

Nanoscale Science Research Centers

National User Facility Organization

April 24, 2008

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Center for Nanophase Materials Sciences
Oak Ridge National Laboratory

Nanoscale Science Research Centers – New DOE User Facilities

- **Began with proposals in FY01; First on-line in FY06**
- **Operated as user facilities**
 - Available to all researchers; access determined by external peer review of proposals
 - No cost for research published in the open literature; Proprietary access - full cost recovery
 - Provide specialized equipment and support staff
- **Co-located with existing user facilities (synchrotrons, neutron scattering facilities, etc.)**
 - Provide extraordinary characterization and analysis capabilities
- **Conceived with input from academia and industry**
- **Multidisciplinary – Materials, Chemistry, Computational, Biology, Engineering,....**
- **Have “Nano” environment, safety, and health concerns as a priority**



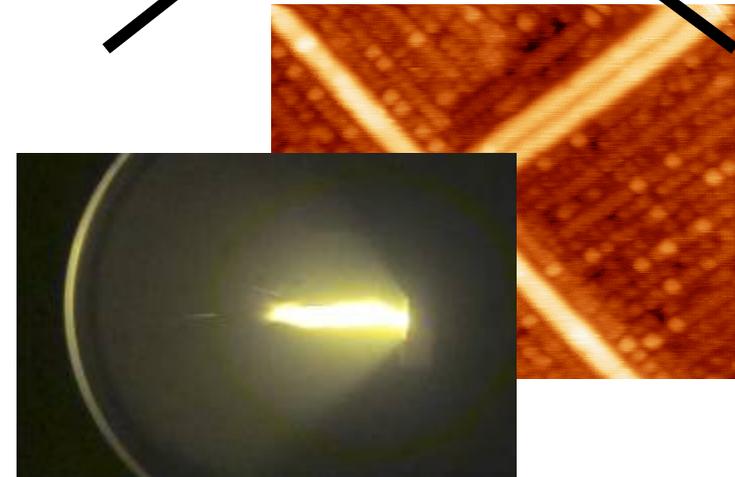
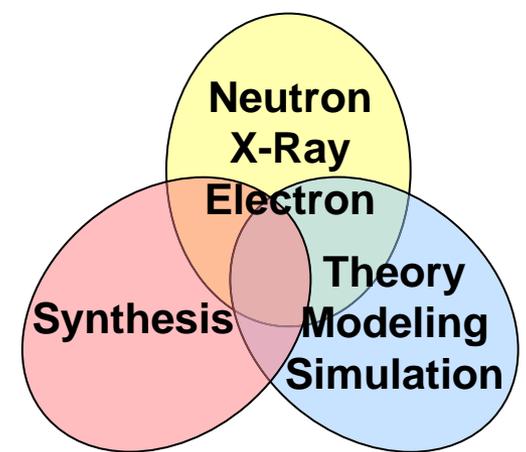
Vision for NSRCs: World-Class User Facility with an In-house World-Leading Science Program

- **Organization, staffing, and capabilities must support both the user and science programs**
- **User metrics**
 - Publications, citations
 - Survey input
 - Numbers of users, user hours, etc.
- **In-house science measures**
 - Publications, citations, etc.
 - Awards, patents, other recognition, etc.
 - Unique capabilities and expertise
 - Foundation to attract future users

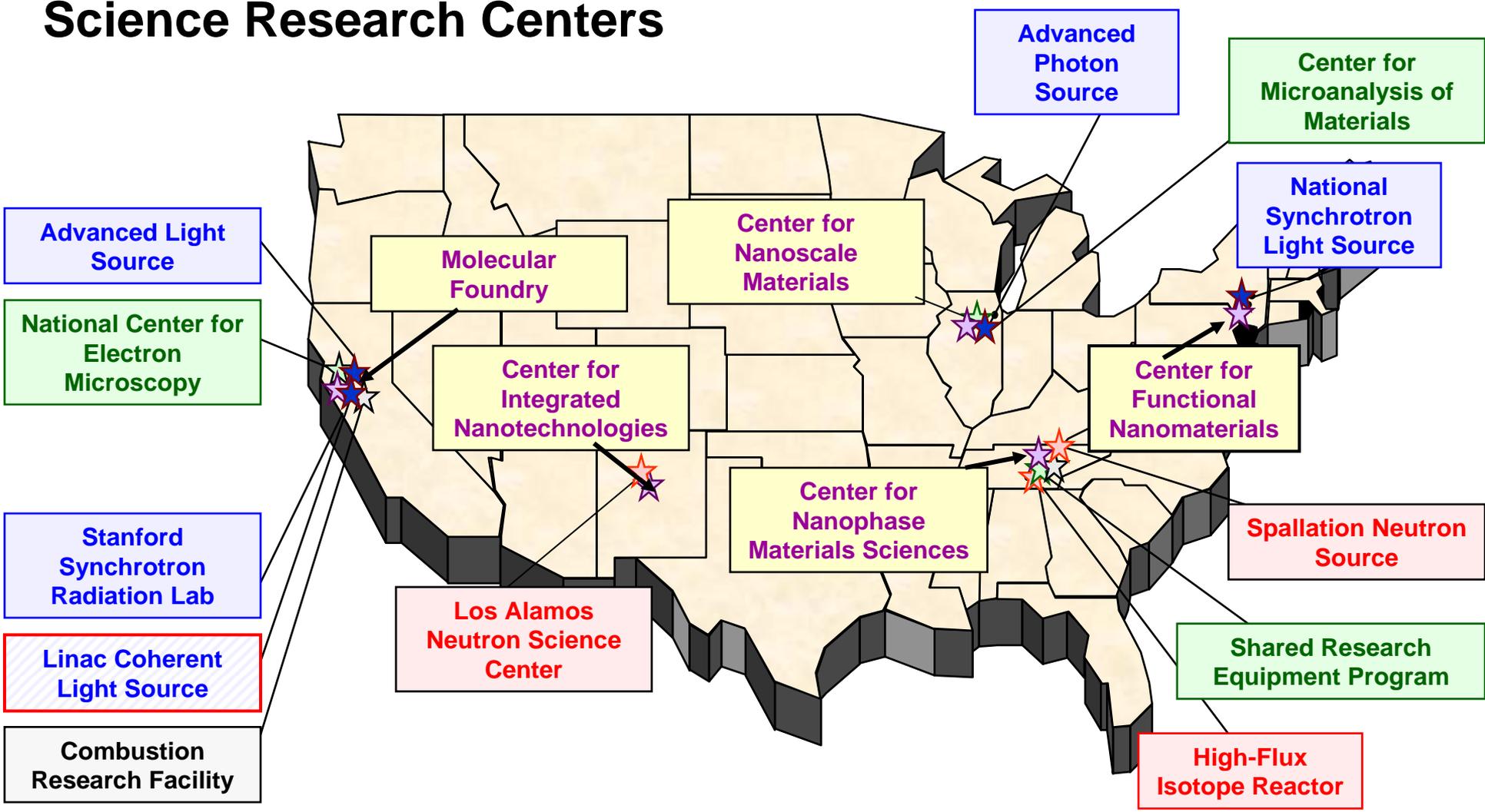


Nanoscience Centers Integrate Nanoscale Science with Synergistic Research Capabilities

