Nanotechnology News from the University of Minnesota

Issue Three Summer 2005

Summer 2005 Nano Image

Professor Uwe Kortshagen, of the University of Minnesota's Mechanical Engineering department, is working with InnovaLight, Inc. to develop silicon nanoparticle light emitting structures. Shown in the figure above are containers of ~5 nm (red) and ~3 nm (green) particle containing solutions. Photo courtesy of Nancy Johnson.

Welcome to the Summer 2005 NNIN/Nanotechnology Coordinating Office newsletter. The purpose of this newsletter is to highlight the nano-related capabilities of the University of Minnesota and especially, to attract new users for the Labs.

One of the activities that I am often asked to do is to escort prospective University of Minnesota faculty members on tours through our facilities. I have never had such a tour go badly. Most often, the candidate comes away amazed at our capabilities, particularly if they have used similar facilities at other schools. A recent candidate who does BioMEMS research came away saying that our facilities are better than where he did his doctoral work: MIT. Some former faculty members who were hired away to other schools continue to use our facilities because they are better than what they have at their new institutions. In one case, the faculty member actually set up a company located at the University of Minnesota expressly to use our facilities.

Clearly we have a great deal to be proud of. However, we cannot take these capabilities for granted. We need your help to recruit new users and to work with us on new projects. The more users we can attract, the lower the rates and the broader the capabilities that all of us can enjoy. I hope that you have an enjoyable and successful summer.

Best regards,
Steve 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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CharFac News

NNIN
www.nnin.org
Nanoscale Science, Engineering & Technology

Equipment News

We are pleased to announce the installation of the Molecular Imaging (MI) PicoPlus scanning probe microscope, positioned in "CharFac South", our labs in Hasselmo Hall (formerly the BSBE building next to Coffman union, a facility housing two cryo-SEMs and our bio-emphasis TEM). The Pico+ is a second-generation microscope from MI that offers critical improvements over the PicoSPM, the first generation system in the CharFac Shepherd Labs facility since 1999 ("SPM3"). Among other things this SPM will serve a key NNIN function: remote control (via the internet). The Pico+ includes many of the conveniences of our Digital Instruments (DI) Multimode SPMs such as large scan range, optical access and vertical engage, but adds advantages such as environmental control (humidity, temperature), more data acquisition channels, higher pixel resolution, true phase measurement, low coherence laser, open liquid cell, magnetic AC mode for acoustic-vibration free operation in liquids, "spectroscopy volume" (force-distance curves interlaced with conventional images), and current sensing for I-V point characterization or current measurement during imaging or load ramp.

Witec pulsed force mode (PFM) AFM is now available on all scanning probe microscopes in the CharFac. A sample shaker has been added to make PFM compatible with our DI Nanoscope III based systems, and PFM also functions on both of our MI SPMs. PFM measures force during rapid approach-retract cycles to characterize stiffness and adhesion, and does so at high pixel resolution.

In early May we installed the new Hysitron Triboindentor in the CharFac. This expands many of the capabilities beyond our existing Hysitron Triboscope, which interfaces with a DI Multimode SPM. The Triboindentor is a complete, ultra-low load test system designed to measure the nanomechanical properties of thin films and material surfaces. Unlike the Triboscope it is a stand-alone system. It includes lateral scanning capability and dynamic mechanical analysis (e.g., complex modulus).

A Nikon Optiphot light microscope has been moved from Shepherd Labs to "CharFac South" in Hasselmo Hall. Equipped for Bright Field, and potentially Phase Contrast and Nomarski imaging, with an adapter to couple a digital camera, it will be primarily used by staff to document biological sample preparation for TEM.

Characterization Facility at the University of Minnesota

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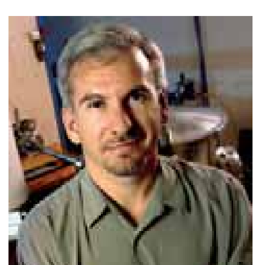
Greg Haugstad, Director
Mike Boucher, Lab Manager

CharFac Director's Message

As the CharFac heads into the busy "on season" (May-Sept) in anticipation of new users and activity, we highlight areas of rejuvenated emphasis: "bio-microscopy" and thin film analysis. Developments in staffing and instrumentation are conducive to substantial growth of usage in these areas.

