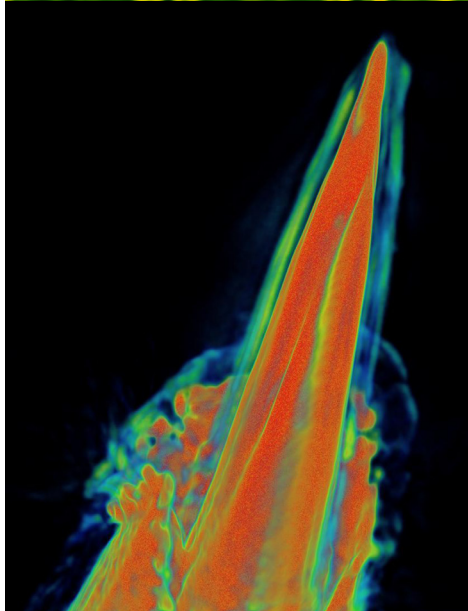
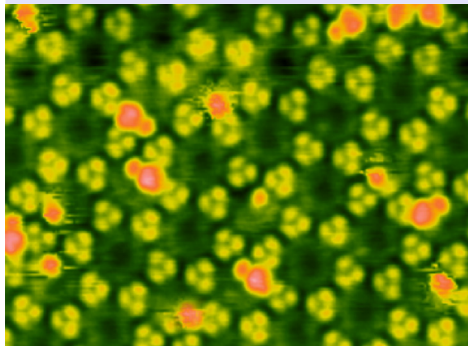
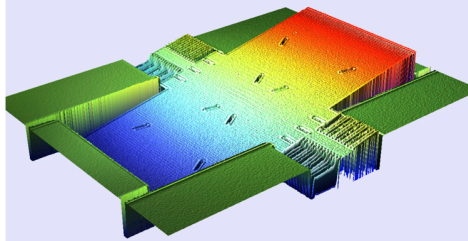
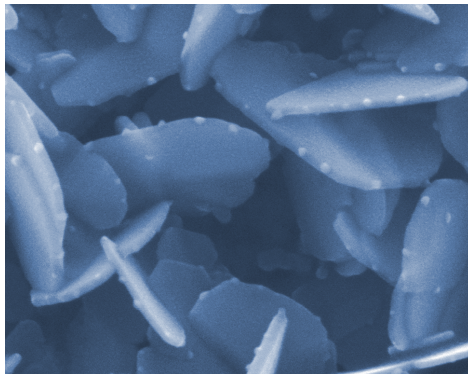


The Center for Nanoscale Materials

at Argonne National Laboratory

world-class user facility



revolutionary
breakthroughs in
nanoscience &
nanotechnology



OVERVIEW

The Center for Nanoscale Materials (CNM) at Argonne National Laboratory is a premier user facility, providing expertise, instruments, and infrastructure for interdisciplinary nanoscience and nanotechnology research. Academic, industrial, and international researchers can access the center through its user program for both nonproprietary and proprietary research.

The center's goal is to support basic research and the development of advanced instrumentation that will help generate new scientific insights, create innovative materials with unique functionality, and contribute significantly to energy-related research and development programs. High-impact staff and user science is accommodated at the CNM within the primary cross-cutting theme of "Energy and Information Transduction at the Nanoscale". There are three critical pillars underpinning this theme in the general areas of Materials Discovery, Manipulation and Visualization.

The CNM is one of five U.S. Department of Energy (DOE) Office of Science Nanoscale Science Research Centers (NSRCs) dedicated to nanoscience and nanotechnology. It was constructed under a joint partnership between the DOE and the State of Illinois, as part of DOE's NSRC program. Together the NSRCs comprise a suite of complementary facilities that provide researchers with state-of-the-art capabilities to fabricate, process, characterize, and model nanoscale materials, and they constitute the largest infrastructure investment of the National Nanotechnology Initiative. For more information about the DOE NSRCs, please visit www.nano.energy.gov.

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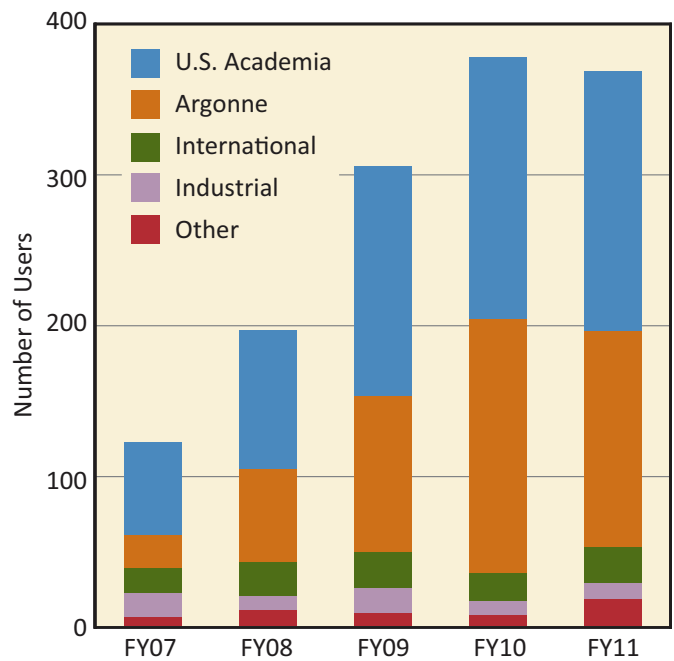
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USER COMMUNITY



The CNM user program provides access to equipment and technical expertise and is open to academia, industry, government agencies, and research institutes worldwide. Proposals are submitted electronically then reviewed and rated by an external Proposal Evaluation Board. There are three calls-for-proposals per year with nominal deadlines in March, July, and October. Proposals are active for one year or until the allocation allotment has been expended, whichever occurs first. Access is available at no charge for work that is intended for publication. Access is also available on a cost-recovery basis for proprietary research that is not intended for the public domain. Safety is an essential part of all work done at the CNM and users are required to be properly trained before working in its facilities. A Users Executive Committee acts as a liaison between the user community and CNM management, and also organizes the technical program for the annual Users Meetings.