- Build on Unique National Facilities for X-Ray, Neutron, and Electron Microscopy Characterization
 - Opportunity for world leadership
- Synthesis Science
 - **Science-driven** synthesis using unique instrumentation and integrated with characterization
- Theory / Modeling / Simulation
 - Stimulate U.S. leadership in using theory, modeling and simulation to design new nanomaterials
 - Investigate new pathways for materials synthesis



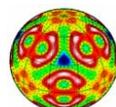
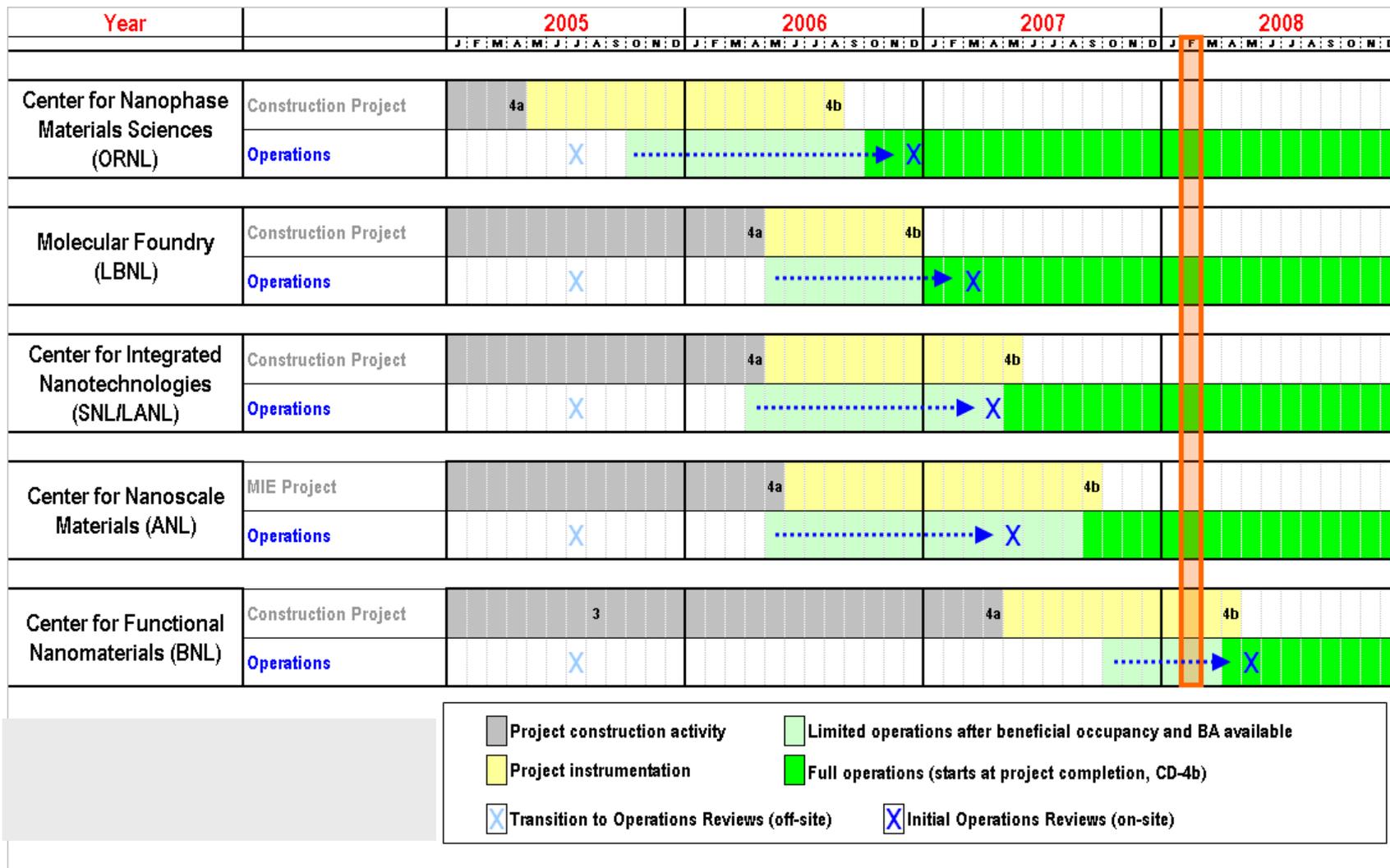
DOE-BES' 5 Nanoscale Science Research Centers



Note: All Labs are working toward single proposals for multiple facility access

- 4 Synchrotron Radiation Light Sources
 - Linac Coherent Light Source (CD3 approved)
 - 4 High-Flux Neutron Sources
 - 4 Electron Beam Microcharacterization Centers
 - Special Purpose Centers
 - 5 Nanoscale Science Research Centers
- Under construction**

Schedules for NSRCs



Nanoscale Science Research Centers are Completed and open for Users



Center for Functional Nanomaterials (Brookhaven National Laboratory)



Molecular Foundry (Lawrence Berkeley National Laboratory)



Center for Nanoscale Materials (Argonne National Laboratory)



Center for Nanophase Materials Sciences (Oak Ridge National Laboratory)

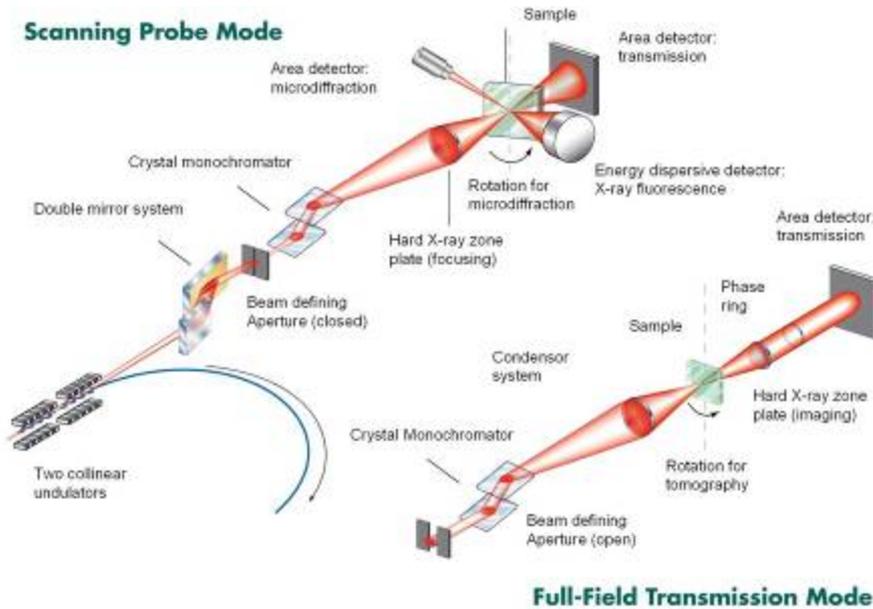


Center for Integrated Nanotechnologies (Sandia & Los Alamos National Labs)



