NNIN
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Nanoscale Science, Engineering & Technology

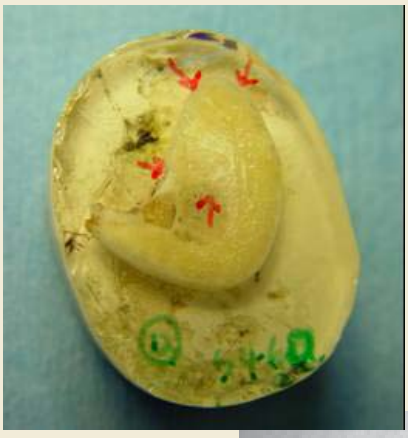
UNIVERSITY OF MINNESOTA

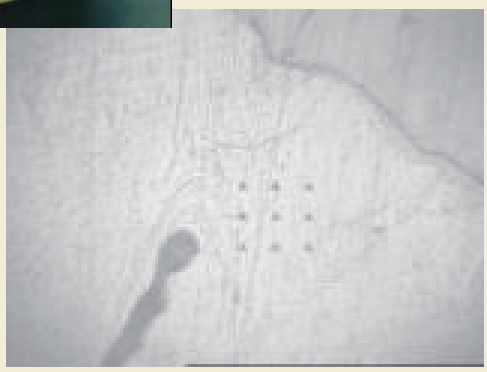
NANOTECHNOLOGY NEWS FROM THE UNIVERSITY OF MINNESOTA

ISSUE FIVE WINTER 2006

WINTER 2006 NANO IMAGE

Professor Wook-Jin Seong from the Department of Restorative Sciences in the School of Dentistry is using CharFac's Nano Indenter to measure the Young's modulus and hardness of regions of the human jawbone to understand dental implant success and failure.





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

Welcome to the Winter 2006 issue of the University of Minnesota's Nano Newsletter. Inside you will find updates on the NNIN nano labs, profiles of users, and descriptions of upcoming events. This semester we are welcoming the first class of students pursuing an AAS degree in Nanoscience from Dakota County Technical College in Rosemount, Minnesota. They will be spending a capstone semester here at the University, learning to operate the machines in the NanoFabrication Center, Characterization Facility, Particle Technology Laboratory, and the BioTech Institute in Saint Paul. The capstone provides them a unique experience in gaining hands-on training in a wide variety of equipment and processes.

The National Institutes of Health recently announced the Mentored Quantitative Research Career Development Award. This award is meant to attract to NIH-relevant research from those investigators whose quantitative science and engineering research has thus far not been focused primarily on health and disease. Stephen Ekker, Associate Professor in the Department of Genetics, Cell Biology, and Development, can help in indentifying suitable biological mentors. For more information on this funding opportunity through NIH as well as others including the EPA and DARPA, we encourage you to consult the nano website: **www.nano.umn.edu**. Here you will also find information on upcoming nano events, important news and interesting links.

NNIN has a number of open **discussion lists** for technology exchange for both users and non-users. These include lists for computation, biology, fabrication, and materials. Sign ups are accessible from the contacts page on the NNIN web site: http://www.nnin.org/nnin_contact.taf

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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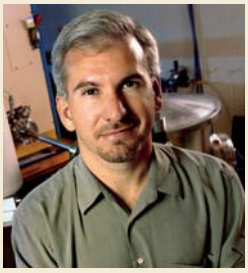
Characterization Facility News

CharFac Director's Message



CharFac Director, Greg Haugstad

The CharFac is happy to report three recent awards from the Graduate School's Grant-in-Aid program. This will bring in five new pieces of equipment as described in the New Equipment section: one instrument, one attachment and three tools for specimen preparation. The items will be placed in the CharFac's Shepherd Labs or Nils Hasselmo Hall facilities. Interdisciplinary collaborations underlie our successes (i.e., six for six in GIA proposals over the past couple of years). The authors of the three recent proposals are from four colleges: Institute of Technology, Medical School, College of Pharmacy, and College of Biological Sciences (CBS). Also key to these infusions of new equipment are matching funds: some grants qualified for special match from the IT Dean (capabilities important to the Materials Research Science and Engineering Center). Other matches were provided by the Medical School and the Department of Genetics, Cell Biology and Development (Medical School/CBS).