The CNM user community grew quickly after the facility opened and has now leveled off with a healthy balance of users, reflecting the diversity and breadth of nanoscience and nanotechnology.

RESEARCH AREAS

Electronic & Magnetic Materials & Devices

The Electronic & Magnetic Materials & Devices (EMMD) Group is dedicated to discovering, understanding, and using new electron- and spin-based materials and phenomena in constrained geometries.

Potential benefits of this work include:

- ▶ Reduced power dissipation
- ▶ New medical imaging methods and therapies
- ▶ Improved efficiency of data storage by spin current and electrical field-assisted writing
- ▶ Enhanced energy conversion in photovoltaic devices

Research Activities

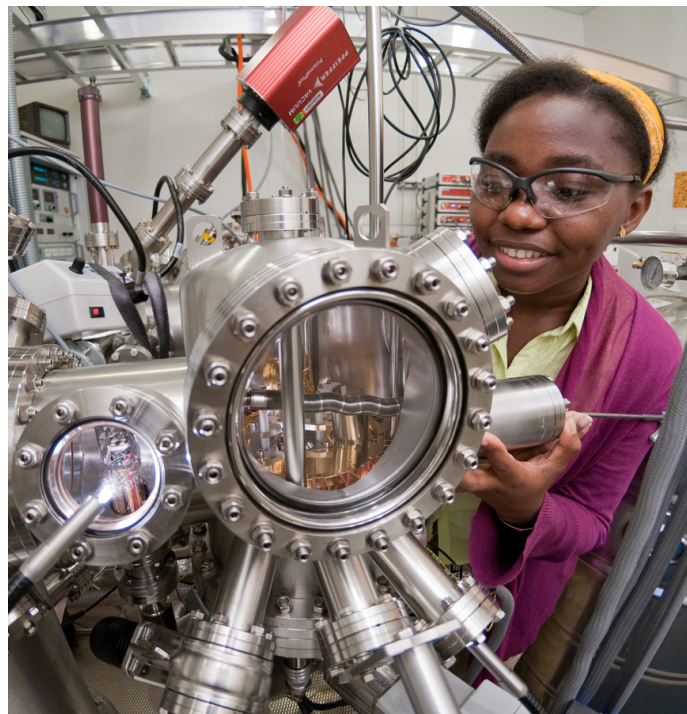
- ▶ Understanding complex magnetic order and coupling phenomena
- ▶ Exploring energy, charge, and spin dynamics in optically active nanoscale systems
- ▶ Understanding charge and spin transport
- ▶ Controlling synthesis of materials with tailored electronic and magnetic properties
- ▶ Polymeric materials and templates
- ▶ Functionalization of colloidal nanoparticles

Key Capabilities

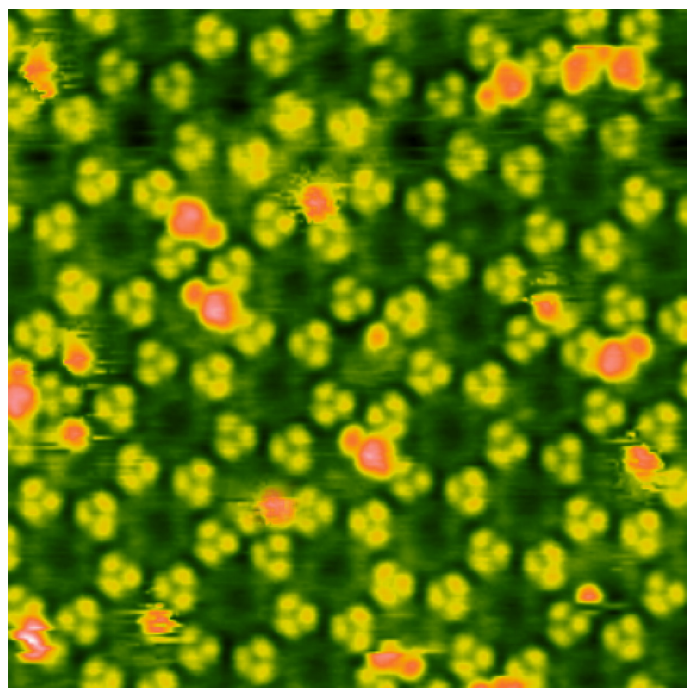
- ▶ Complex oxide digital/multilayer synthesis via molecular beam epitaxy (DCA R450 Custom)
- ▶ Characterization of electrical transport properties at the nanoscale via state-of-the-art ultrahigh-vacuum nanoprobe (4-tip SEM Omicron UHV Nanoprobe)
- ▶ Advanced scanning probe microscopy and spectroscopy at variable temperatures, including spin-sensitive imaging (Omicron VT-AFM XA, Veeco MultiMode with NanoScope V Controller)
- ▶ Variable-temperature magneto-optical microscopy with high-resolution spectroscopy capability

Research Highlight: Thinnest Nanofiltration Membrane to Date

The thinnest nanofiltration membrane achieved to date, at ~30 nm, made of just four layers of nanoparticles, was created by exploiting capabilities at the CNM. Separation membranes are a key component in both nanofiltration and reverse osmosis filtration systems. Reducing the thickness of the membrane reduces the pressure that needs to be applied across the membrane in order to achieve a certain amount of flux, which is a major operational cost in these devices. This research opens up new possibilities for using nanoparticles in nanofiltration and separation.



CNM postdoctoral fellow Esmeralda Yitamben uses a scanning tunneling microscope to track molecular motion of amino acids in chiral quantum corrals. These chiral nanoscale molecular 'race tracks' could find applications in fields as diverse as spintronics, biophysics, and astrophysics.