Nanoscience Centers provide new kinds of capabilities

Scanning Probe Mode

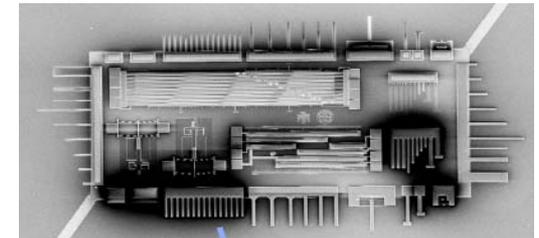


X-Ray Synchrotron Beamlines with Nanoscale Resolution

- Unique instruments to study individual nanostructures
- Quantitative structure, strain, orientation imaging
- Sensitive trace element and chemical state analysis

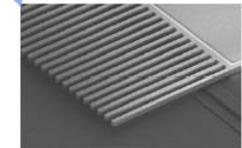
“Discovery Platforms”: modular micro-laboratories for nanoscience

- Standardized and batch fabricated
- Access to a range of diagnostic and characterization tools

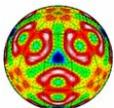


Cantilever Array

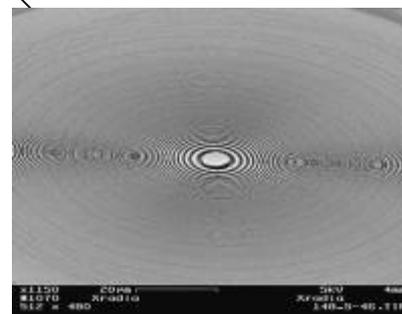
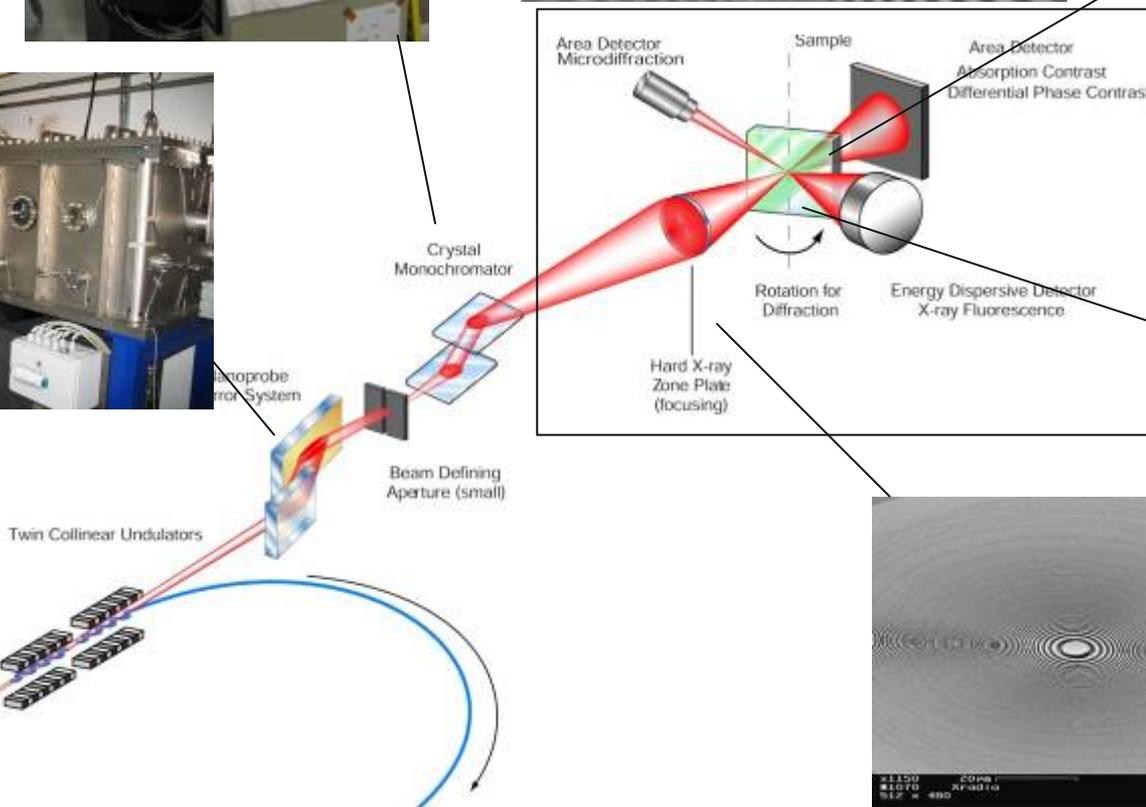
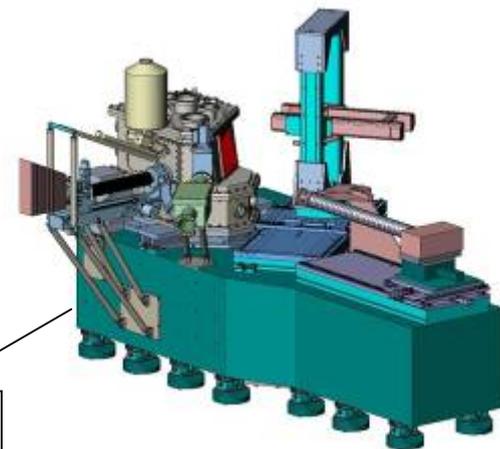
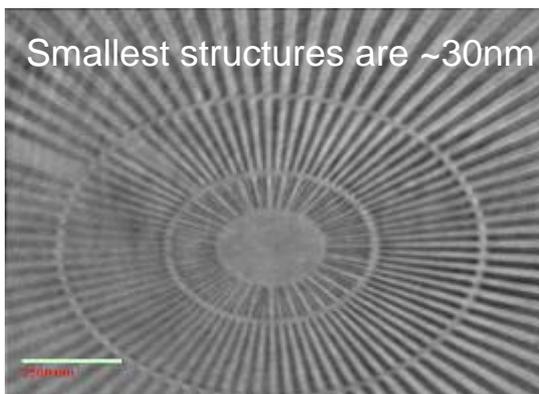
Chip



Microfluidic Synthesis



The Worlds Premier Hard X-ray Nanoprobe: 30 nm and beyond



Xradia zone plate and instrument



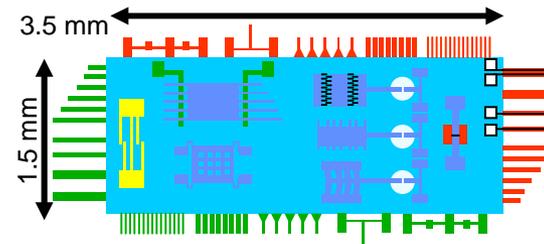
Discovery Platforms™ are a unique tool for nanoscience users

