NANOTECHNOLOGY NEWS FROM THE UNIVERSITY OF MINNESOTA

UNIVERSITY OF MINNESOTA

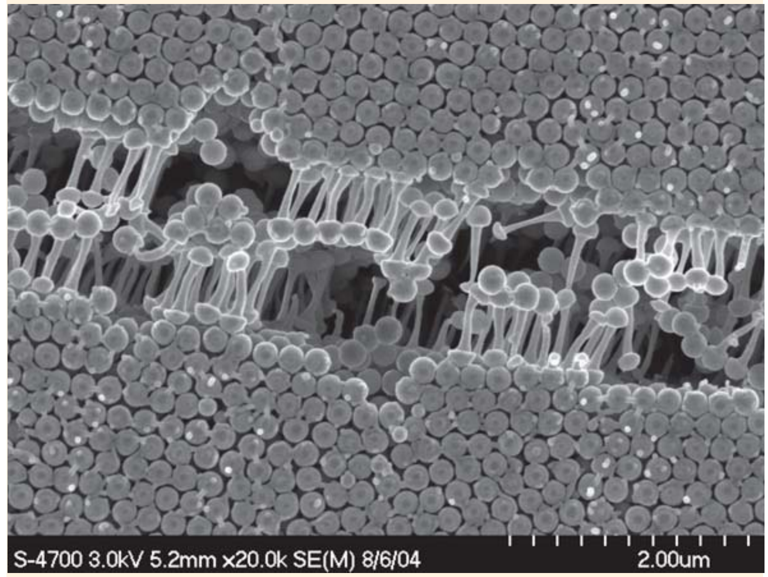
ISSUE SEVEN FALL 2006

Welcome to the Fall 2006 National Nano Infrastructure Network (NNIN) / Nano Coordinating Office (NCO) newsletter. The purpose of this newsletter is to highlight the nano-related capabilities of the University of Minnesota facilities, present a brief introduction to a few of our users, and inform you of future events.

NNIN is currently in its third year of operation. The grant is expected to run for five years, with renewal for another five years considered very likely. This will be the first year that the nodes will be evaluated, with changes in funding based on their performance. In most areas, Minnesota has done well, with a very large number of users. Our largest problem is a paucity in the number of external users, particularly academic researchers. Unlike Stanford and the east coast schools, we are not in a region of the country with a high concentration of research universities, so attracting this type of user is difficult. Furthermore, because we have so many internal users, the ratio of external to internal, which NSF uses as a primary yardstick of our success, is hard to improve. Please recommend the nano labs to colleagues – send Becky (vondi001@umn.edu) contact information for anyone who might be interested in our facilities.

This year will see a change in the NCO as well. The University has allocated funding to improve the competitiveness of its researchers working in Nano. The nature of these changes has not yet been fully worked out. Look for an update in the January newsletter.

FALL 2006 NANO IMAGE



Crack in latex coating induced by plunge freezing into liquid ethane. Latex particles are elongated to form pull-outs. Some of them break into two parts. Published in *Macromolecules* **2006**, *39*, 5531-5539. Courtesy Xiaobo Gong and Professors L. E. Scriven and H. Ted Davis.

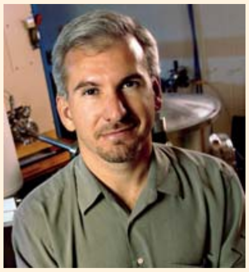
Reminder: If your work uses CharFac, NFC, or PTL, please add the following in the acknowledgements section of any publication: "Parts of this work were carried out in the Minnesota (Characterization Facility, Nanofabrication Center, or Particle Technology Lab) which receives partial support from NSF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