The CharFac also has received a gift time-of-flight secondary ion mass spectrometer (TOF-SIMS) from 3M, as described in the New Equipment section. Contributions to defray installation costs were provided by the IT Dean, the Chemistry department and the NNIN. Another contributor to much of the above is the CharFac's capital equipment fund from the IT Dean; it is increasingly used to leverage grant proposals and various contributions. The CharFac is open to ideas for grant proposals from all users, as well as ideas on supplementary funding sources (including industry). The CharFac staff will provide help in writing proposals, identifying users, connecting with potential contributors, etc.

All of the above underscores the value the University places on centrally managed user facilities. This in turn raises the importance of *feedback* from our users regarding the development of instructional procedures, usage protocols, master classes, etc. We also look forward to working with industrial clients to increase the impact of new capabilities.

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Greg Haugstad, Director
Mike Boucher, Lab Manager

New Equipment at CharFac

Time-of-flight secondary ion mass spectrometer (TOF-SIMS)
SIMS analyzes and maps the elemental and chemical composition of material surfaces. A material is irradiated with a focused beam (submicron resolution) of primary ions that transfer their kinetic energy to the target atoms via a collision cascade. Atoms, clusters and molecules are emitted from the material, ionized (secondary ions) and analyzed via mass to charge ratio. Because the secondary ions originate from the outermost atomic/molecular layers of the material, the technique is highly surface sensitive. (Longer irradiation also can be used to produce depth profiles.) TOF-SIMS is based on pulsed primary ion beams. Secondary ions are accelerated and fly a substantial distance to the detector, where their intensity is measured as a function of flight time (giving mass). TOF-SIMS offers a unique combination of wide mass range, high mass resolution and high transmission (sensitivity). Available in February.

Confocal Raman microscope
Incident laser light excites molecules in a material such that the light scatters at a different wavelength. The energy difference between incident and scattered light is equal to the energy involved in changing the rotational or vibrational state of molecules or crystal lattices. The Raman spectrum is thus a nondestructive chemical fingerprint; moreover one can obtain 2D resolution down to the submicron regime (much better than FTIR). Confocal further resolves in depth to provide 3D spatial resolution. Available in May.

Temperature and humidity stage for X-ray diffractometry
Allows for controlled cooling or heating, plus relative humidity. Direct applications include (i) characterization of phase transitions, (ii) physical and chemical stability of pharmaceutical and other soft formulations, and (iii) dehydration control during heating experiments. Available in March.

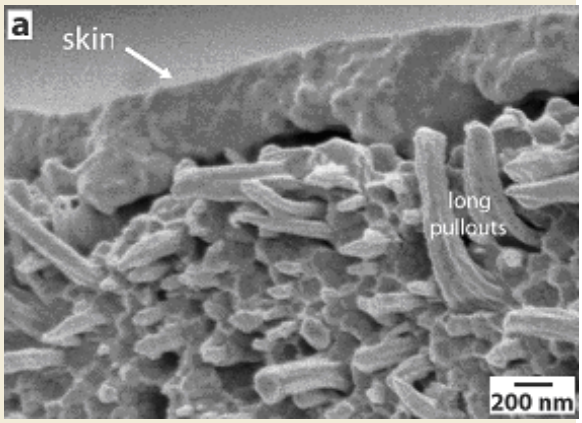
Three specimen prep tools for electron microscopy
(a) new polisher for material sciences, (b) tissue processor to embed samples in epoxy resins, reducing technical costs and increasing investigator efficiency and (c) a microwave processor to improve the penetration of fixatives and other reagents for difficult to preserve biological samples. Available in February.

CharFac Featured User and Research

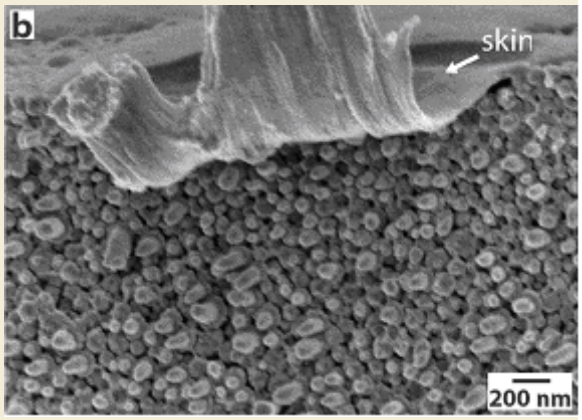
Skinning in Drying Latex Coating

Haiyan Ge, Advisors: H. Ted Davis and L. E. Scriven, Department of Chemical Engineering and Material Science
University of Minnesota

