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NANOTECHNOLOGY NEWS FROM THE UNIVERSITY OF MINNESOTA

FALL 2009

NATIONAL NANOTECHNOLOGY INFRASTRUCTURE NETWORK FEATURED RESEARCH

Nanowires for Solar Energy Conversion and Energy Storage

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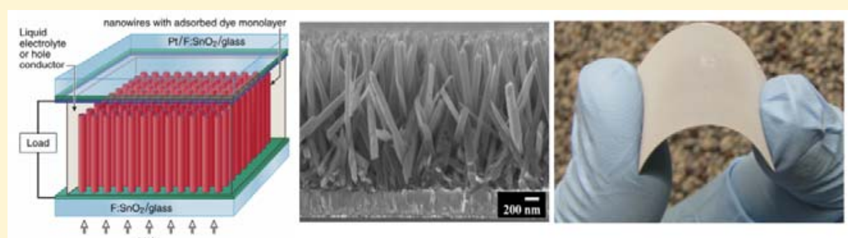
Chemical Engineering and Materials Science (CEMS), University of Minnesota

Nanowires are emerging as useful components in solar cells and lithium-ion batteries (LIB) because they have unique combination of properties such as large surface-to-volume ratios, high charge conductivity and ability to accommodate strain without material degradation. At UMN, Prof. Aydil and his students have been developing methods for synthesizing ZnO and TiO_2 nanowires on a variety of substrates for dye-sensitized solar cells (DSSC), quantum-dot solar cells (with Prof. Norris, UMN CEMS) and LIBs (with Prof. Jim Yang Lee, National University of Singapore).

DSSCs are promising alternatives to the conventional solar cells because they can be fabricated from inexpensive materials with low cost processes. The key component in a nanowire-based DSSC (see Figure) is the photoanode, which is made by growing nanowires on a transparent substrate. A monolayer of a dye adsorbed on the nanowire surfaces absorbs the light and generates photoexcited electrons, which are injected into the nanowire to provide photocurrent. The best material for DSSCs is TiO_2 but there was no way to grow single-crystalline TiO_2 nanowires on transparent substrates. Recently, the UMN group achieved liquid-phase epitaxial growth of single crystal TiO_2 nanowires on TCO-coated glass substrates and demonstrated 3% efficient solar cells.



The ideal LIB electrodes must have large surface area and must be able to repeatedly accept and reject a large number of Li ions without significant degradation after many charging-discharging cycles. UMN group developed a simple method for growing oriented single-crystalline TiO_2 nanowire arrays on flexible foil (see Figure) and, in collaboration with the Singapore group, demonstrated batteries that can deliver a capacity of 200-250 mAh/g over as many as 200 charging cycles. The performance, the improved safety and the ability to roll foils into compact 3-D structures without additional binders or additives make these TiO_2 nanowires promising materials for LIBs.

Reminder: If your work uses the Nanofabrication Center please add the following in the acknowledgements section of any publication: "Parts of this work were carried out in the University of Minnesota Nanofabrication Center which receives partial support from NSF through the NNIN program."

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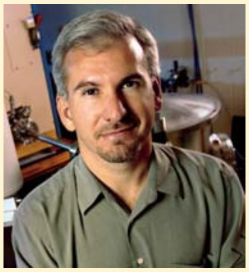
CHARACTERIZATION FACILITY NEWS

CHARFAC DIRECTOR'S MESSAGE



CharFac Director, Greg Haugstad

A Gatan Enfina Electron Energy Loss Spectroscopy (EELS) system has been installed on our 300-kV high resolution transmission electron microscope (FEI F30) in Shepherd Labs. In TEM, the incident electron beam interacts with the sample such that some electrons undergo inelastic scattering and lose energy. EELS measures the *amount* of energy loss. These losses may involve plasmon and phonon excitations, inter and intra band transitions, and inner shell ionizations. By analyzing the EELS spectrum one can obtain information on *electronic structure, bonding, excitations and elemental composition* of the sample.

DigiScan from Gatan also has been installed along with the Enfina. With Digiscan and Enfina one can do EELS *Spectrum Imaging* (SI), where a data cube is created including the EELS spectral energy scale along with the (X,Y) coordinates of each image each pixel location. EELS SI is a highly useful for many advanced applications such as identifying chemistry changes along an interface. Small area mapping also can be performed using EELS SI. The high resolution imaging capabilities of the F30 TEM and the EELS SI allow atomic scale chemical analysis. Because DigiScan

(continued, top right)

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Greg Haugstad, Director

The Gatan Enfina Electron Energy Loss Spectroscopy (EELS) system is now operational on the 300-kV high resolution TEM (FEI F30) at the Characterization Facility's Shepherd Lab location.

controls the scan coils, Scanning Transmission Electron Microscopy (STEM) now can be performed on the new (much faster) EELS computer. The old computer was too slow and tended to cause software crashes.