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

NSF OVPR INSTITUTE OF TECHNOLOGY engineering | physical sciences | mathematics

CHARACTERIZATION FACILITY NEWS

CHARFAC DIRECTOR'S MESSAGE



CharFac Director,
Greg Haugstad

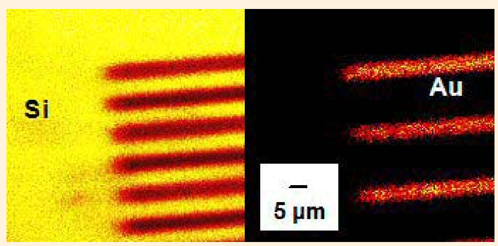
We are excited to announce a new era of Raman microscopy, one of several developments providing *spectroscopic chemical imaging*. In Raman spectroscopy, irradiation excites chemical vibrations. Changes in the wavelength of the scattered light appear as spectral peaks corresponding to different energies of excitation. Rather than image one excitation, our new Witec confocal Raman microscope allows collection of a *full spectrum* at each pixel location, and in a matter of minutes. Thus complex spectral fingerprints can be rendered in images. User training in Raman is now being scheduled with staff member Dr. Jinping Dong. Raman analyses have been performed on specimens such as polymer-drug complexes, carbon nanotubes in polymer matrices, geological silicates, nanoparticles and self-assembled monolayers. Some of these first results are featured on the next page.

Keeping with the theme of chemical imaging, we highlight other additions to the CharFac: time-of-flight secondary ion mass spectrometry (ToF-SIMS), atomic force microscopy in temperature-controlled digital pulsed force mode (T-DPFM), and energy-filtered cryogenic transmission electron

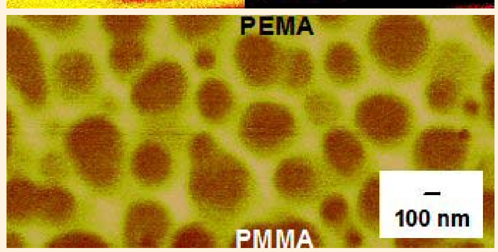
(continued, top right)

microscopy (cryo-TEM). The ToF-SIMS images below (top) are of gold and aluminum lines grown on silicon. In addition to selected elemental masses (bright), one can discriminate other mass fragments at the surface (e.g., organic contamination). As with Raman, full spectroscopic imaging can be performed: a broad mass spectrum acquired at each location.

Whereas Raman and ToF-SIMS can spatially resolve down to a few hundred nanometers, AFM in T-DPFM can resolve below 100 nm. The tip-sample adhesion image (below bottom) is of a phase segregated blend of poly ethyl/methyl methacrylate (PEMA/PMMA) heated above the glass transition temperature (T_g) of PEMA such that its "stickier" (brighter) domains are distinguished from the glassy PMMA. "Force spectroscopy" imaging also can be performed by measuring the distance-dependent tip-sample force at each pixel location. Such detailed analyses of approach-retract cycles may be needed to clarify contrast mechanisms.



ToF-SIMS images of gold and aluminum lines grown on silicon. Si and Au atomic mass images shown.



Tip-sample adhesion image of a phase segregated blend of poly ethyl/methyl methacrylate (PEMA/PMMA) heated above the glass transition temperature of PEMA.

Finally, a new 300-kV cryo TEM (FEI) will be operational by early 2007. This system includes 3D reconstruction and energy-filtered images (energy loss as electron beam traverses specimen) to better control chemical contrast. Check for more details in our winter newsletter.

CHARFAC 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

STAFF NEWS

Dr. Stuart McKernan has taken a position in the central analytical labs of 3M. We wish Stuart well in his future endeavors. Upon defending, Jingshan Dong will be joining the CharFac in electron microscopy with an emphasis on cryo TEM. Dr. Bing Liu is a new postdoc working half-time in the CharFac in surface analytical techniques (XPS, Auger, ToF SIMS).

