masters degree.

Alice Ressler joined the CharFac as Assistant Scientist in the electron microscopy area in the BSBE building on the biomedical side of campus. Alice is active in performing or assisting with specimen preparation, electron microscopy and image processing; training users in these methods; and maintaining equipment and administering the facility. Alice's many strengths derive from more than three decades of experience including technical aspects of EM as applied to biological and biomedical research, curricular development and teaching at St. Thomas, and client interaction in both academic and industrial sectors.

Upcoming CharFac Events

Spring semester 2005 will include curricular courses taught by CharFac staff and utilizing CharFac labs, primarily targeting graduate students but also qualified undergraduates. Staff member **Stuart McKernan** will be teaching courses through the materials science department including Electron Microscopy (MATS 5517, three credits, full semester) and a sequence of three 1-credit, 5-week practical courses (including training) entitled Imaging and Diffraction in the Scanning Electron Microscope, Basic Transmission Electron Microscopy, and Basic Analytical Electron Microscopy (MATS 5518-5520).

Greg Haugstad will be teaching a 4-credit survey course through the chemistry department, Materials Characterization (CHEM 5210), which includes demonstrations and analysis of data acquired with techniques such as scanning probe microscopy, ion beam analysis and Auger/photoelectron spectroscopy.

NFC News

Micro-and Nano-Fabrication Services

If your college or university does not have the equipment you need for fabricating your micro- or nanostructures, the NanoFabrication Center may be able to help. As part of the National Nanotechnology Infrastructure Network (www.nnin.org), we are partially funded to work with academic researchers at other institutions to assist them with fabrication needs. NFC has a full-time staff ready to assist with your project, and our facility is equipped with processing equipment commonly used for micro-and nano-based fabrication. We have capabilities for thin film deposition using sputtering, evaporation, and chemical vapor deposition of silicon, silicon nitride, and silicon dioxide. We routinely deposit low stress silicon nitride films in our LPCVD system. Etch capabilities include both dry (plasma) and wet etching, such as standard RIE of dielectric films, deep silicon etching (Bosch process) for MEMS applications, and KOH anisotropic wet etching. Available lithography tools include three Karl Suss contact aligners, one with backside alignment capability, and electron beam lithography with our Raith 150 system (50 nm minimum feature size). Silicon oxidation, chemical mechanical polishing, and wafer bonding are a few of the other processes that are available. For more information, see the NFC website (www.nfc.umn.edu) or contact nfc@ece.umn.edu.

Staff News

In October, NFC added a new staff member, **Gary Olin**, as a member of our maintenance staff. Gary comes to us from Polarfab, Inc., a Bloomington, MN company that manufactures analog circuitry. Gary has extensive experience in cvd systems, and will work in that area, as well as the etching area.

NanoFabrication Center at the University of Minnesota

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Steve Campbell, Director
Greg Cibuzar, Lab Manager

NFC Director's Message

As a new year begins I would like to thank you, the NFC user, for making our Lab successful. We are glad that our NNIN funding has allowed us to keep our rates down this year, and our usage is strong, particularly among internal users. Our external usage, on the other hand, reflects the generally sluggish economy and is off somewhat from some previous years.

In 2005 we will work hard to improve our Lab's capabilities and user services. Certain aspects of our training will be moving on-line, in the form of videos. These videos, which will be part of our new website, will be available via terminals in the clean room so that infrequent users can refresh their memories regarding the correct operation of machines in the Lab. In addition, we also plan to double the number of general safety training sessions to allow new students to begin work in the Lab more promptly.

I hope that you can help NFC in the new year in two ways. First, let me know if there is equipment or fabrication capabilities that we do not have and you feel that we need. This is particularly true if multiple groups need the same capability. Second, if you know of someone who may have some interest in our services, please tell them about NFC or pass along their contact information to us. A strong base of external users is essential to our evaluation as a NNIN node and helps to distribute the costs of operation over a broader base. I hope that you all have a successful 2005.