Standardized modular, micro-laboratories—designed and batch fabricated for:

- Integrating nano and micro length scales
- Studying properties of nanoscale materials and devices
- Directly accessing wide range of CINT characterization tools

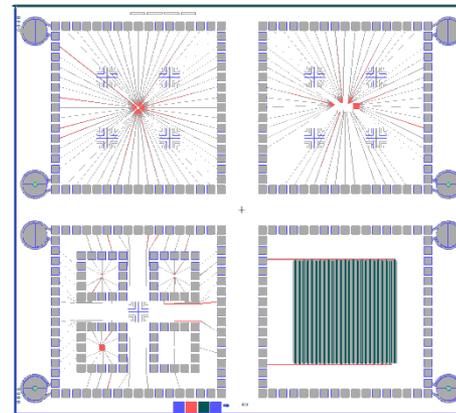
Cantilever Array Platform

- Mechanics at nanoscale



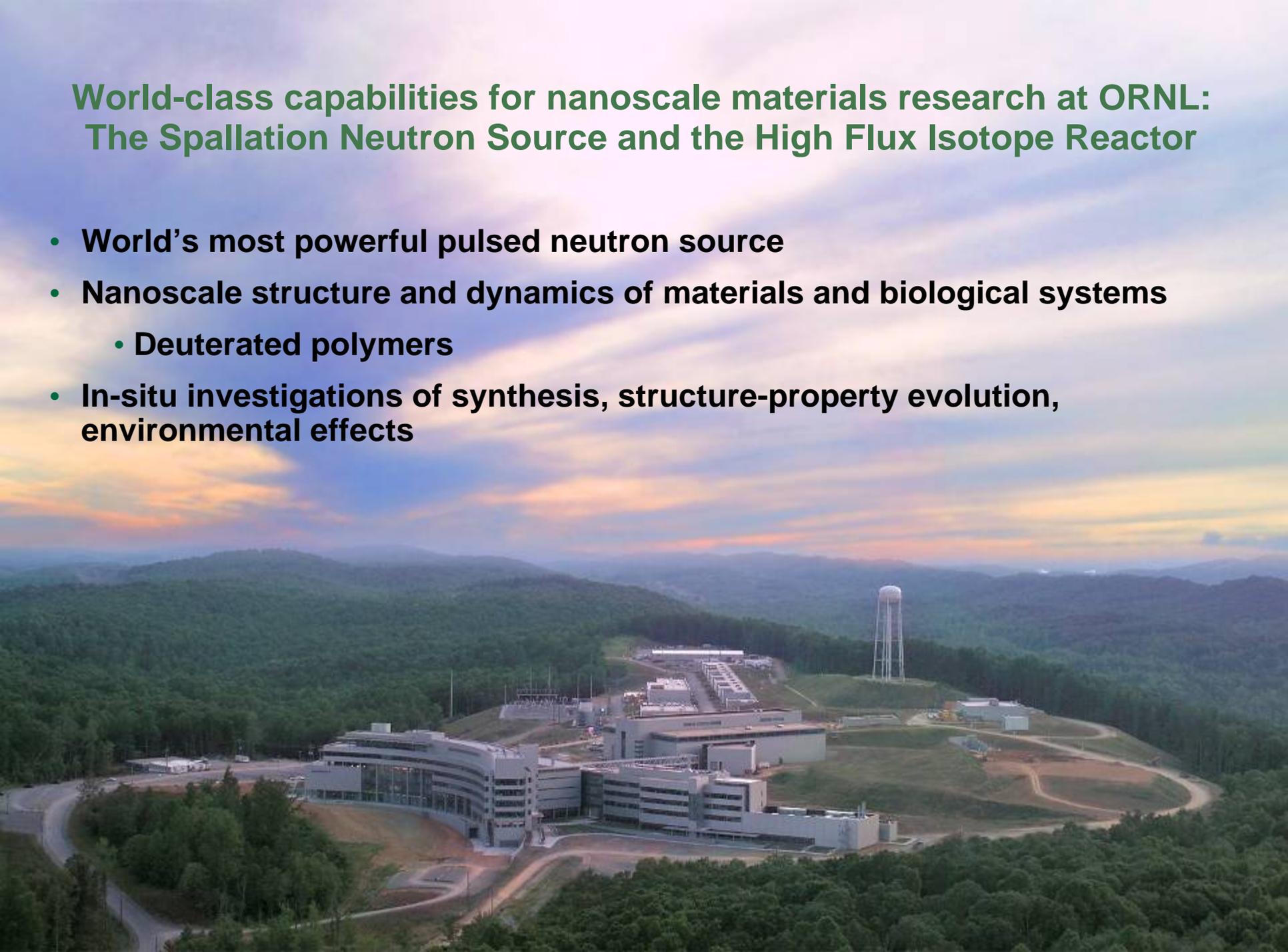
Electrical Transport & Optical Spectroscopy Platform

- Silicon based qubits
- Nanowires



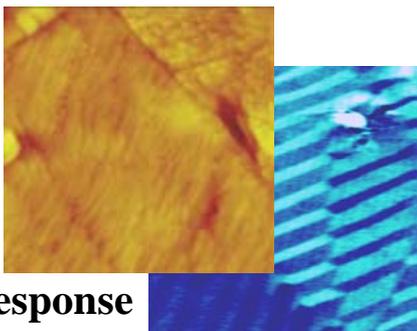
World-class capabilities for nanoscale materials research at ORNL: The Spallation Neutron Source and the High Flux Isotope Reactor

- **World's most powerful pulsed neutron source**
- **Nanoscale structure and dynamics of materials and biological systems**
 - **Deuterated polymers**
- **In-situ investigations of synthesis, structure-property evolution, environmental effects**

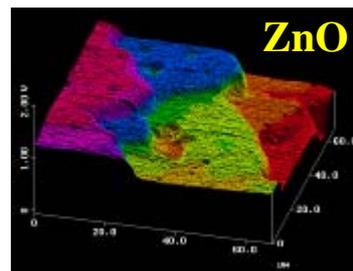


Advanced Scanning Probe Microscopy

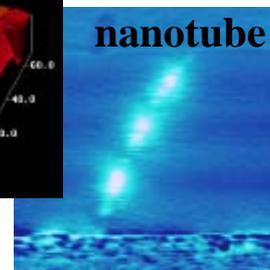
- Imaging of physical properties
- Conductivity
- Ferromagnetism
- Ferroelectricity
- Catalysis