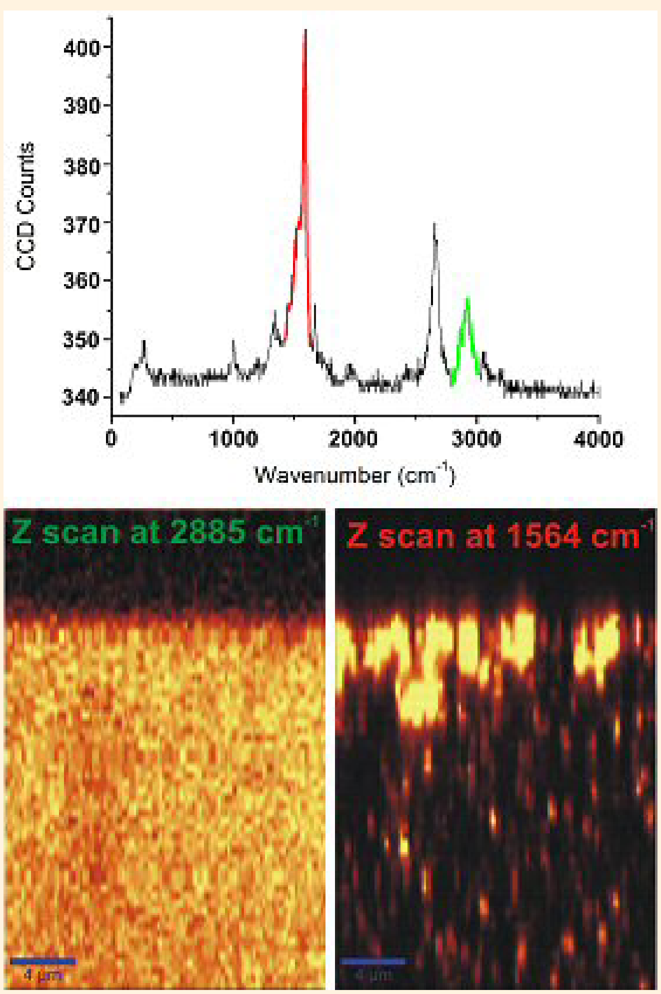
Determining the Orientation and Dispersion of Electro-oriented Carbon Nanotubes Using Raman Microscopy

Randy A. Mrozek, Advisor: T. Andrew Taton, Department of Chemistry, University of Minnesota

Single-walled carbon nanotubes (SWNTs) are conductors, and composites of SWNTs and commodity polymers are conductive, processible materials that are useful for a variety of technological applications. Because SWNTs have such large aspect ratios, they form percolating, conducting networks through the composite at very low (< 1 wt %) loadings. Conductivity can be achieved at even lower loadings if the molten composite material is poled in an electric field—such that the nanotubes are oriented to electrically "short" the field gap—before it is cooled into its desired form.

Randy Mrozek
Department of Chemistry
University of Minnesota

Randy Mrozek, a graduate student working with Professor Andrew Taton in the Department of Chemistry, has recently used the new WITec confocal Raman microscope to characterize the dispersion and orientation of nanotubes in poly(styrene-isoprene-styrene) (SIS) thermoplastic elastomers. The instrument collects Raman spectra from individual, finite volumes and then maps specified peak intensities as two- or three-dimensional images. The Raman spectrum of the bulk composite shows distinct peaks that correspond to SWNTs (*G* band, 1500-1600 cm^{-1}) and polymer (C-H, 2775-3000 cm^{-1}). As a result, it was possible to construct independent Raman images of the two components of the material. Because the microscope is confocal, it can image deep into the material, and can collect Raman spectra as a function of depth (Z-scan) or as X-Y cross-sections at a specific depth. As expected, the image of the polymer component showed equal concentration throughout the sample. The image of the nanotubes, on the other hand, showed two interesting features: first, that the nanotubes were successfully oriented by the applied field, as shown by the vertical streaks in the SWNT Z-scan image; and second, that the field had also attracted the dispersed SWNTs to the sample surface. The intense Raman signal from the nanotubes makes confocal Raman microscopy an ideal tool to pinpoint the location and orientation of SWNTs in the composite material. Randy expects to use this technique to help determine the field requirements to produce alignment, minimize migration, and lead to the production of bulk aligned materials that exhibit anisotropic electrical and other material properties.