Surface topography of alanine molecules on Cu(111) surface. Once on the Cu(111) surface, alanine molecules form a supramolecular network of hexagonal pores. Image acquired using a variable temperature scanning tunneling microscope.

NanoBio Interfaces

The NanoBio Interfaces Group seeks to understand how deliberate tailoring of multiphase materials at the nanoscale can lead to enhanced functionalities for energy and information transduction. This work includes investigating the fundamental parameters that govern energy conversion in functionally integrated multicomponent nanoparticle hybrid systems, capable of energy storage in the form of separated charges.

Research Activities

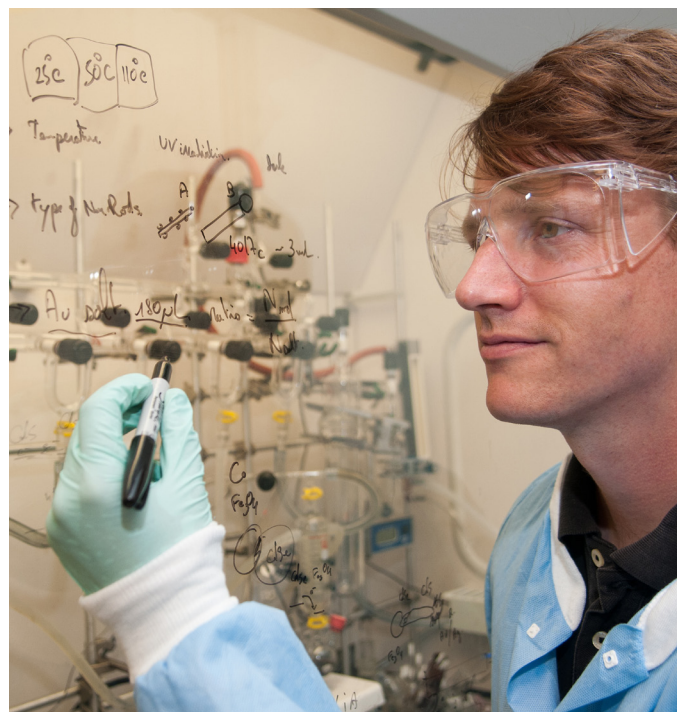
- ▶ Specialized synthesis of quantum dots (QD) and their assemblies
- ▶ Probing QD interfaces with biomolecules and solid-state matrices
- ▶ Incorporation of QD assemblies and QD bio-hybrids into devices
- ▶ Photogenerated charge separation of nano-TiO₂-based complexes
- ▶ Nanobio conjugates for mapping of ligand-receptor interactions
- ▶ Study of plasmon and exciton coupling in metal-semiconductor binary core-shell nanoparticles
- ▶ Manipulation of interparticle spacing in self-assembled highly periodic single-component structures
- ▶ Response of hybrid molecular materials to external stimuli
- ▶ Nanocatalysis and photocatalysis; nanomaterials for energy

Key Capabilities

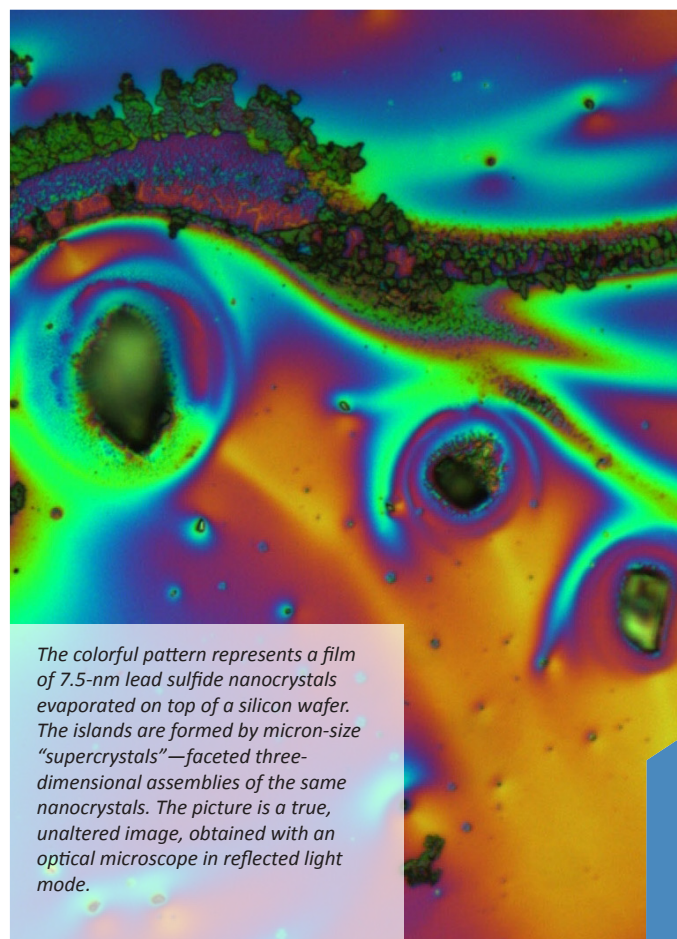
- ▶ Bioconjugation and biochemical techniques including recombinant DNA/protein techniques
- ▶ Field emission scanning electron microscopy (JEOL JSM7500F)
- ▶ Laser scanning confocal microscopy (Zeiss LSM 510 Meta)
- ▶ Transmission electron microscopy (JEOL JEM-2100F)

Research Highlight: Biofunctionalized Magnetic-Vortex Microdiscs

Research is showing that nanostructured magnetic materials offer exciting avenues for probing cell mechanics, activating mechanosensitive ion channels, and advancing potential cancer therapies. A new discovery employing ferromagnetic microdiscs with a spin-vortex ground state demonstrates magneto-mechanical cellular signal transduction at unprecedentedly low AC field frequencies. The stimulus creates two dramatic effects: compromised integrity of the cellular membrane and initiation of programmed cell death. With promising results for cancer cell destruction observed in the laboratory, studies are now being carried out at the University of Chicago Animal Facility, with preclinical trials to follow.