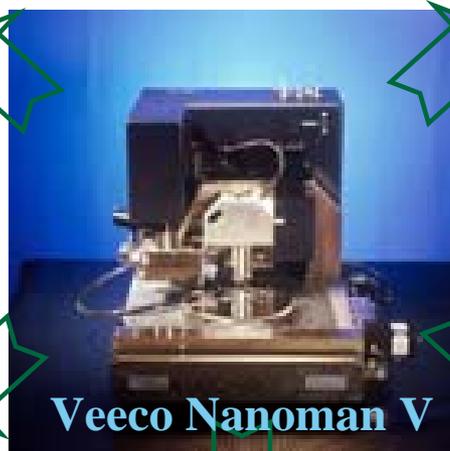
Piezoresponse
Force Microscopy



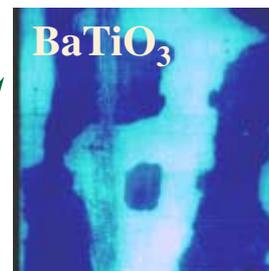
Transport



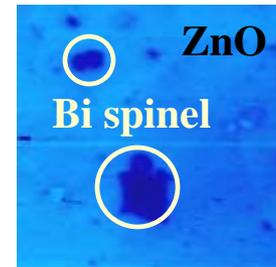
nanotube



Veeco Nanoman V



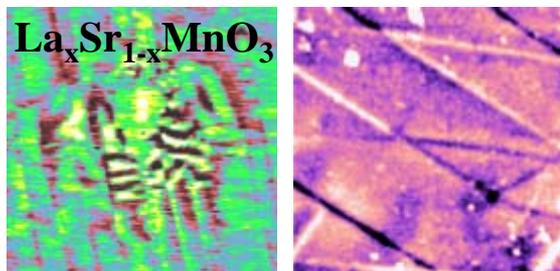
BaTiO₃



ZnO

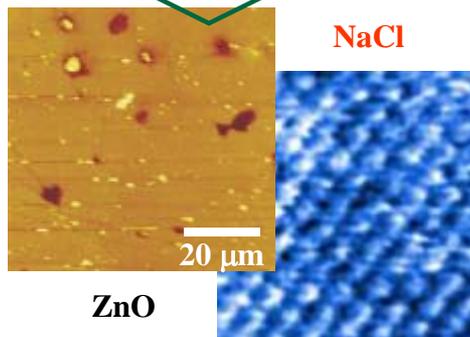
Bi spinel

Potential imaging



La_xSr_{1-x}MnO₃

Magnetic Force Microscopy



NaCl

20 μm

ZnO

AFM Topography

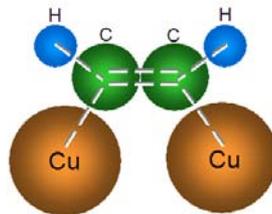
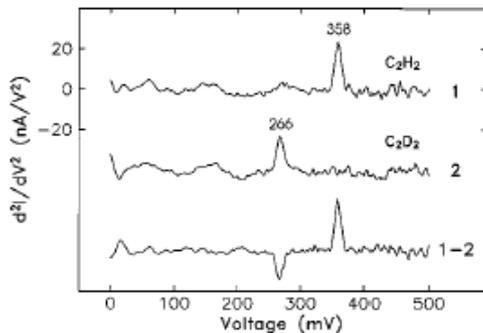
Low Temperature-High Field STM

Scientific Drivers

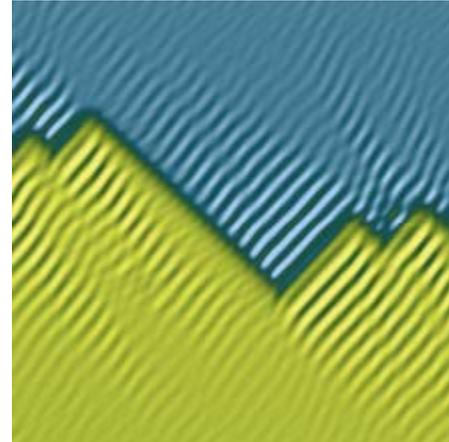
- Atomically-resolved topography and spectroscopy maps
- Quantum response at low T and High B
- Real Space – K-space
- Single Molecule Spectroscopy

Capabilities

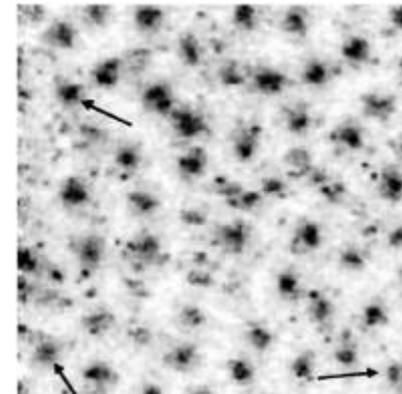
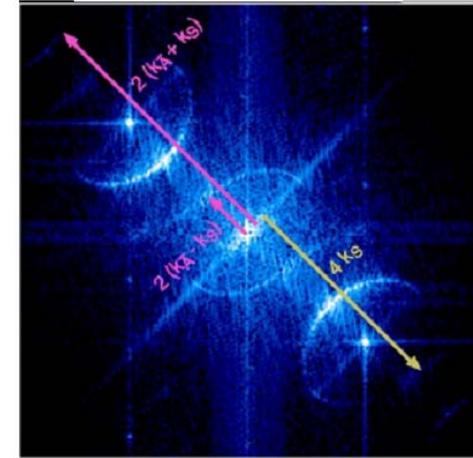
- Low T - 300 mK
- High B - 9 Tesla
- STM rotates in magnetic field
- Optical access to sample
- High Resolution
- Cryogenic UHV cleaving
- Sample Fabrication in UHV