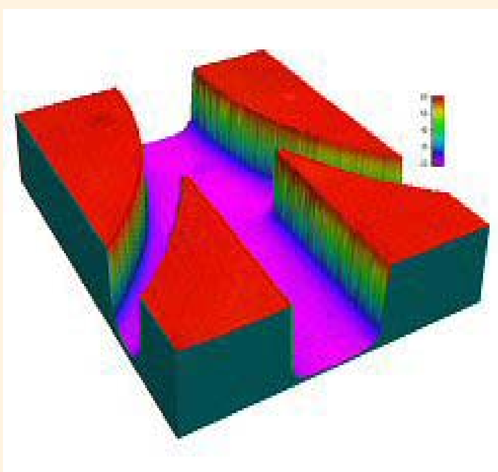
Top: Raman spectrum of a SWNT-polymer composite. The red peak is specific to SWNTs, and the green peak to the polymer matrix.
Bottom left: Z-scan (vertical slice) image of the polymer component.
Bottom right: Z-scan of dispersed SWNTs.

NANOFABRICATION CENTER NEWS

NFC DIRECTOR'S MESSAGE

NFC Director,
Steve Campbell

This has been a rather quiet period in NFC with regard to significant changes in equipment or staff. Our Lab Manager, Greg Cibuzar, discusses planned upgrades to our microfluidics capabilities in his column in this section. In addition, as part of a successful $150,000 NNIN Major Research Instrumentation Award obtained from NSF this spring, we will be taking delivery of a new HS200A Optical Profiling System. This system uses confocal microscopy and analysis techniques to enable non-contact 3-dimensional surface imaging with submicron horizontal resolution and a vertical resolution of several nanometers. The system will have



Confocal image of a microfluidic flow channel from H-S.

(continued, top right)

an automated x-y stage for detailed mapping of the surface. The system will be an important tool for anyone interested in MEMS, microfluidics, critical dimensions, resist and etch profiles, defect characterization and film topography. We expect the system to be available later this fall. Go to www.hyphenated-systems.com for more information.

MICROFLUIDICS PROCESSING

Microfluidics is the name given to fluid-based devices and systems with features at the microscale. Examples of microfluidic devices include miniature capillary electrophoresis arrays, DNA amplification systems, and blood processing devices. Fabrication of these types of devices can be done using materials such as silicon, silicon dioxide, and various glasses, or organic polymers such as PDMS, polyimide and SU-8. In conjunction with a group of faculty members, NFC staff members are working to develop processes for the fabrication of microfluidics devices using a variety of materials. As part of this effort, we are adding equipment. Recently we placed orders for two pieces of equipment that are critical to bonding the components of the microfluidics device together. The first is a visible light flip chip bonder. This tool will enable the alignment and bonding of the device components to a positional accuracy of two microns, and can handle samples to 100mm square, with maximum temperature and applied pressure of 400° C and 20kg equivalent. In order to improve the bonding of polymers, we are also purchasing a UV processing chamber to treat the surfaces of polymers with ozone prior to bonding. If you need assistance with fabrication of microfluidic devices, please contact us for help.

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 of the month sessions begin at 10:00AM. The training includes watching our safety video and taking a brief quiz. Also, a NFC staff member provides a tour showing some of the 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 two hours to complete, and must be done before users will be granted access to NFC facilities.

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

NFC Featured User and Research

Polymer MEMS and Microfluidic Biochip

Professor Euisik Yoon, Dr. Hyung-Kew Lee and Young-Ji Kim
Department of Electrical & Computer Engineering, University of Minnesota

Professor Euisik Yoon of the University of Minnesota's Department of Electrical & Computer Engineering

Professor Yoon's research interest is on the integration of NEMS/MEMS devices with interface circuitry on a single-chip platform. He joined the University's Department of Electrical and Computer Engineering in the fall of 2005. Since then, he has established a few biomedical applications of flexible polymer sensors and microfluidic devices, which have been fabricated at the Nanofabrication Center (NFC). Recently, his group has successfully implemented the multiple stacking of polymers with embedded copper interconnection layers to build an 8 X 8 tactile image sensor array for three-axis contact force measurements (**Fig. 1**). This sensor allows the mapping of stress distribution normal to the sensor surface as well as the shear stress applied in the same plane. Professor Yoon is exploring many interesting applications of this device such as artificial human skins in remote surgery, virtual reality and transposing sensory systems for the impaired.