Electron microscopy of biological and biomedical specimens in our Hasselmo Hall labs is poised for substantial new levels of activity, thanks to recent instrumentation and staffing infusions (highlighted in the present and winter newsletters). For transmission electron microscopy there have been developments in specimen preparation and digital image acquisition. Bio-emphasis scanning probe microscopy (i.e., AFM) has improved with the acquisition of a new multi-purpose instrument that is especially conducive to *in vitro* or *in vivo* research, and has several "second-generation" features. This system has been placed in the Hasselmo Hall facility, the first multi-user AFM on that side of campus to our knowledge. Staffing and digital technology is also being repositioned to cater to these users. (Contact Greg Haugstad to learn more.) The AFM system also will have an important role in our node of the National Nanotechnology Infrastructure Network (NNIN): remote control. A staff member will configure the system and specimens for web-based control by users at other locations in the country. Web-based control is also being configured soon on the JEOL field emission SEMs in the Shepherd Labs CharFac.

For thin film and surface analysis, we now have trained users of X-ray photoelectron spectroscopy (XPS) and clients for Auger spectroscopy with sputtering. We have added these capabilities by acquiring and resuscitating older systems. As usage of these techniques rejuvenates, the CharFac will push for newer systems with higher-order capabilities (such as small spot). Staffing is of course an essential part of the equation in these areas as well as ion beam analysis (Rutherford backscattering and related techniques), where staffing loss occurred in recent times. The CharFac staff has reorganized and found practical ways to add expertise and effort in these areas. For more information, contact John Thomas (XPS/AES) or Greg Haugstad (IBA).



CharFac Director, Greg Haugstad

CharFac Featured User and Research

Characterization of TPA-Silicalite-1 Precursor Solutions

Tracy M. Davis, Department of Chemical Engineering and Materials Science, University of Minnesota
Advisor: Michael Tsapatsis

Zeolites are a unique class of inorganic materials, defined by their highly ordered cages and channels of nanometer scale. These aluminosilicate materials are used as membranes in a variety of industries due to their sorption and catalytic properties. The extent to which membrane properties can be controlled is dependent upon an understanding of membrane growth. Tetrapropylammonium (TPA)-silicalite-1, a pure siliceous zeolite, can be prepared as a selective membrane through a seeded growth technique [1]. This process begins with the growth of seed crystals, which, in the case of TPA-silicalite-1, are synthesized from aqueous solutions. Thus, initial steps towards controlling membrane properties include the characterization of such precursor solutions and research into how TPA-silicalite-1 seeds form.

Tracy M. Davis, University of Minnesota, Materials Science Graduate Student

Simultaneous small and wide angle x-ray scattering studies of the TPA-silicalite-1 solutions were performed on the Anton-Parr SAXSess. This instrument allows in-situ identification of particles on the order of 5-100 nm in diameter, and also the determination of crystallinity. Our studies have verified the existence of amorphous particles within the precursor solutions. The particles are believed to be the primary growth units of TPA-silicalite-1 seeds, and therefore the initial building units for TPA-silicalite-1 membranes (see figure 1). According to our studies, the primary growth units are on the order of 5-nm in diameter and non-spherical (see figure 2). Size and shape are determined from experimental x-ray scattering curves through an indirect Fourier Transform using GIFT, a complimentary software program. The resulting pairwise-distance distribution function (PDDF) is plotted as p(r) versus r, where p(r) is the probability of occurrence of dimension, r, within the scattering unit. The particle size is found to increase with time at room temperature (see figure 1). These results show clearly that the particles are evolving and provide a glimpse into the initial stages of membrane growth.