Real Space

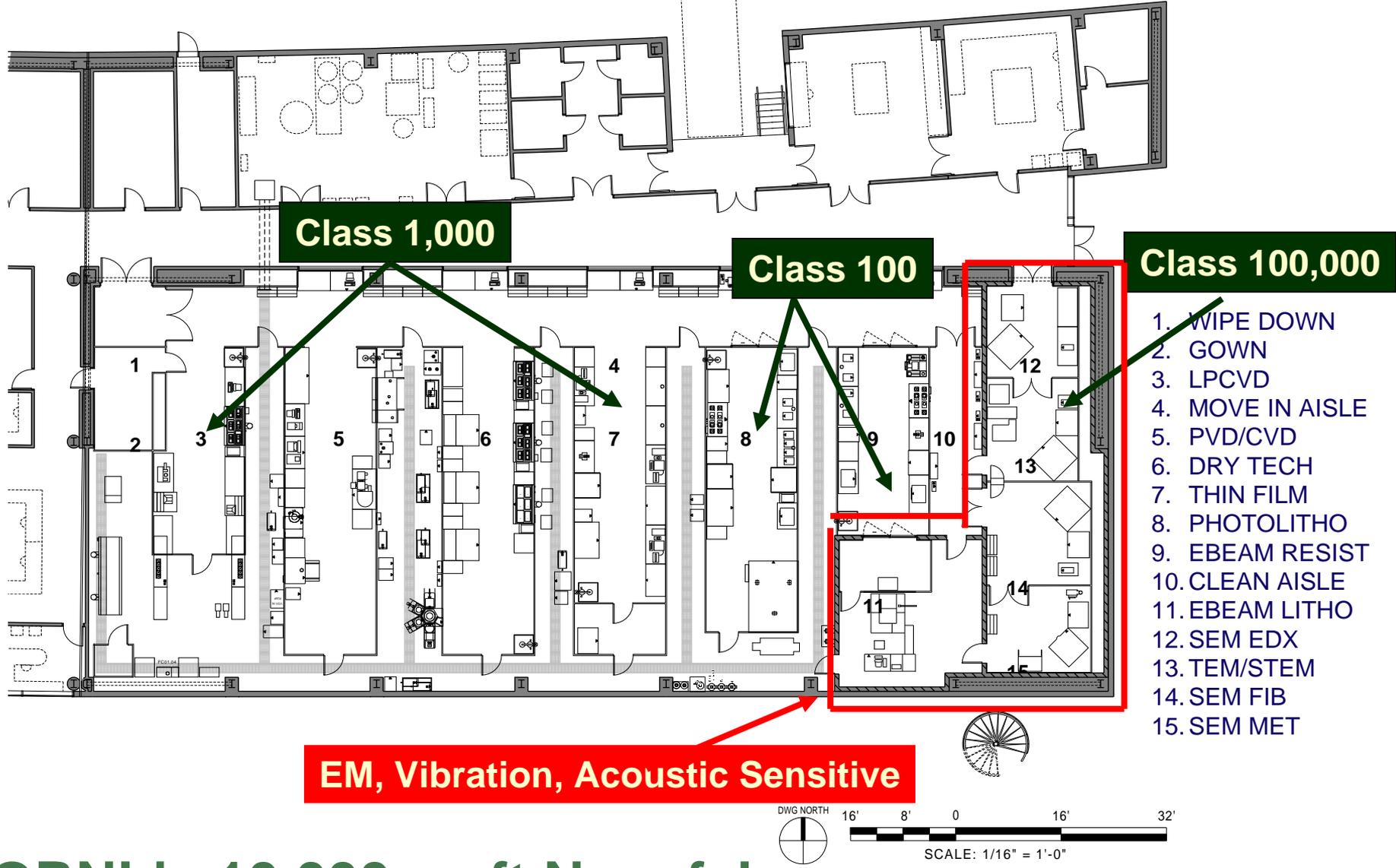


K-space



DOS map showing vortices at the surface of BSCCO in 7.2T

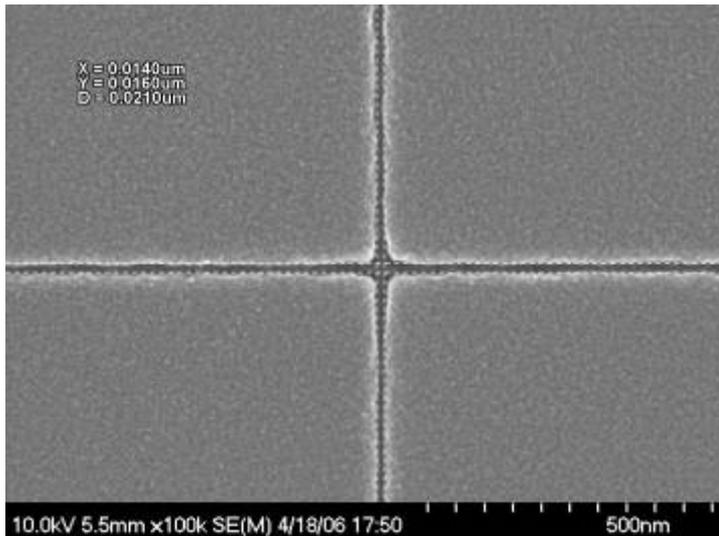
And – Some Common Capabilities: Clean Room Nanofabrication Facilities



ORNL's 10,000 sq ft Nanofab

JEOL JBX-9300 E-Beam Lithography System

- 50 & 100 kV accelerating voltage
- 4 nm minimum spot size
- substrate sizes from 50 - 300 mm plus pieces



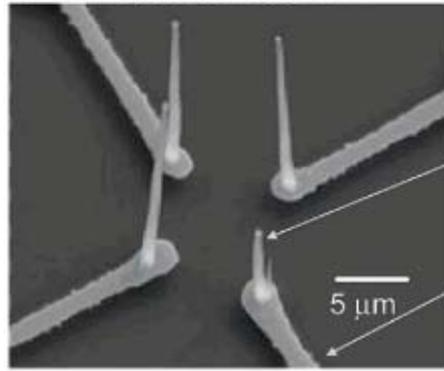
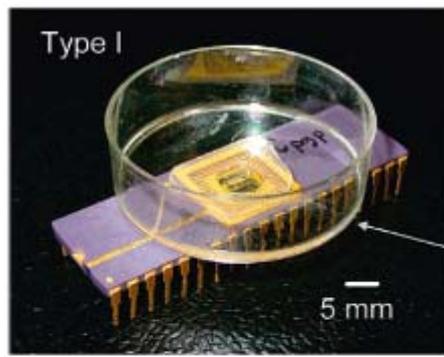
Minimum linewidth resolution
testing in ZEP-520 resist
written at 50 kV



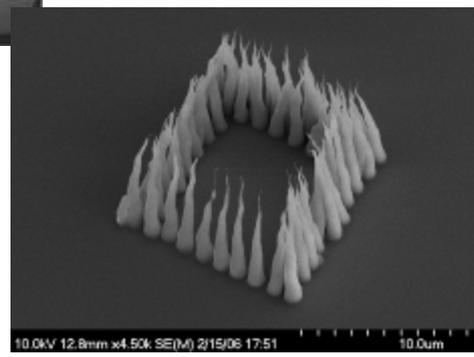
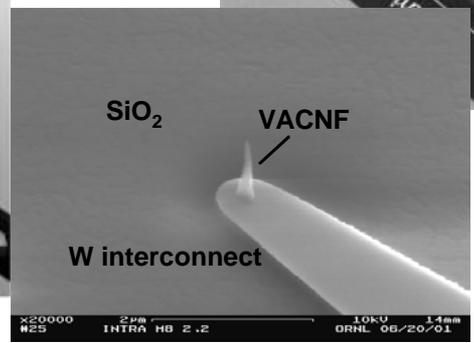
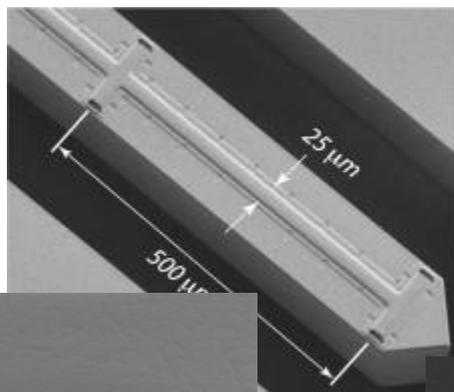
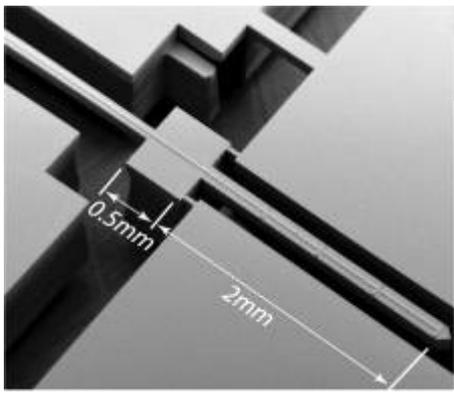
Nanofabrication Research