CMN scientist Chris Fry uses a custom-designed Schlenk-Line to create metal-oxide nanoparticles to be interfaced with peptides capable of assembling into large networks with predictable architectures for applications as novel bioelectronic materials.



The colorful pattern represents a film of 7.5-nm lead sulfide nanocrystals evaporated on top of a silicon wafer. The islands are formed by micron-size "supercrystals"—faceted three-dimensional assemblies of the same nanocrystals. The picture is a true, unaltered image, obtained with an optical microscope in reflected light mode.

RESEARCH AREAS

Nanofabrication and Devices

The Nanofabrication and Devices Group is advancing the state of the art in nanofabrication and the fundamental science of nanoscale systems. The group's multidisciplinary approach combines theory, simulation, and experiments to achieve unprecedented control in the fabrication, integration, and manipulation of nanostructures that will ultimately lead to the implementation of functional nanoscale devices.

Research Activities

- ▶ Creation of new processes capable of achieving sub-10-nm critical dimensions with large area patterning
- ▶ Development of micro- and nano-electromechanical systems as platforms for manipulation and control of nanostructures and for microenergy harvesting
- ▶ Integration of novel nanomaterials into devices
- ▶ Manipulation of nanoscale interactions
- ▶ Nonlinear phenomena at the nanoscale
- ▶ Development of novel instrumentation for the CNM user community to advance their own research programs

Key Capabilities

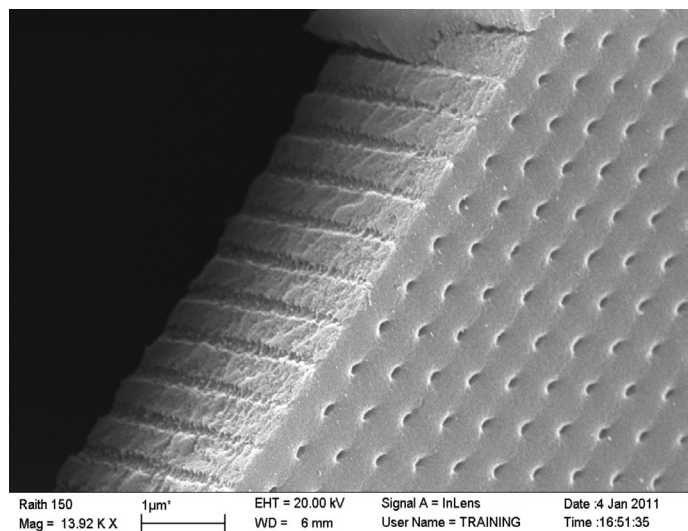
- ▶ High- and low-voltage electron beam lithography (JEOL 9300 FS and Raith 150)
- ▶ Optical lithography (Karl Suss MA6 and Microtech Laser-Writer 405)
- ▶ Nanoimprint lithography (Nanonex NX-3000)
- ▶ Focused ion beam patterning/scanning electron microscopy (FEI Nova 600 NanoLab Dual Beam)
- ▶ Microwave plasma chemical vapor deposition of nanocrystalline diamond (Lambda Technologies)
- ▶ Plasma-enhanced chemical vapor deposition of carbon nanotubes (Atomate)
- ▶ Wide variety of deposition, etching, and metrology techniques

Research Highlight: Reusable Template for the Production of Nanowires

A fast, simple, scalable technique for solution-based, electrochemical synthesis of patterned metallic and semiconducting nanowires was discovered by employing a reusable, nonsacrificial, ultrananocrystalline diamond (UNCD) template. The technique quickly produces patterned nanowires on a large scale with diameters that are not predefined by the template, and do not require vacuum or clean-room processing. This offers a path for studying nanoscale phenomena and allows for process-scale development of a new generation of nanowire-based devices.



CNM scientist Anirudha Sumant produces ultrananocrystalline diamond (UNCD) films, promising for applications in communication, biomedical devices, and tribology, using a microwave plasma chemical vapor deposition system installed in the CNM clean room.



High aspect ratio nanostructures have emerged as a new platform for many applications including X-ray optics, nanofluidics, bio-nano interfaces, and nanofilters. In a CNM project spearheaded by an industrial user group, this scanning electron microscope image shows a high aspect ratio nanofilter consisting of 250 nm pores going through a 10 μm thick SU 8 membrane. Aspect ratio: 40.

Nanophotonics

The Nanophotonics Group seeks to control optical energy and its conversion on the nanometer scale by combining the properties of metal, organic, semiconductor, and dielectric materials to create new, combined states of light and matter. To achieve these objectives, researchers use a three-pronged approach:

- ▶ Materials generation via synthesis and lithography
- ▶ Optical instrumentation development for advanced characterization
- ▶ Rigorous numerical simulations