EELS is also a complementary technique to Energy Dispersive Spectroscopy (EDS). EDS is more sensitive to heavier elements, whereas EELS is more sensitive to light elements. Between those two techniques we are now able to cover the entire periodic table with very high sensitivity on the 300-kV F30. Additionally, the F30 in Shepherd Labs has been aligned to work at any of three accelerating voltages (100kV, 200kV and 300kV). This allows users to operate at lower voltages to protect their beam sensitive samples and still get high resolution images and use all the accessories. The energy resolution of the EELS system at 100kV is 0.45eV.

A new EDS system has been installed on the FEI T12 TEM. This system from Oxford Instruments brings state-of-the-art chemical analysis to users of a 120-kV machine. Thus TEM beginners will not have to reach the F30-level of user proficiency to bring EDS characterization into their research. The Oxford EDS system has a simple user interface, hugely improved compared to the 20+ year old system previously attached to the T12.

The new data-acquisition and goniometer-control computer for our Ion Beam Analysis lab is now installed and operational. This replaces a 15-year old 486 DOS system that, among other things, had developed problems with goniometer communication. Channeling analysis is now fully functional, and rotating random scans no longer require babysitting to catch glitches. The new system also can acquire data from all four detectors at once: normal and grazing scattering ions, X-rays and gamma rays (e.g., for Li and F sensitivity). The ability to run third party software in parallel with the data acquisition system – such as spreadsheet/graphing and data analysis programs – is a boost to analyst productivity.

NANOFABRICATION CENTER NEWS

NFC DIRECTOR'S MESSAGE

NFC Director,
Steve Campbell

This summer NFC took delivery on a new piece of equipment, a Canon 3i-2500 i-line stepper. This system provides an optical lithography capability for 4" wafers with resolution down to 400 nm. Overlay accuracy should be about 100 nm. We expect the system will accept reticles made on our existing mask making tool since the system incorporates a 4x optical reduction. The primary funding for the system was provided through the NNIN grant. The system that we bought had been used for GaAs device manufacturing.

The system has now been placed on the clean room floor, reassembled, and facilitated. Several deficiencies in the system were found during the installation process. The replacement parts to address these problems have been acquired and are expected to be installed in mid September. Once they are installed, the system will be turned *(continued, top right)*

The Canon 3i-2500 i-line stepper at the Nanofabrication Center

over to Tony Whipple of the NFC staff who will develop standard operating procedures and a training program, and Gary Olin who will develop a maintenance program. We hope to have the system available for first users by late October. Please see Tony if you want to be part of the first group being trained on the system.

FOCUSED ION BEAM TECHNOLOGY

The Quanta 200 3D is a Dual Beam (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

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 1:00PM, 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.

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Center for Nanostructure Applications

The primary mission of the Center for Nanostructure Applications is to seed interdisciplinary nano research projects that will go on to attract external support. Active nanostructures include applications of nano as diverse as energy conservation and production, large area displays and lighting, printed electronics, smart fabrics, electronic noses, drug delivery, cancer therapy, and new types of medical imaging.

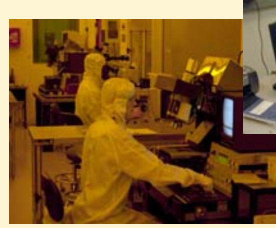
These applications often require significant participation across traditional disciplines and the Center is designed to foster the cross-disciplinary research necessary to bolster the nano applications area at the University.

The Center also organizes workshops, speaker series, and short courses, as well as serving as a focal point for nano at the University.

For more information, visit http://www.nano.umn.edu/

The National Nanotechnology Infrastructure Network





The National Nanotechnology Infrastructure Network (NNIN) is an integrated networked partnership of user facilities, supported by the National Science Foundation, serving the needs of nanoscale science, engineering and technology. The mission of the NNIN is to enable rapid advancements at the nano-scale by efficient access to nanotechnology infrastructure. The NNIN supports the Nanofabrication Center at the University of Minnesota. As a node in NSF's National Nanotechnology Infrastructure Network (NNIN), the NFC provides access to advanced multi-user facilities to both industry and academic researchers, the latter at a subsidized rate.

For more information, visit www.nnin.org