Thin Film Deposition and Characterization

- Chemical and Physical Vapor Deposition
- High Temperature Growth and Annealing
- SEM, SPM and optical visualization
- Multiscale Lithography
 - Optical and electron beam lithography
- Material Removal
 - Dry etching and ablation
 - Wet Chemical Etching

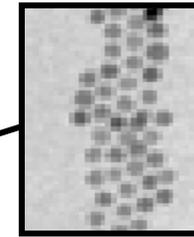
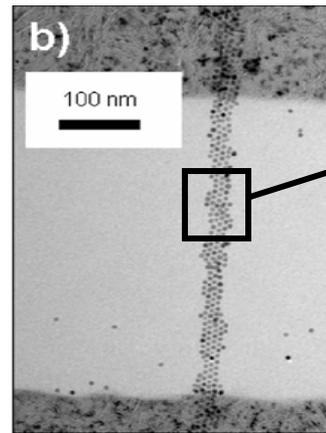
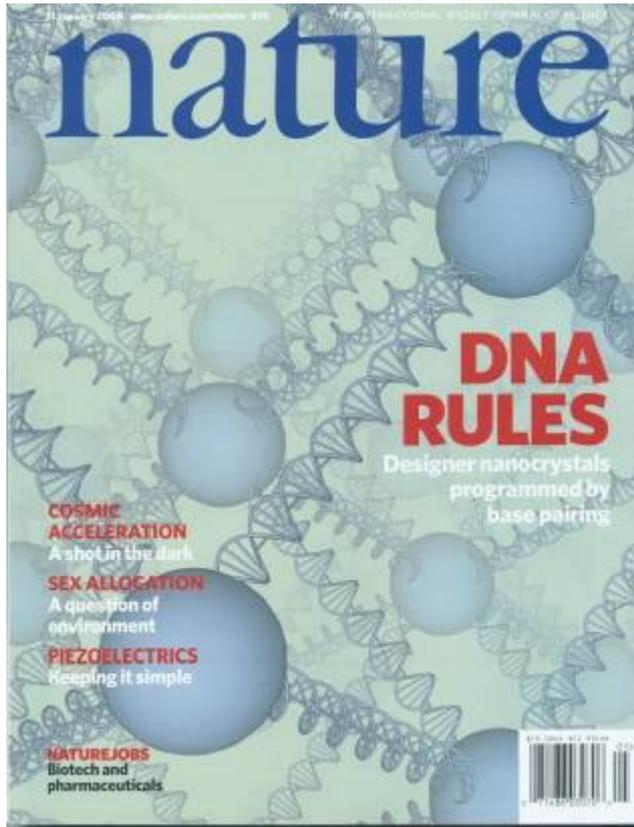


McKnight et al.
CNMS2003-042

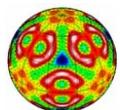
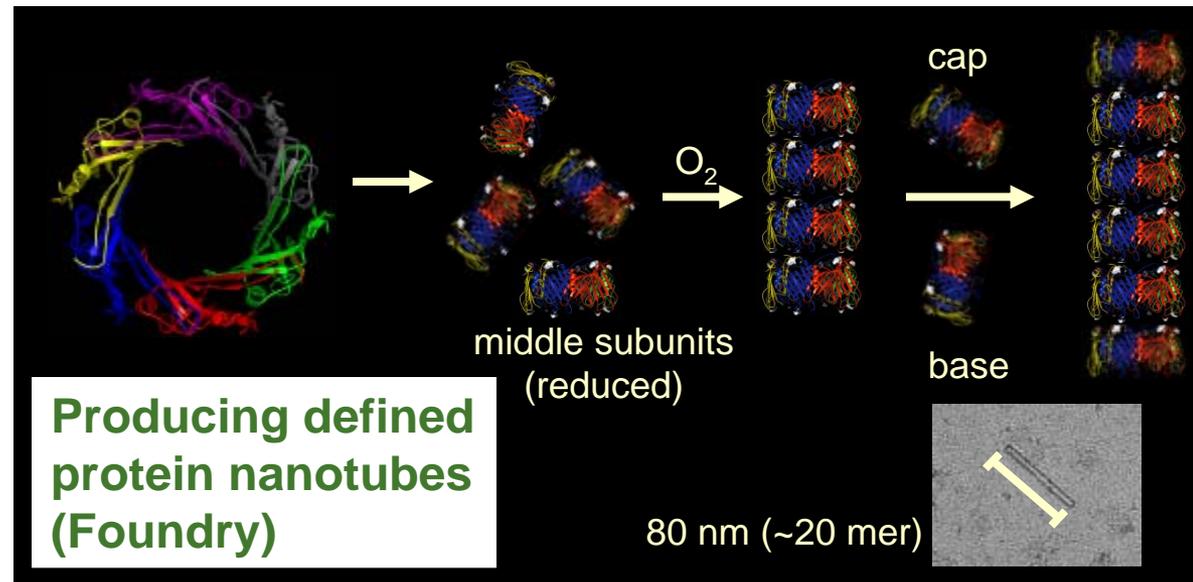


Controlled Synthesis

Using DNA to guide controlled 3-D crystallization of nanoparticles (CFN)