Research Activities

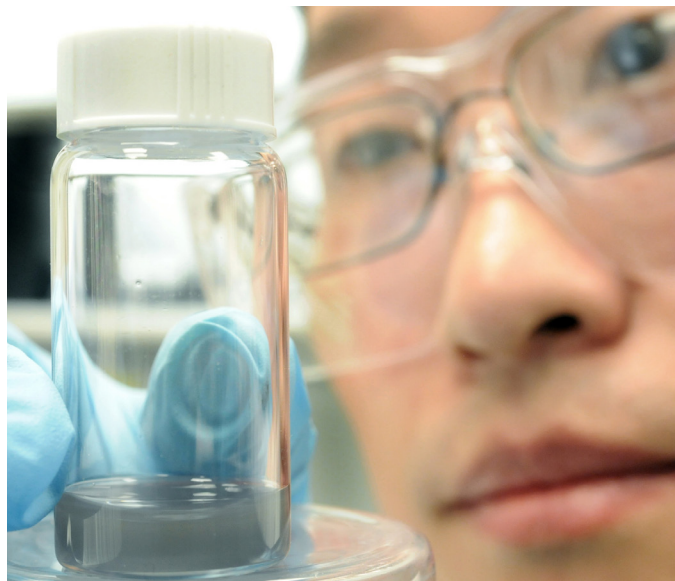
- ▶ Controlled quantum coupling at the nanoscale
- ▶ Understanding ultrafast processes at ultrasmall length scales
- ▶ New routes to functional nanophotonic materials
- ▶ Efficient energy transport in plasmonic nanostructures

Key Capabilities

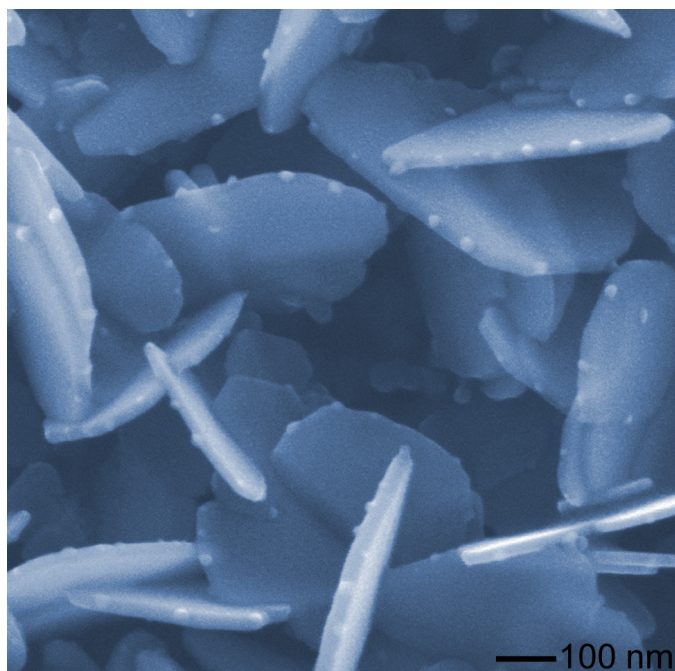
- ▶ Ultrafast transient absorption spectroscopy
- ▶ Ultrafast microscopy
- ▶ Confocal Raman microscopy
- ▶ Near-field scanning optical microscopy
- ▶ Time-correlated single-photon counting
- ▶ Two-dimensional Vis-NIR absorption-emission spectroscopy with time-resolved capabilities
- ▶ Nanophotonics nanoparticle synthesis laboratory
- ▶ Size-selected clusters and cluster-based nanomaterials

Research Highlight: New Solar Cell Technology Gives Light Waves “Amnesia”

Luminescent solar concentrators (LSCs) have traditionally been studied as a means to concentrate sunlight without tracking the sun. These devices typically operate by absorbing light and then re-emitting it at lower frequency into a transparent slab. However, optical propagation loss due mostly to reabsorption has limited the concentration ratios. Using CNM capabilities, researchers have designed and tested a new form of LSC that produces a more than two-fold increase in concentration ratio. In this work, reabsorption is avoided by nanostructuring the cavity to produce ‘resonance-shifts,’ so that directed emission returns to interact with the cavity off-resonance at each subsequent bounce. Near-lossless emission propagation has been demonstrated.



CNM scientist Yugang Sun prepares a sample of silver chloride nanowires decorated with gold nanoparticles. These nanostructures could find applications in photocatalysis and plasmon-enhanced photoprocesses for solar energy conversion.



These plasmonic silver nanoparticles are decorated with silver salt nanoparticles along the edges. The nanostructures were grown under irradiation of high-energy X-rays, which allowed scientists to “watch” them grow in real time. This image is from a scanning electron microscope.

RESEARCH AREAS

Theory and Modeling

The close interplay between theory and experiment is an exciting aspect of nanoscience and oxide formation. In the Theory and Modeling Group, researchers focus on a number of specific, experimentally relevant areas including, but not limited to, nanocatalysis and nanophotonics. CNM scientists also strive to develop the theory, modeling, and computational capabilities with the ultimate goal of designing novel nanoscale materials with user-defined properties.

Research Activities

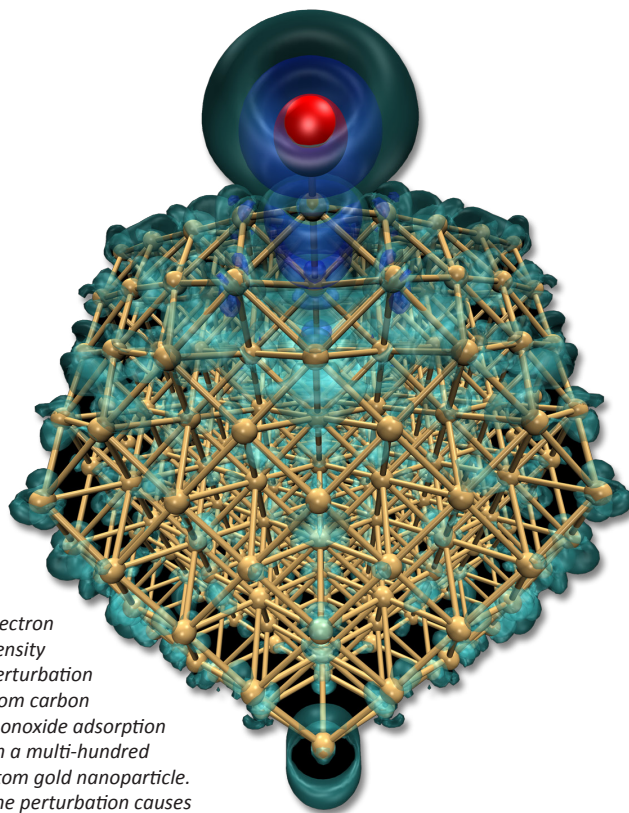
- ▶ Nanocatalysis — electronic structure calculations of nanocluster-catalysis based mechanisms
- ▶ Atomistic studies of nanoscale oxide formation
- ▶ Computational nanophotonics with rigorous electrostatics calculations
- ▶ Quantum dynamics of molecular motion in nanoconfined environments
- ▶ Methods and software development, including work on parallelization of density functional theory codes and new methods that address the multiscale features of many nanoscience problems