The microfluidic biochip is another thrust of his group's efforts to realize the cell-based assay on a chip (**Fig. 2**). This prototype device allows a high-throughput drug (or reagent) screening for the cells captured in a two-dimensional microwell array by selectively delivering a drug (or reagent) to the target microwells. This chip employs the full electrical control which has the capability to manipulate cells at single cell resolution by using electromagnetic forces. The initial applications of the device will focus on muscle stem cell differentiation studies and cancer cell drug screening. This effort involves collaboration with researchers in the Stem Cell Institute and Medical School here at the University. By combining Professor Yoon's previous industrial experience in Silicon Valley with the interdisciplinary collaboration of researchers at the Medical School, it is expected that some of the microfluidic devices coming out of his group's work will be used in practical biomedical applications.

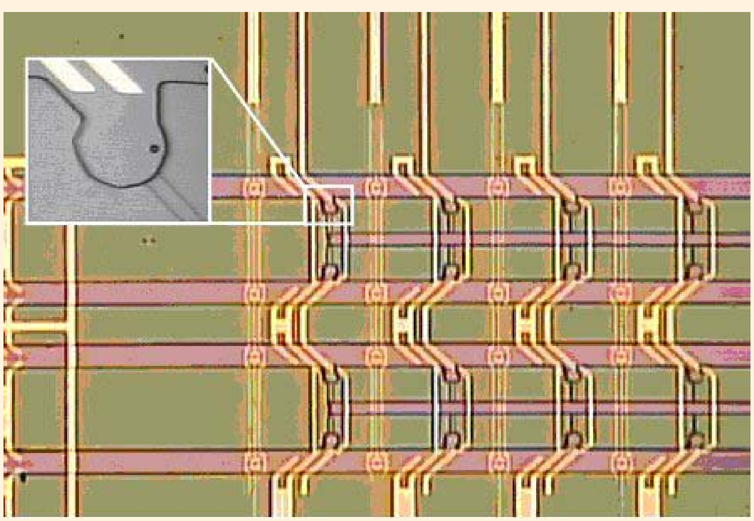


Fig. 1 Flexible polymer tactile sensor array

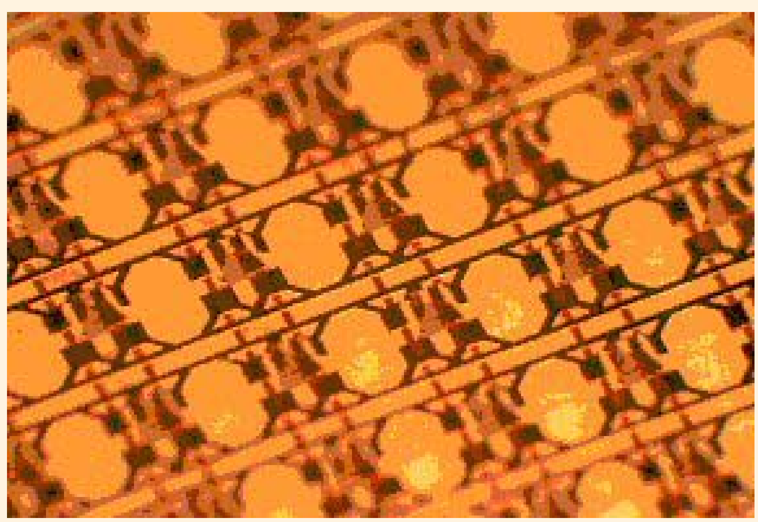


Fig. 2 Microfluidic biochip for cell-based assay

Particle Technology Lab News

PTL Director's Message

Distinguished McKnight University Professor, David Y.H. Pui

We have just completed two successful short courses: Aerosol and Particle Measurement (August 14-16, 2006) with 52 registrants, and Air and Gas Filtration (August 17-18, 2006) with 41 registrants. This is the 31st offering of the Measurement course and the 10th offering of the Filtration course. This year's courses attracted registrants from Norway, Switzerland, Korea, China, England, Germany, Canada and the United States. The Measurement course had two afternoons dedicated to laboratory sessions at the Particle Technology Laboratory and at TSI Inc., a co-sponsor of the course. Registrants learned the latest advances in instrumentation and measurement techniques. With 21 lecturers from around the country and overseas, it was our annual reunion among friends to reminisce and to discuss new developments in the field. The registrants have expressed their satisfaction with the course offerings. We have found consistently year after year that 80% of the registrants come as a result of recommendations by their colleagues who had attended previously.