Fig. 1

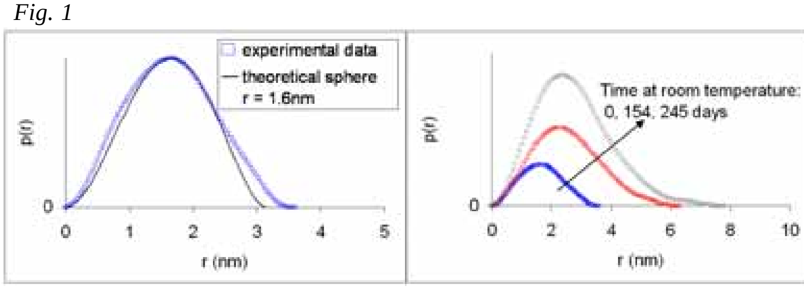
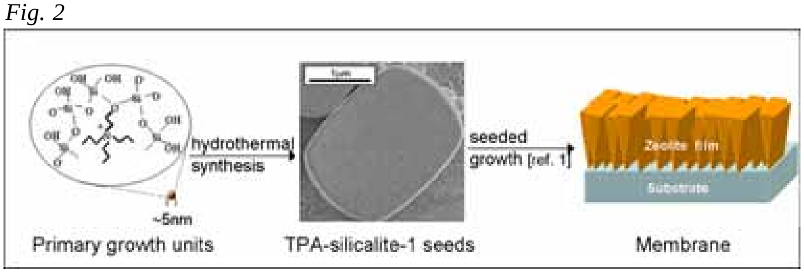


Fig. 2



1. Lai, Zhiping et al., **Science**, 2003. **300** (5618): p. 456.

Staff News

Joysurya Basu has recently joined the CharFac staff in the Electron Microscopy and Microanalysis group after completing his doctoral thesis. He is involved in regular maintenance of the microscopes, training and assisting new users, designing experiments and extending technical support to the students and the industrial users. He has been actively involved in the area of bulk metallic glasses and nano-composites over the last six years. He has twelve research publications and more than fifteen conference presentations to his credit in this area. Presently he is working on microscopy of defects in optoelectronic materials, magnetic heterostructures and nanoparticles as a part of his post-doctoral research.

Bob Hafner joined the CharFac staff as an Assistant Scientist. Bob's responsibilities include assisting with TEM and SEM sample preparation and imaging in Hasselmo Hall's EM facility, working with the U of M's EM Advisory Committee to help implement its initiatives, and facilitating the work of external users in CharFac's two allied EM labs. Bob's past experience includes an accomplished career as a science educator and imaging experience at SUNY Alfred's College of Ceramic Engineering, University of Wisconsin-Madison's Department of Computer and Electrical Engineering, and Northwestern's Biological Imaging Facility.

Upcoming CharFac Events

Molecular Imaging and the CharFac will co-host a **workshop and seminar on Scanning Probe Microscopy** June 16, 2005. Experts will discuss current trends in AFM/SPM nanotechnology research for life science, biotechnology, electrochemistry and material and polymer science, and advances in AFM tools and technology including environmental and temperature control and molecular recognition. For more information contact Bill Domansky (bdomansky@btsbio.com) or the CharFac.

The **annual meeting of the Industrial Partnership for Research in Interfacial and Materials Engineering (IPRIME)** will be held May 31- June 3. See http://www.iprime.umn.edu. The event will include a cryo-TEM "master class": a half day of lectures by experts and a full day of lab demos and sample analysis. The CharFac also will have an exhibit and demo of a new scanning probe microscope at the meeting's poster session.

New Processing Capability: Focused Ion Beam Technology

In early April NFC received a FEI Quanta 200 3D focused ion beam (FIB) system. The Quanta 200 3D is a DualBeam (FIB / SEM) system with a tungsten electron column which can be used to section, image and analyze a wide range of conducting and non-conducting samples. FIB systems are similar to scanning electron microscopes (SEM) except that gallium ions are used instead of electrons. The gallium beam can be focused down to a very small spot (10nm size at 30KV acceleration voltage) and can be scanned across the sample. The large mass difference between Ga ions and electrons causes an effect that is not seen in SEMs, namely material removal. Another name for this removal process is ion milling, and whenever the Ga beam is contacting the surface, some surface material is being removed. By focusing the Ga beam to desired areas, controlled removal of material can be accomplished, resulting in the formation of structures with dimensions in the range of nanometers. The process is purely physical, meaning that no chemical effects are involved, so any material can be milled using the Ga beam. In addition to the milling process, introducing an organic-metallic compound containing Pt near the Ga beam impingement site leads to the controlled deposition of Pt metal. The Pt metal will contain some organic residue, and thus is not as conductive as pure Pt. However, the ability to selectively deposit Pt in areas as small as 50nm is a useful feature for nanofabrication. Common uses for FIB technology include TEM sample preparation, device cross-sectioning for failure analysis, and selective deposition of small Pt conducting lines.