Key Capabilities

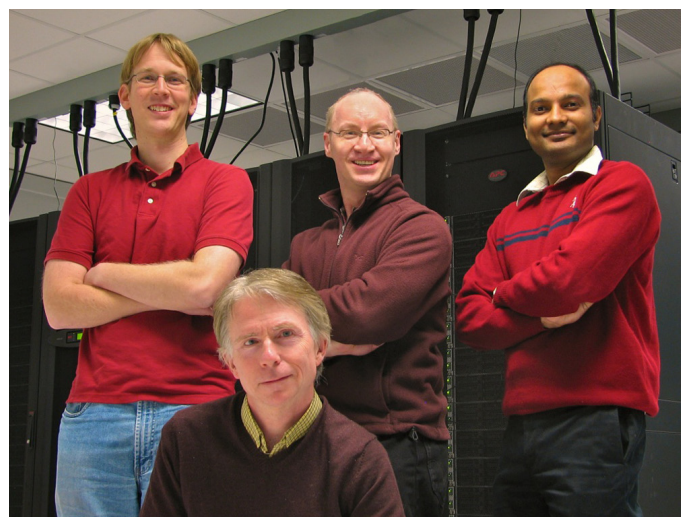
- ▶ High-performance computing cluster (2,800 cores, 30 TFlops)
- ▶ Development tools (GNU and Intel compilers and math libraries)
- ▶ Serial and parallel versions of the density functional theory codes: Dacapo, GPAW, VASP, Q-Chem
- ▶ Serial and parallel two- and three-dimensional finite difference time-domain codes (home-grown and MIT's MEEP)
- ▶ Wave packet and Lanczos (bound state) quantum codes

Research Highlight: Using Light to Build Nanoparticles into Superstructures

A simple and cost-effective way of assembling nanoparticles into larger mesoscale structures of any desired shape and form was recently demonstrated experimentally at Argonne. The process, called optically directed assembly (ODA), involves optical trapping, heating, convective fluid flow, and chemical interactions. The CNM Theory and Modeling Group developed a multiscale model featuring molecular dynamics, fluid dynamics, and kinetic Monte Carlo calculations in order to understand and predict how ODA works.



Electron density perturbation from carbon monoxide adsorption on a multi-hundred atom gold nanoparticle. The perturbation causes significant quantum size effects in CO catalysis on gold particles.



The Theory and Modeling Group members are (left to right, standing) Jeffrey Greeley, Michael Sternberg, Subramanian Sankaranarayanan, and (in front) Stephen Gray.

X-Ray Microscopy

The CNM's Hard X-ray Nanoprobe (HXN), located at Argonne's Advanced Photon Source (APS), is a next-generation hard X-ray microscopy and X-ray imaging beamline with the highest spatial resolution in the hard X-ray range. This unique instrument provides unprecedented capabilities to characterize extremely small structures. These capabilities are key to the broader nanoscience community in studying nanomaterials and nanostructures, particularly for embedded structures.

Research Activities

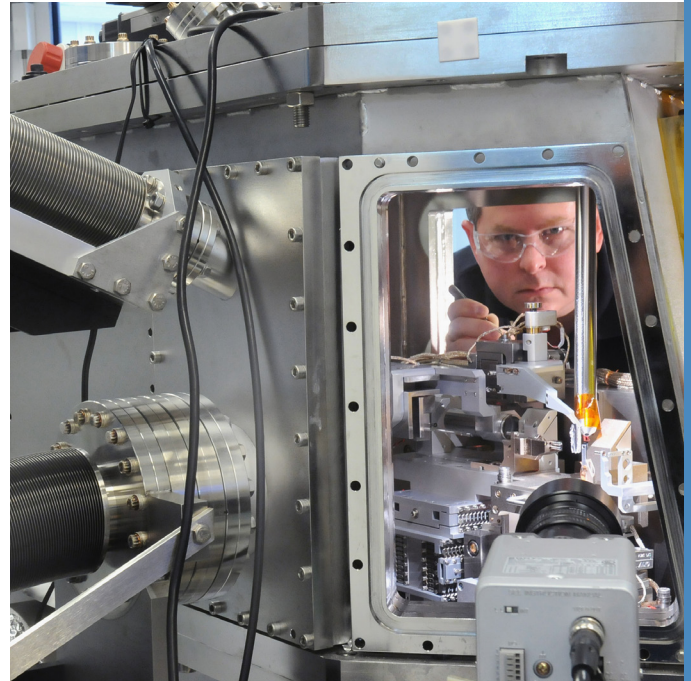
- ▶ Time-resolved stroboscopic measurements
- ▶ General nanomaterials characterization with X-rays, including Bragg coherent diffraction
- ▶ User science has included understanding of strain in systems such as silicon-based devices and resistive RAM systems, distribution of matrix elements in geopolymers and novel nanocomposites, and nanocomposites in tissues and cells

Key Capabilities

- ▶ Scanning probe X-ray diffraction microscopy
- ▶ Scanning probe X-ray fluorescence microscopy
- ▶ Full-field two-dimensional transmission imaging and tomography