As a latex coating—a deposited layer of colloidal particles of polymer suspended in solvent—dries, it is transformed into a more or less coherent solid coating. This happens in three stages, which may overlap: (1) consolidation—solvent evaporation and particle packing; (2) compaction—particle deformation and pore-space shrinkage; and (3) coalescence—particle adhesion and inter-particle diffusion and entanglement of polymer. Skin can form at the top surface of coating in Stages (2) or (3). It can slow down the diffusion of solvent and cause serious defects such as curling and cracking in industrial coatings. Whether it forms or not depends on the polymer particles' size, surface properties, and viscoelastic properties—hence on glass transition temperature; on the coating thickness; and on the drying conditions, viz. temperature, humidity and velocity of air.



The way to visualize skinning is time-sectioning high-resolution cryogenic scanning electron microscopy, the method pioneered by Sutanto and Ma [1]. The method consists of freezing coatings at successively longer drying times, fracturing them to expose cross-sections of their interior, sublimating a small amount of frozen liquid from the fracture surface to increase topographic contrast, applying thin metal coating, and finally imaging the cross-section with a Hitachi 4700 field emission scanning electron microscope equipped with cold stages.



The Cryo-SEM images in Figure a and b show a skin was identified after the coating of 80 nm diameter styrene-butadiene latex particles with exceptional low Tg (- 65 °C) dried for 11 minutes in room air. It is thicker near the edge (Figure a) than in the center (Figure b); it grows from edge in and from top down, as seen in further Cryo-SEM images (not shown). Figure b demonstrates that skinning can form early during consolidation and compaction when latex particles in the top layers adhere strongly enough. Those particles under the skin were still consolidating as indicated by their still nearly spherical shapes.

[1] Sutanto, E., Ma, Y., Davis, T. D., Scriven, L. E. 2001. ACS Symposium Series, 790, p174.

Upcoming Events

Greg Haugstad, John Thomas, Jinping Dong and John Nelson are co-teaching the 4-credit survey course **Materials Characterization (CHEM 5210)**, offered each spring. This year's class includes enrollees from seven departments, highlighting the multidisciplinary character of the CharFac's user base (and perhaps last year's note in this newsletter!). The course has an emphasis on surface, interface and thin film analysis including far-field compositional techniques such as electron spectroscopy, secondary ion mass spectrometry and Rutherford backscattering, as well as near-field techniques like AFM and nanoindentation; plus FTIR and Raman imaging.

Farther on the horizon is the **IPRIME annual meeting** from May 30 through June 2. The CharFac will hold a nanomechanical master class (including speakers plus lab demos and attendee specimen analysis) and have a booth at the poster session/exhibit. We will also report on analytical methods development during the program reviews. This open research is in collaboration with IPRIME member companies Guidant, Surmodics and Boston Scientific, and related primarily to polymeric coatings (biomedical applications).

Tours and in some cases **demos** take place throughout the year, usually *ad hoc*. Given fundamentally new systems (e.g., TOF-SIMS and Raman), we are eager to preview capabilities; contact Greg Haugstad if interested. On-site seminars also can be arranged with industry.

NanoFabrication Center News

NFC Director's Message

NFC Director, Steve Campbell

I wanted to bring you up to date on many changes in the Lab. We recently completed a major upgrade of our LPCVD system. You may also have noticed that we recently removed the second bank of Tylan furnaces. This move was taken as a result of our upgrade to a 150 mm capability and eliminates our reliance on these systems' 1970's vintage electronics. The old Tylan bank is in the hallway at this writing, but will be shipped out soon. Since we are going from 8 tubes to 4, a small, manual furnace is being set up. This furnace will be available to anneal a wider range of materials than the high purity automated furnaces. The system should be on-line near the end of January. You may also have noticed the removal of three user-owned systems from the lab. This reflects the changing research requirements of our user base, although we try to encourage our users to remove systems no longer needed for funded research projects. The 2" high-k CVD tool in bay 4 was removed and will be scrapped. The metal CMP system in area 2 was sold to a group in Europe and has been shipped out. Finally, the plasma particle system that occupied the northeast corner of area 1 has been removed and the parts distributed back to the owners.