NFC Director, Steve Campbell

NFC Featured User and Research

Triad Sensors, Inc., Minneapolis, Minnesota
MicroElectroMechanical Systems ("MEMS") Technologies Development
Research Directed by Dr. Peter Schiller, Triad CEO/CTO

MEMS are tiny, micron-sized devices that are fabricated on silicon wafers and share many electrical and structural properties with integrated circuits. In addition, MEMS have a mechanical component that is also integrated at the chip level.

MEMS devices function in two broad categories, sensors and actuators, and provide the ability to sense or manipulate the surrounding environment. The primary advantages of MEMS devices are the miniaturization and cheaper production of faster, more reliable devices than conventional technologies.

Over the past four years Triad developed and optimized Kaleidoscope®, a system of MEMS design and fabrication that successfully counters the problems that have stifled growth in the industry: manufacturing standardization, development time and costs, device performance and process flexibility.

A highlight of Triad's Kaleidoscope technology is the inclusion of PZT thin-films into the process. Kaleidoscope is the first MEMS process to successfully integrate PZT thin-films into a practical, manufacturable process and leverage the myriad benefits of this high order piezoelectric.

Dr. Schiller is an alumnus of the University and has spent the last fifteen years in semiconductor-related research and product development.

Piezoelectric materials convert mechanical energy to electrical energy and vice versa. For instance, if force is applied to a piezoelectric crystal, a voltage is generated in proportion, thereby producing the function of a sensor. Similarly, if an electrical voltage is applied to a piezoelectric crystal, it will expand in proportion, creating an actuator. Kaleidoscope combines simultaneous operation of both effects, enabling higher precision and a far broader range of application possibilities.

Triad's most recent development effort produced a solid-state silicon rotational rate sensor (gyroscope) intended for the automotive marketplace. Triad's gyro promises to provide a more cost-effective and higher performing solution for use in electronic stability control (ESC) and rollover detection systems.

Dr. Schiller commented on Triad's experience at the NFC, "As a technology development company, it is crucial that we have access to leading-edge facilities. We have found the NFC to be a truly unique and invaluable resource."

Upcoming NFC Events

Research Experience for Undergraduates

Undergraduate students interested in spending summer doing research at NFC as part of the **NSF Research Experience for Undergraduates** (REU) program should check out the NNIN REU website at http://www.nnin.org/nnin_reu.html. Each year students from all over the US go to NNIN sites for interesting research studies, capped off by a final weekend at one of the NNIN sites in August where all REU students present their work. A great opportunity for undergraduates.

Safety Training

Beginning in January 2005, NFC will be 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.

New Condensation Particle Counter from TSI Incorporated

The Model 3785 CPC uses supersaturated water (not butanol) to enlarge submicrometer particles to optically detectable sizes.

This water-based CPC is nontoxic, odor-free and non-contaminating. Patented* technology eliminates the need for alcohol as a condensing fluid. Instead, the properties of water that make it unsuitable for use in conventional laminar-flow CPCs are exploited to create this general-purpose particle counter.

Photo courtesy of TSI, Inc.

With sensitivity to particles in the range from 5 nm to over 3 μm in diameter, the Model 3785 provides performance that is comparable to other condensation devices.

It employs a continuous, 1 liter per minute, laminar sample flow for quick response (< 2 seconds) to fast-changing aerosols and improved performance for low concentration measurements. Photometric and single-particle-counting detection modes offer continuous, "live-time" coincidence correction and result in a wide dynamic particle-concentration range (10^{-3} to 10^{7} particles/cm^3).

*Technology from Aerosol Dynamics Inc., United States Patent Number 6,712,881

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

The Particle Technology Laboratory (PTL) is a unique component of the NNIN network. We provide resources, tools and facilities to assist NNIN users in generating, classifying, and measuring airborne nanoparticles. Faculty and staff members at PTL have collectively developed over 20 commercial instruments for aerosol measurements. An example is illustrated in this issue. The TSI Inc. water-based condensation particle counter (CPC), capable of counting nanoparticles down to 5 nm, is the only commercial CPC operated with water as working fluid (most others use butanol). It is expected to find applications particularly in confined environments such as in a clean room. Dr. Mark Stolzenburg, our Laboratory Manager, is instrumental in the development of the CPC.