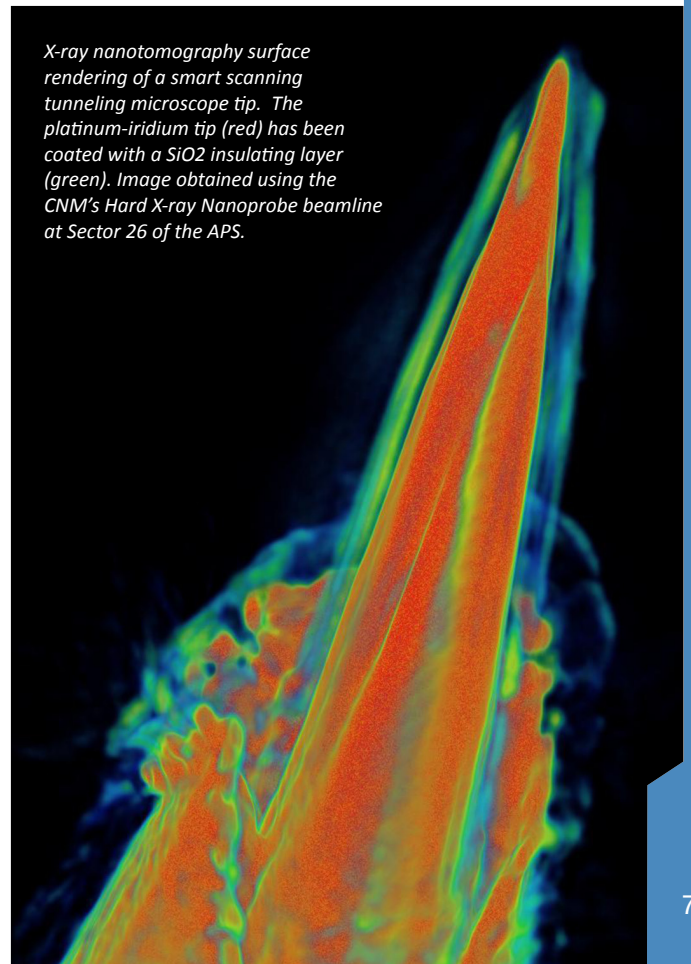
Research Highlight: Structural Consequences of Nanolithography

Research at the CNM's Hard X-ray Nanoprobe beamline has shed new light on the physics of structural changes that occur during nanoscale lithography of ferroelectric polarization domains. X-ray nanodiffraction microscopy was performed to probe a pattern written into a ferroelectric layer using scanning-probe ferroelectric nanolithography. The resulting stable strain pattern showed that the overall shape of the film was unchanged, but the electrical polarization was modified. Ferroelectric lithography is a promising approach to control nanoscale degrees of freedom with scanning probes. In other systems, it can also provide control of magnetic and charge-ordered domains. Based on this new insight, it will be possible to extend piezoresponse force microscopy and other nanoscale patterning methods using direct local structural information.



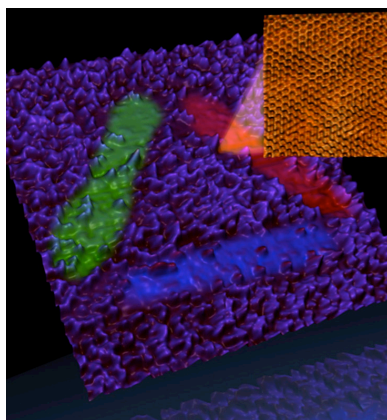
CNM scientist Robert Winarski uses the Hard X-ray Nanoprobe to discern the physical structure and spatial distribution of individual elements inside advanced battery components. Research into new battery technologies could lead to more durable and safer batteries with much larger capacities.

X-ray nanotomography surface rendering of a smart scanning tunneling microscope tip. The platinum-iridium tip (red) has been coated with a SiO₂ insulating layer (green). Image obtained using the CNM's Hard X-ray Nanoprobe beamline at Sector 26 of the APS.



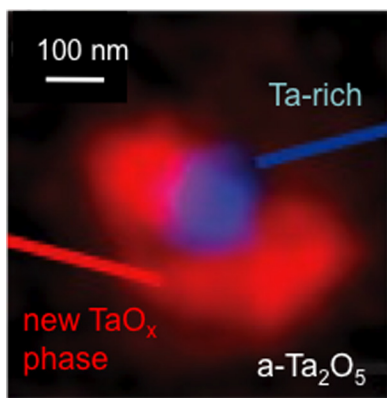
SUCCESS STORIES

Since opening its doors in 2006, the CNM has been responsible for a large number of pioneering research projects. Check out a few more of the center's recent successes below.



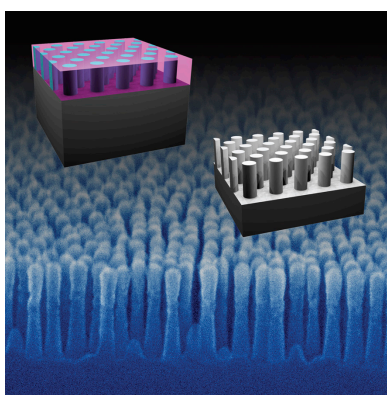
Graphene Research at the CNM

Graphene, the subject of the 2010 Nobel Prize in Physics, is transparent, thermally conductive, flexible, chemically inert, biologically compatible, and 200 times stronger than steel. A sheet is only one atom thick making it an ideal two-dimensional material. CNM graphene research explores state-of-the-art synthesis, characterization, processing, and novel applications. The highest resolution microscopes available characterize its structural, electronic, and chemical properties at the atomic scale. These tools have identified material defects and guided the optimization of large-scale synthesis. CNM also modifies graphene through chemical modification, engineering, and nanolithography for applications in advanced solar cell design, enhanced electronic material performance, and enhanced energy efficiency processes.