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Steve Campbell, Director
Greg Cibuzar, Lab Manager

Along with changing equipment, we also have to say goodbye to old friends and welcome new users and staff. Jennifer Reilly, our accountant, took as new position in the School of Public Health last fall. I hope that you will join me in welcoming Mr. Bruno Scalici as the new NFC accountant. If you have any questions regarding billing at the lab, please contact Bruno at 624-5263. We also have many new users, but let me single out Mr. Peter Eames from Non Volatile Electronics. Pete is using the Raith to create nanoscale magnetic structures.

Photomask Services

Many academic institutions and companies have some capability for thin film processing, such as deposition, etching and lithography. Photomasks, a key component of the lithography process, often consist of a glass plate with a patterned layer of chromium metal. The pattern corresponds to the design of the layer currently being processed. The fabrication of photomasks requires specialized equipment that many research labs do not have. At NFC we have the capability to make photomasks with feature sizes down to 1.5 microns. The masks can be made on soda lime or quartz plates, with mask sizes as large as 8 by 10 inches (4 and 5 inch square plates are stocked). Both darkfield and lightfield masks can be fabricated. The cost for academic institutions for darkfield masks is $210 (higher for more complex designs), and lightfield masks cost $230. If you are interested in learning more about having photomasks made at NFC, please send an email to nfcmasks@umn.edu.

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.

NFC Featured User and Research

Nano Spintronic Devices and Systems

Hao Meng, Jianguo Wang, Yunfei Ding and Xiaofeng Yao
Research Directed by Prof. Jian-Ping Wang

Nano Magnetic and Spintronic Laboratory (NMSL) led by Prof Jian-Ping Wang has recently demonstrated how nano spintronics technology can be used to produce **programmable spin logic gates**, including AND, OR, XOR, NAND, and NOR gates, just based on a single magnetic tunnel junction (MTJ) element, as well as **a full adder** using only seven MTJ elements. Prof. David Lilja's group has started a design and new microachitectures for constructing **a complete processor** that exploits the unique characteristics of these spintronic devices.

Associate Professor JianPing Wang of the University of Minnesota's department of Electrical & Computer Engineering

Spintronics is a new field of research to use both electron features: spin and charge. Application of spintronics has been just triggered by the recent advance of nanostructured materials and devices. The 2005 *International Technology Roadmap for Semiconductors* projects that by approximately 2020, charge-based electronic devices will reach fundamental limits of speed, size, energy, and noise immunity. S*pintronics* devices, in contrast, store state information in the up or down spin of the electrons. The state can be changed by controlling the magnetic fields generated by currents or spin currents on the device's input lines. These spin states can be mapped to the binary states of the logic components used to construct a digital computing system. Thus magnetic random access memory (MRAM) provides non-volatile storage and is expected to operate faster, consume less power, eliminate soft error problems, and be more resistant to all types of noise than conventional charge-based devices.

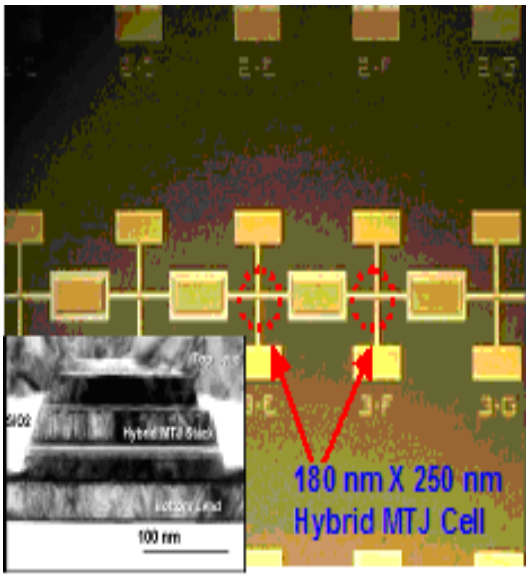

Fig 1. Prototype of a spin transfer hybrid MTJ cell array. Inset shows a cross TEM image of hybrid MTJ cell. (Wang, MRS Fall Meeting 05)

In contrast to traditional field-based MRAMs, Wang's team is investigating MRAM using spin transfer writing scheme (current-induced-magnetization-switching) which can provide several additional benefits such as better scalability, lower write power, higher speed, and enhanced robustness. His team has recently proposed and fabricated an MTJ-based spin transfer device prototype with low writing threshold (Fig.1). Low critical switching current density and thermal stability for such device were achieved (Fig.2). A first prototype of a spin transfer device with perpendicular anisotropy has also been demonstrated by Wang's group, which can support 50 Gbit/in^2 MRAM in the future.