We are very pleased to host the 2nd International Symposium on Nanotechnology and Occupational Health, October 3-6, 2005. This symposium follows on from last year's symposium (held in the UK, October 2004), and is aimed at providing a forum for multiple stakeholders to network and present/discuss current research and related activities relevant to nanotechnology and occupational health. The Symposium will enable the stakeholders to exchange knowledge and best practices in monitoring and handling nanomaterials, and in protecting workers. With the increased public concern on the potential risks of nanomaterials to human health and the environment, the Symposium will help to facilitate the development of responsible nanotechnologies and to gain public trust. Please follow the latest progress on the Symposium preparation on http://www.cce.umn.edu/nanotechnology .

Distinguished McKnight University Professor, David Y.H. Pui

PTL Featured Conference News

NNIN-UMN Node to host 2nd International Symposium on Nanotechnology and Occupational Health
October 3-6, 2005
Radisson Hotel Metrodome, Minneapolis, Minnesota

The NNIN-UMN Node will host the 2nd International Symposium on Nanotechnology and Occupational Health, to be held in Minneapolis from October 3 – 6, 2005 (details at http://www.cce.umn.edu/nanotechnology). This will be the premier international conference of 2005 specifically focusing on research into how nanotechnology will potentially impact health and safety in the workplace, how the impact can be minimized, and how nanotechnology can be used to improve occupational health. It follows the highly successful 1st Symposium, held in Buxton, England, in October 2004.

The commercial interest in nanotechnology applications is increasing at a rapid pace. Many potential new users and workers lack the experience of handling nanomaterials. There is also an increased public concern about its potential risks to human health and the environment. The purpose of this Symposium is to bring together scientists, engineers, and health professionals to exchange knowledge and best practices in monitoring and handling nanomaterials, and in protecting workers.

The symposium will be co-chaired by Professor David Pui of the Particle Technology Laboratory at the University of Minnesota, and Dr. Andrew Maynard of the National Institute for Occupational Safety and Health. The symposium will be primarily sponsored by the US National Institute for Occupational Safety and Health and the University of Minnesota, a National Nanotechnology Infrastructure Network (NNIN) node. The first day of the symposium will be devoted to tutorials aimed at bringing attendees up to speed in cross-disciplinary areas, followed by three days of platform and poster sessions. There will be a mix of submitted and invited presentations. Platform time is also planned to provide an opportunity for research centers, laboratories and organizations to update the symposium on current activities.

A Request for Abstracts was sent on January 5, 2005 with abstracts due by March 15, 2005. A partial list of the session topics includes:

1. Nanomaterials, structures and devices production
2. Nanomaterial emission and exposure routes in the workplace
3. Exposure monitoring and characterization
4. Nanomaterials and health risk
5. Nanomaterial product characterization
6. Emission Control and exposure protection
7. Good working practices
8. Developing health policy and regulatory frameworks for nanomaterials
9. Current nanotechnology and occupational health activities

The organizers would like to invite NNIN academic and industrial users to submit abstracts and to learn the latest knowledge and advancement on this timely topic.

Upcoming PTL Events

February 7-11, 2005
AAAR Supersites Conference
Sheraton Atlanta Hotel
Atlanta, Georgia
www.aaar.org/05Supersites/ConfInfo.htm

March 14-18, 2005
International Society for Aerosols in Medicine 15th International Congress
Perth, Australia
www.isam.org

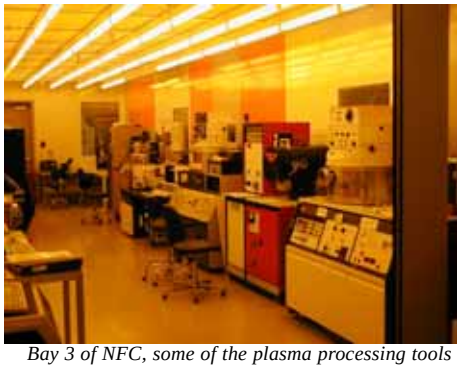
The IT Characterization Facility mission relates directly to the core teaching, research and outreach missions of the University

- 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 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

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and the National Nanotechnology Infrastructure Network. Edited by Becky Von Dissen.
Comments and suggestions are welcome! Would you like to be added to or removed from our distribution?
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