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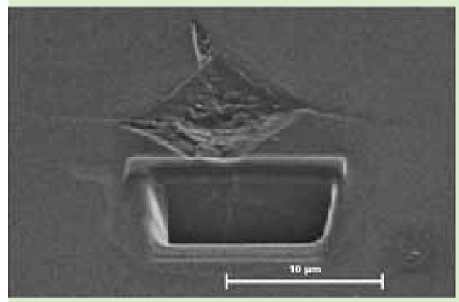
Steve Campbell, Director
Greg Cibuzar, Lab Manager

NFC Director's Message

As discussed in the column at left, a new Quanta 200 3D dual beam FIB will soon be available to our users. This system is a direct result of requests from many faculty for such a tool. This summer we will be substantially upgrading the LPCVD system. Originally built in the 70's, the system uses large (~8") floppy disks as the primary storage media. All of the electronics are on the order of 25 years old as well. The upgrade will allow us to reuse most of the existing tubes, pumps, and gas plumbing, while upgrading the electronics and adding a dedicated tube for our low stress nitride process. This makes the upgrade far less expensive and less disruptive than buying a new system.



Micro indent in Silicon exposed in the 3rd dimension by FIB milling

Money for new systems or repair of old systems often comes from several sources. In the case of the FIB, we received some opportunistic funding from the Dean's office. In other cases we have been helped by grants obtained from interested faculty. The new CMP system, which is now available for use, is an example of such a system. Unfortunately, however, we often take on debt by allowing a deficit in one of our accounts. As an Independent Service Organization (ISO) we are permitted to do this. Payment of this debt comes from user fees and accounts for a sizeable fraction of the overall NFC budget. NFC has a good record of meeting these obligations, but the burden is not easy. I hope that you will help me in finding users for these tools so that we will be able to pay for these systems.

NFC Director, Steve Campbell

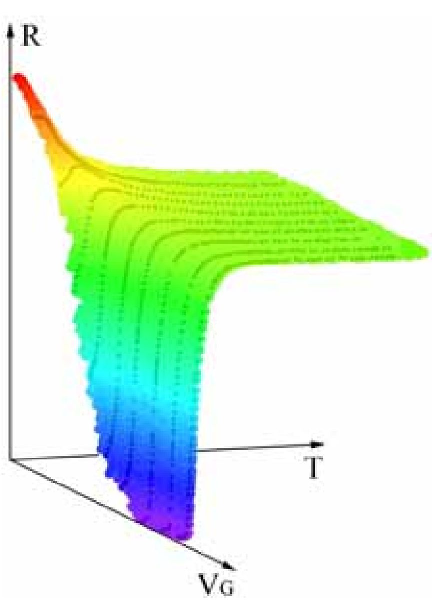
NFC Featured User and Research

Electrostatic tuning of the superconductor-insulator transition in two dimensions

Kevin A. Parendo, K. H. Sarwa B. Tan, A. Bhattacharya, M. Eblen-Zayas, N. E. Staley, and A. M. Goldman,
School of Physics and Astronomy, University of Minnesota.

Field effect devices made from silicon are at the core of modern technology. The field effect can also be used to change the properties of materials by electrostatic rather than chemical doping. In this work superconductivity has been induced in a ten Angstrom thick insulating film using the electric field effect. The resultant insulator-to-superconductor transition is a quantum phase transition, an important and very general paradigm in contemporary condensed matter physics. Quantum phase transitions are driven, not by thermal motion, but by the quantum fluctuations associated with Heisenberg's uncertainty principle. These quantum fluctuations for complex systems such as superconductors, are associated with "zero-point motion." Although a film can never be cooled down to a quantum critical point, which is found at the absolute zero of temperature, dramatic effects can be felt long before this point is reached. It is this influence that elevates quantum phase transitions from a hypothetical phenomenon found at the absolute zero of temperature, to a real-world phenomenon that can profoundly change nonzero-temperature material properties. The superconductor-insulator transition in ultrathin films of metallic atoms is by many measures the simplest quantum phase transition and is thus an excellent model system for exploring the phenomenon. Other examples are found in complex superconducting and magnetic compounds exhibiting strong correlation effects, two-dimensional electron systems, and helium adsorbed on surfaces. This work will appear in the 25 May issue of *Physical Review Letters*.