Last but not least, integrated with his recently developed novel magnetic nanoparticles, Wang's team is also developing novel spintronic devices array with ultra high sensitivity for biomedical targets detection.

A unique etch-back type electron beam lithography process and an advanced phase shift mask technique have been developed by Wang's group to make sub 100 nm – several 100 nm spintronic devices based on NFC facilities.

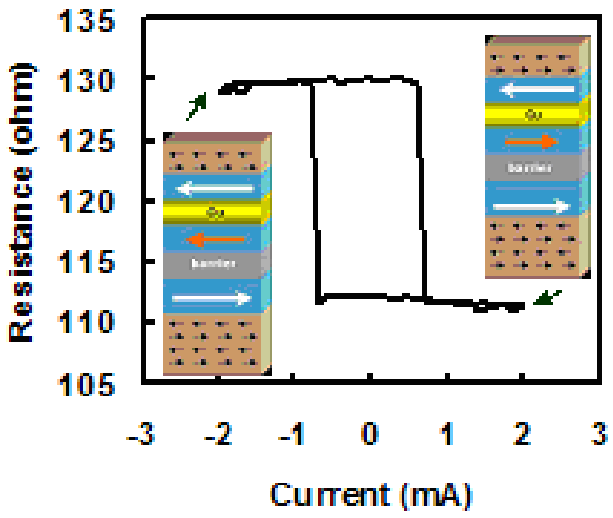

Fig 2 Spin transfer induced magnetization-switching loop for a 180 nm x 250 nm hybrid MTJ cell (Meng and Wang, APL 05, in press)

PARTICLE TECHNOLOGY LAB NEWS

PTL DIRECTOR'S MESSAGE

Distinguished McKnight University Professor, David Y.H. Pui

The 2nd International Symposium on Nanotechnology and Occupational Health (October 3-6, 2005), hosted by the Minnesota Node of the NNIN, was a resounding success. The Symposium attracted 416 registrants from 20 countries, about 22% from outside of the U.S. In addition, approximately 150 attendees also registered for the tutorial sessions offered at extra fees. Several of the tutorials, e.g., Basic Nanotechnology (M. Roco), Fundamentals of Nanotoxicology (K. Donaldson), Nanoparticle Aerosol Behavior (W. Hinds), and Basic Principles of Occupational Health and Safety (G. Ramachandran), drew more than 100 attendees. The success of the tutorial sessions demonstrated the desire of the industrial participants to learn more about health effects and control of nanoparticle exposure. The 4-day Symposium consisted of 1 day of tutorials, 2 days of technical sessions with more than 115 presentations, and 1 day of industrial forum. Many ideas on information exchanges and exposure control were generated by the participants of the industrial forum, which was preceded by program overviews presented by representatives from government agencies and research institutes.

The 3rd International Symposium on Nanotechnology and Occupational and Environmental Health will be held in Taipei, Taiwan during the summer of 2007. This will expand the global scope of the Symposium (1st and 2nd Symposia were held in U.K. and U.S., respectively) and will highlight Asian nanotech communities' efforts in addressing the potential implications and applications of nanotechnologies in the workplace.

Meanwhile, a Special Symposium on Nanoparticles and Occupational Health will be held this year at the International Aerosol Conference (IAC 2006), September 10-15, 2005, in St. Paul, Minnesota. The IAC is held once every four years. We expect 1,200 aerosol researchers and practitioners will attend this Conference. To receive more information on IAC 2006, please check the website www.aaar.org/iac2006.

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Mark Stolzenburg, Lab Manager

INTERNATIONAL AEROSOL CONFERENCE 2006

September 10 – 15, 2006
Saint Paul, Minnesota
http://www.aaar.org/IAC2006/index.htm
Sponsored by: American Association for Aerosol Research (AAAR)
On behalf of: International Aerosol Research Assembly (IARA)

Abstracts are now being accepted (submission deadline: February 1, 2006)
Go to http://www.aaar.org/IAC2006/abstract.htm for more information and to submit an abstract.