Our focus now turns to the hosting of the International Aerosol Conference, September 10-15, 2006, in Saint Paul, Minnesota *(continued, top right)* (http://www.aaar.org/iac2006). We expect to have more than 1,200 registrants from 40 countries attending this conference. IAC 2006 is the main event for international aerosol associations and is held once every four years. Aerosol is an enabling discipline with many applications, including nanotechnology, semiconductor manufacture, air pollution, bioaerosol, global climatic effects, emissions from energy technologies, and environmental health and inhalation therapy. The attendees will come from academia, industry and government sectors. There will be nanotechnology sessions throughout the five-day conference, and a technical tour of the Particle Technology Laboratory, for which the 200 available reservations have already been filled. The Particle Technology Laboratory, a component of the Minnesota node of the National Nanotechnology Infrastructure Network (NNIN), is both a host and a sponsor of the event. We will distribute printed NNIN flyers and newsletters to the IAC registrants. We believe this conference will help inform many registrants of the NNIN in general and the Minnesota node in particular.

Particle Technology Lab at the University of Minnesota

Mechanical Engineering 3101
111 Church Street SE
Minneapolis, MN 55455

www.me.umn.edu/labs/ptl
Main telephone: 612-625-8354
Fax: 612-625-6069

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

IAC 2006

The 7th International Aerosol Conference (IAC) will be held September 10-15 at the Radisson Riverfront Hotel in Saint Paul, Minnesota. The conference, co-chaired by David Pui of the PTL and Gilmore Sem of TSI, Inc., is sponsored by the American Association for Aerosol Research (AAAR) on behalf of the International Aerosol Research Assembly (IARA).

The conference will feature:

- over 1,150 high-quality papers on the latest aerosol science and technology
- several specialty symposia including a series of four on nanoparticle-related topics
- 16 tutorials taught by experts in the field
- 23 exhibitors of instrumentation and other aerosol research-related goods and services

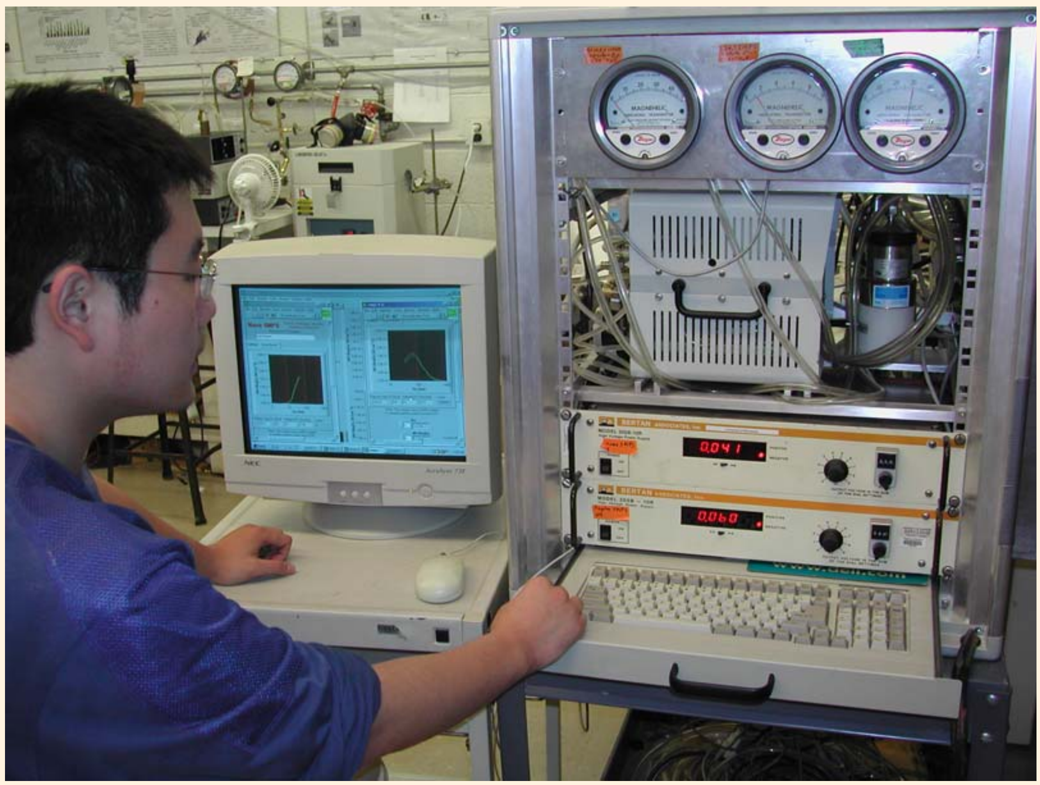
A tour of university particle-related research will include three world-renowned centers - the Particle Technology Laboratory, the Center for Diesel and Renewable Fuel Research and the High Temperature and Plasma Laboratory - all located within the Department of Mechanical Engineering.