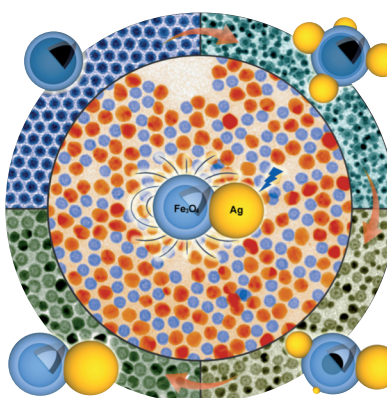
Observing the Nanoscale Origins of Memory-Resistive Switching

Using X-ray fluorescence nanospectroscopy at the Hard X-ray Nanoprobe beamline, an industrial user group recently performed one of the first nondestructive studies of subsurface chemical state change. This was accomplished with spatial resolution better than 70 nm, without sectioning or otherwise modifying the sample. By identifying the nanoscale regions of material change in switched TaO memristor devices, scientists have shed new light on the microscopic origins of memory-resistive switching in oxide thin film devices.



Enhancing Electron Beam Lithography Using Sequential Infiltration Synthesis

A new method dramatically increases the etch resistance achieved with standard electron-beam lithography. This technique, called sequential infiltration synthesis (SIS), uses alternating and self-limiting reactions between gas phase precursors and polymeric resists to grow inorganic material within a film. These inorganic materials, such as aluminum oxide, are highly etch-resistant and enable the transfer of very deep patterns without the need for a traditional hard mask, thereby improving image quality and reducing process costs. Transferring patterns more deeply into materials would allow scientists to craft better electronics.



Bifunctional Plasmonic/Magnetic Nanoparticles

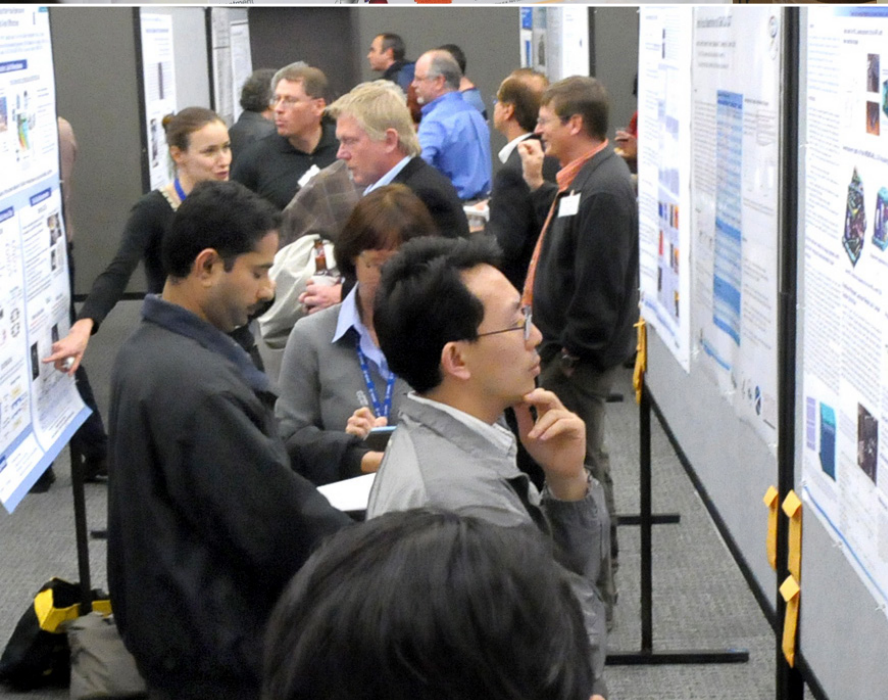
An amorphous-seed mediated strategy has been developed for creating bifunctional nanoparticles composed of silver and iron oxide nanodomains. These hybrid particles exhibit unique optical properties due to surface plasmon resonance from the silver and superparamagnetic responses from the iron oxide. This is significant because multicomponent hybrid nanoparticles can exhibit multiple functionalities for applications that are difficult (or even impossible) to achieve from single-component nanoparticles. With this new approach, the multifunctional hybrid nanoparticles are expected to be useful in surface-enhanced Raman scattering (SERS) for chemical and biological sensing, magnetic/optical dual-modal imaging, and drug delivery.

OUTREACH PROGRAMS AND ACTIVITIES

The CNM's outreach programs aim to raise awareness about the unique features that keep our center on the cutting edge of nanoscience and nanotechnology research.

Activities include:

- ▶ Annual Users Meeting with plenary sessions, workshops, poster sessions, and a vendor expo
- ▶ Technical workshops on various scientific topics in nanoscience and nanotechnology
- ▶ Short courses that offer tutorials and hands-on training on various instruments and capabilities
- ▶ Facility tours
- ▶ Triannual newsletters
- ▶ Educational outreach and public awareness events



On the cover:

Plasmonic silver nanoparticles are decorated with silver oxy salt nanoparticles along the edges. The nanostructures were grown under irradiation of high-energy X-rays, which allowed scientists to “watch” them grow in real time. This image is from a scanning electron microscope.

Optical profile of an actuated Microelectromechanical System (MEMS) device that was designed, fabricated, and tested in the CNM clean room. This MEMS device is used to generate X-ray pulses of variable width.

Surface topography of alanine molecules on Cu(111) surface. Once on the Cu(111) surface, alanine molecules form a supramolecular network of hexagonal pores. Image acquired using a variable temperature scanning tunneling microscope.

X-ray nanotomography surface rendering of a smart scanning tunneling microscope tip. The platinum-iridium tip (red) has been coated with a SiO₂ insulating layer (green). Image obtained using the CNM’s Hard X-ray Nanoprobe beamline at Sector 26 of the APS.



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