The conference will include a variety of programs and activities for attendees:

- **Scientific Programs** such as tutorials, lectures, and symposia.
- **Social Activities** including a special offsite dinner and a variety of cultural, culinary, educational, and leisure activities planned by the Local Arrangements Committee.
- **Exhibitions** of instrumentation and other aerosol/powder research-related goods and services. For a list of exhibitors, go to: http://www.aaar.org/IAC2006/exhibitorinfo.htm.
- **25th Anniversary Celebration** of the AAAR.
- **Fuchs Award** recognizing outstanding original research contributions to the field of aerosol science and technology – considered the highest honor for researchers in the field.
- **International Aerosol Fellow Award** presented by the International Aerosol Research Assembly (IARA).

For information about **registration**, please visit http://www.aaar.org/IAC2006/registration.htm

PTL Featured User and Research

Development of a New Atomizer for Producing Nano Size Droplets

John Scattergood, LaGrange, Georgia

The fast-evolving nanoparticle market is driving an increasing demand for new and improved nanoparticle generation techniques. Atomization and drying of solutions and suspensions is a common way to generate fine particles. However, initial droplet size and solvent purity limit the useful particle size range of current methods to the upper end of the nanoparticle range.

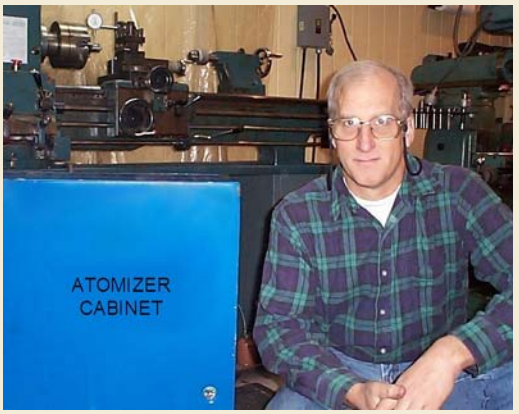
Through a combination of eclectic work experience and insight, it became apparent to John Scattergood that a scale modeling technique used in civil engineering could be used to design a new atomizer. Using this technique he is developing an atomizer based on different fluid mechanics principles than those used in current aerosol generation devices. It will atomize a liquid, converting the bulk liquid mass into nano size droplets.

At the University of Minnesota Particle Technology Laboratory scanning mobility particle sizers are used to characterize the particle size distribution produced by the new atomizer. This information is then used to analyze performance of the instrument and assist in design changes.

One application of this technology is the manufacture of pharmaceuticals. Approximately 40% of the new pharmaceuticals are poorly water soluble (< 1 mg/ml). When formulated as a nanoparticle, these compounds dissolve more readily resulting in pharmaceuticals of improved bioavailability, more consistent bioabsorption, and lower potential for toxicity. Additionally, pharmaceuticals formulated as nanoparticles may allow the rejuvenation of discarded chemistries and patent extensions of existing products.

This atomization method produces particles finer than ball or jet milling without the contamination issues. There is no need for solvents as with super critical processing or other liquids as with homogenization. Furthermore, the atomization technique permits controlled cooling rates which allows the manufacture of particles containing amorphous or crystalline microstructure.

This technique's capacity to manufacture nanocrystalline or amorphous metallurgical nanoparticles is beneficial. Applications include: propellants (explosives and rocket propellants), catalysts, magnetic materials, and nanocrystalline materials for hydrogen storage as metal hydrides. Other applications include atomization of fuels prior to combustion and agriculture spraying.



***John Scattergood**, career researcher turned independent entrepreneur, is designing and building a new atomizer based on a novel approach.*

Upcoming Events

April 24-25, 2006
Overcoming Obstacles to Effective Research Design in Nanotoxicology
Cambridge, MA
http://www.tfilearning.com/tfi/c/portal_public/layout?p_l_id=27.48

August 14-16, 2006
Aerosol and Particle Measurement Short Course
Minneapolis, MN
For description of last course offering, see
http://www.me.umn.edu/education/shortcourses/apm_course.shtml

August 17-18, 2006
Air and Gas Filtration Short Course
Minneapolis, MN
For description of last course offering, see
http://www.me.umn.edu/education/shortcourses/agf_course.shtml

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

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Published 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

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