A Novel Templating Route Towards Shaped Silica Nanoparticles With Mesoporosities

Fan Li, Advisor: Andreas Stein, Department of Chemistry, University of Minnesota

Silica nanoparticles with mesoporosity are of great interest due to their potential applications in enzyme encapsulation, drug delivery and as cell markers. They can also serve as basic building blocks for more complicated porous materials and as hard templates for porous structures with other compositions. Typically, the preparation involves spontaneous nanoparticle growth with supramolecular templating. However, due to the complex, multiphase interactions in such systems, process optimization to obtain size and shape control is challenging. In addition, particle agglomeration usually is a big concern in this context.

Self-assembly is a simple but efficient way towards 3D nanostructured materials with different morphologies. Many complicated structures with perfect ordering can be achieved from the assembly of simple units. In this project, we started from a polymeric template via self-assembly, and used it to control the silica evolution. Through a template-controlled evolution, silica nanoparticles were formed with well-defined shape and size distribution. Because the morphology evolution of nanophased silica is very delicate and extremely sensitive to the experimental conditions, a detailed characterization is indispensable for this research.



Third year graduate student Fan Li of the University of Minnesota's Department of Chemistry

At the University of Minnesota Particle Technology Laboratory, a liquid suspension of the solid particles is aerosolized and the liquid evaporated. The resultant airborne nanoparticle size distribution is characterized with a scanning mobility particle sizer (SMPS). The resolution and accuracy of the SMPS is better than 5% for particle diameter and 10% for concentration. It can readily resolve the dried silica particles from the generally smaller non-volatile residue particles coming from droplets not containing silica particles. This technique possesses low-detection limit at ppb level with capability down to several nanometers. Thus the silica particle evolution process can be carefully monitored, which is very supportive to the mechanism analysis and also facilitates the process optimization.

In summary, the templating-based strategy guaranteed uniform dimensions and specified size distribution for the resulting silica particles and eliminated the agglomeration effect common in traditional syntheses. The exploration of different structural features with a variety of materials is very promising.

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 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 this 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 Nanofabrication Center (NFC) is an interdisciplinary facility supporting faculty and industrial research within the Institute of Technology to foster education, research and industrial collaboration in microelectronics and other related research involving nanofabrication. Capabilities include:

- E-Beam, Ion Beam & Mask Making
- Optical Photolithography
- Chemical Vapor Deposition
- Metalization & Sputtering
- Dry Etching
- Annealing, Oxidation & Doping
- Wafer Bonding, Polishing & Sawing

The Particle Technology Laboratory mission is to foster research and educate students and the greater community in the areas of aerosol and small particle research and instrumentation. Major facilities of the laboratory include:

- Cleanroom, wind tunnel, filter testing, plasma and vacuum facilities.
- Instruments for particle generation, measurement, sampling and analysis in the 0.002 to 100 um diameter range.
- Gas-, liquid- and surface-borne particle measurement instrumentation
- Single-particle mass spectrometer
- Bioaerosol sampling and measurement capabilities

Nanotechnology News from the University of Minnesota

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

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