Allen Goldman, professor and head of the University of Minnesota's School of Physics and Astronomy



The figure at left shows the resistance versus temperature as a function of gate voltage illustrating the transition from superconducting behavior, high gate voltage to insulating behavior.

Upcoming NFC Events

CMP Training

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

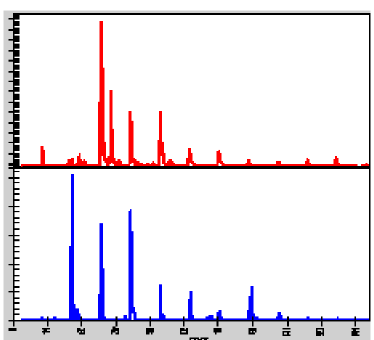
Safety Training

NFC is offering safety training for new users twice each month. On the first Thursday of every month, training sessions begin at 1:30PM, and on every third Thursday sessions begin 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.

TSI 3800 Aerosol Time of Flight Mass Spectrometer

The Particle Technology Laboratory is proud to make a customized TSI 3800 Aerosol Time of Flight Mass Spectrometer (ATOFMS) available to our research community. This technology, developed by Dr. Kim Prather at University of California Riverside, yields single particle aerodynamic size and single particle chemical composition. The ATOFMS uses an aerodynamic sizing technique, similar in concept to the TSI 3321 Aerodynamic Particle Sizer, to size individual particles in real time. The particles are then desorbed and ionized by a pulsed laser and the composition is determined in a bipolar time-of-flight mass spectrometer. Our instrument can be equipped with two different inlets. The standard nozzle inlet allows particles between 0.5 µm and 3 µm to be analyzed, while the second inlet, an aerodynamic lens, allows particles between 100 nm and 600 nm to be efficiently analyzed. This instrument is fully transportable allowing the possibility of conducting off site analysis.

The ATOFMS has many possible applications and has already been used for atmospheric sampling, identification of contaminant sources in industrial applications, indoor air quality measurements, powder process identification and diesel particulate matter characterization. We look forward to collaborating with you on projects using this innovative technology.



Positive and negative mass spectra of a 100 nm soot particle

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

PTL Director's Message

The Particle Technology Laboratory has a long history of industry-university partnership. We believe such collaboration will help make the industry more competitive globally while helping us to become more relevant in our research and education. The Microcontamination Research Consortium was established in 1985 with 4 companies. It eventually grew to 17 member companies from the semiconductor industry. During 15 years of the Consortium, we graduated 25 Ph.D. and 12 M.S. students, approximately 75% of graduates are working in microelectronic companies. Intel Corporation is currently sponsoring a research project on Nanocontamination to study protection schemes for Extreme UV Lithography (EUVL) systems.

We now have an on-going Center for Filtration Research involving nine companies and an affiliated government agency. Current members include 3M, Donaldson Company, Fleetguard, W.L. Gore, TSI, and Samsung Digital Appliance, and NIOSH as an affiliated member. The Center objectives include performing filtration research, developing improved experimental methods and seeking new applications. Member companies are particularly interested in the filtration of nanoparticles that will be important in protecting workers in the nanomaterials industry.

We found that our Shortcourse for Aerosol and Particle Measurement is very effective in networking with the industry. First offered in 1978, we have since offered it annually, with the participation of more than 1,700 alumni, mostly from the industry. We have recently added a new topic on Nanoparticle Technology to inform the attendees of the latest tools for nanoparticle generation and measurement. The course includes both lecture and laboratory components and has received high marks in attendees' evaluations. This year's Shortcourse, the 30th offering, will be held August 22-24, 2005 at the Radisson Hotel Metrodome in Minneapolis. For more information about the course, please check www.cce.umn.edu/aerosol.

Distinguished McKnight University Professor, David Y.H. Pui

PTL Featured User and Research

Particle Technology Laboratory Instruments Help in Sorting α–Al_2O_3:C Nanoparticles

Victor Weir, Biophysical Sciences and Medical Physics, University of Minnesota

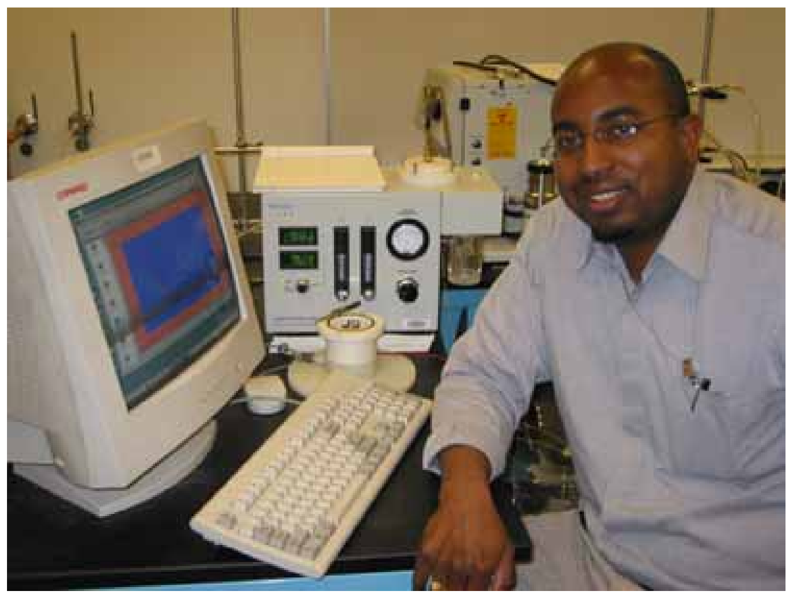
Aluminum oxide is a ceramic material that has various industrial applications. One recent application is in the area of personal dosimetry. As a dosimeter, aluminum oxide competes with the more traditional Lithium Fluoride (LiF) dosimeter. Both dosimeters utilize thermoluminescence (TL). Aluminum oxide also has optically stimulated luminescence (OSL) properties. By taking advantage of these properties, aluminum oxide based dosimeters have higher sensitivity, precision and wide dynamic range of detection with better accuracy. The current research focus is on studying OSL response as a function of both particle size and energy for α–Al_2O_3:C dosimeters. Landauer Inc., a large radiation monitoring and testing company, is providing the funding for this project. Luxel™, an α–Al_2O_3:C based personal dosimeter, is one of the products that Landauer produces and markets.

Previous work has shown that TL dosimeters, such as LiF powders, exhibit a particle size effect that influences their TL sensitivity and that the energy–size dependence may be partly explained by the variation in energy actually deposited in the grains under different experimental conditions. Furthermore, once the particle size approaches the nanometer range interesting effects, such as quantum confinement and surface effects, may become significant.

These aluminum oxide particles are produced using a top-down approach in which a bulk single crystal is reduced to a powder from which desired particle sizes are selected.

For this study, the Particle Technology Laboratory (PTL) is involved in classifying polydispersed α–Al_2O_3:C powders into monodispersed nanoparticles using a Differential Mobility Analyzer (DMA). An aerosol mist of particles is charged and sent through the classification section of the DMA where there is an electric field. This electric field balanced with the sheath flow rate through the instrument allows the classification of particles with a single mobility size. The sorted particles are deposited onto a substrate where they are further analyzed.

Currently, particle samples from 10 to 60 nm have been deposited on TEM grids. The goal of this phase of the project is to study the electronic structure of defect centers by using electron energy loss spectroscopy (EELS). The next stage will be to use vacuum ultraviolet (VUV) spectroscopy to study optical properties and compare to the EELS results. VUV spectroscopy offers a higher energy resolution while EELS extends the energy range. Finally, luminescence sensitivity for various particle sizes and energies will be measured after x-ray irradiation.



Victor Weir, of the University of Minnesota's Biophysical Sciences and Medical Physics graduate program, uses PTL equipment to size, classify and collect α–Al_2O_3:C nanoparticles for luminescence studies.

Upcoming PTL Events

August 13-16, 2005
Particles 2005 Conference on Surface Modification in Particle Technology
San Francisco, California
http://nanoparticles.org/Particles2005/

August 22-24, 2005
University of Minnesota Aerosol and Particle Measurement Short Course
Minneapolis, Minnesota
http://www.cce.umn.edu/aerosol/

August 28 - September 2, 2005
European Aerosol Conference 2005 (EAC 2005)
Ghent, Belgium
http://www.EAC2005.be

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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Comments and suggestions are welcome! Would you like to be added to or removed from our distribution?
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