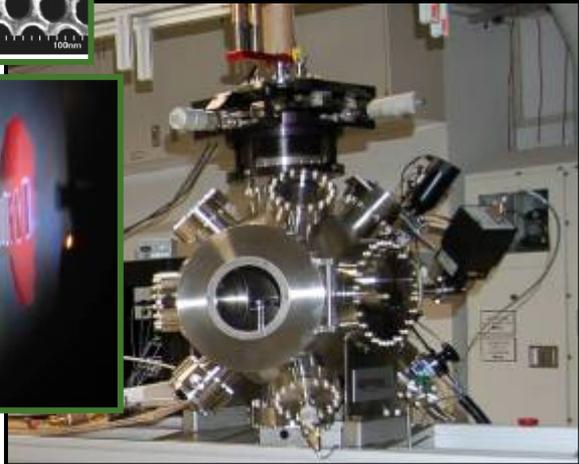
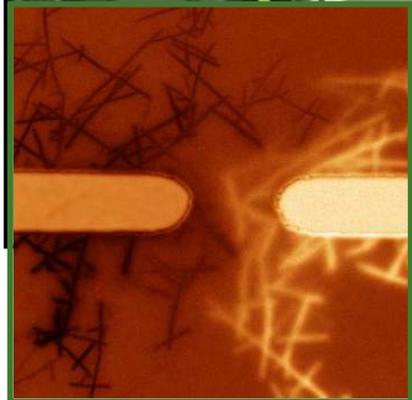
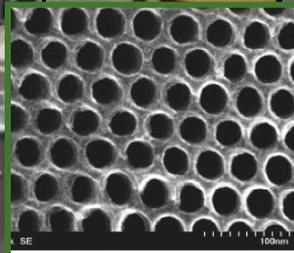


Assembly of and charge transport in quasi-1D nanocrystal arrays (CNM)

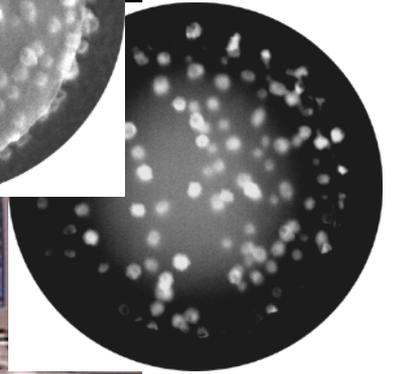
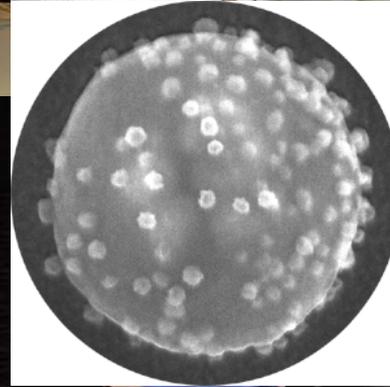
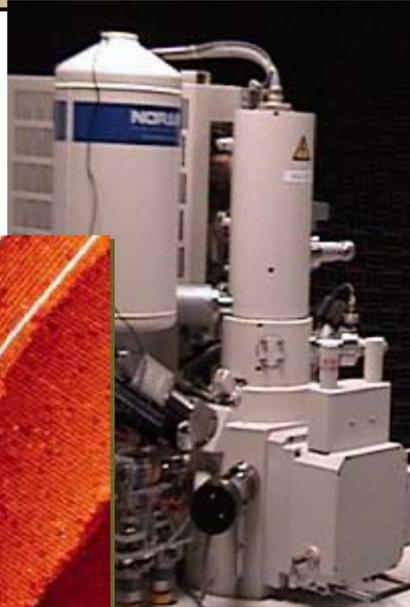
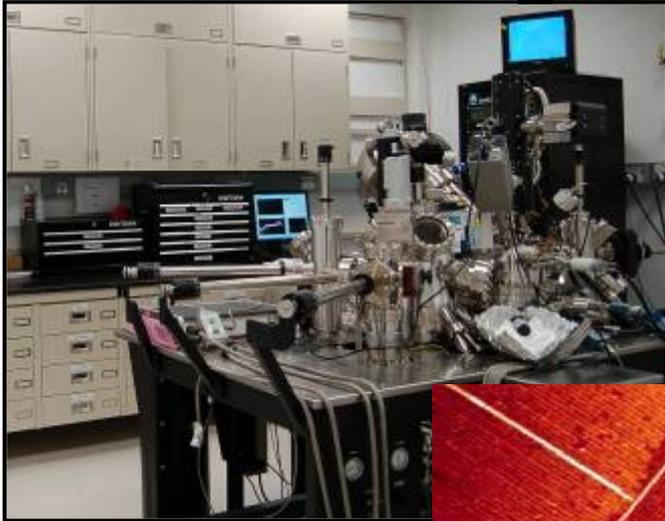
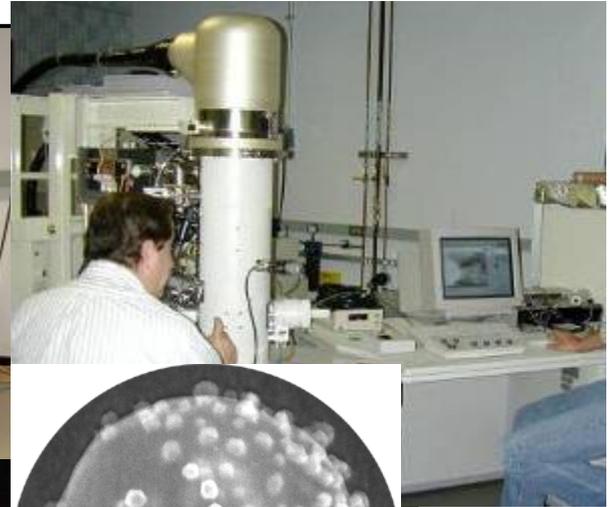


Adapted from A. H. Carim
Basic Energy Sciences

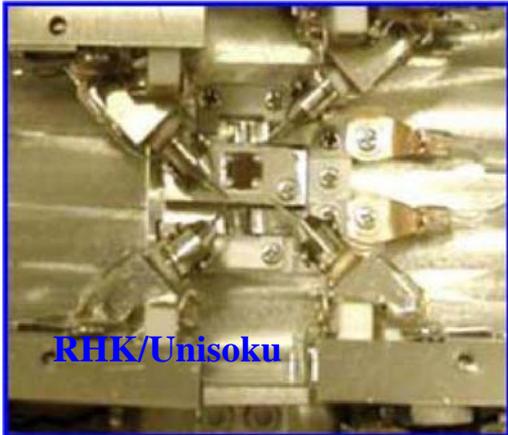
Synthesis Capabilities



Key Characterization Equipment

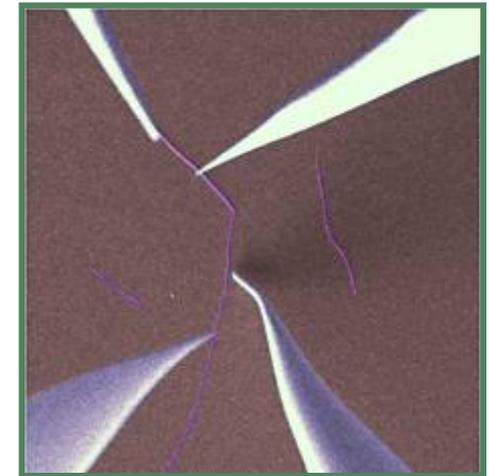


4 probe Scanning Tunneling Microscope with SEM/SAM



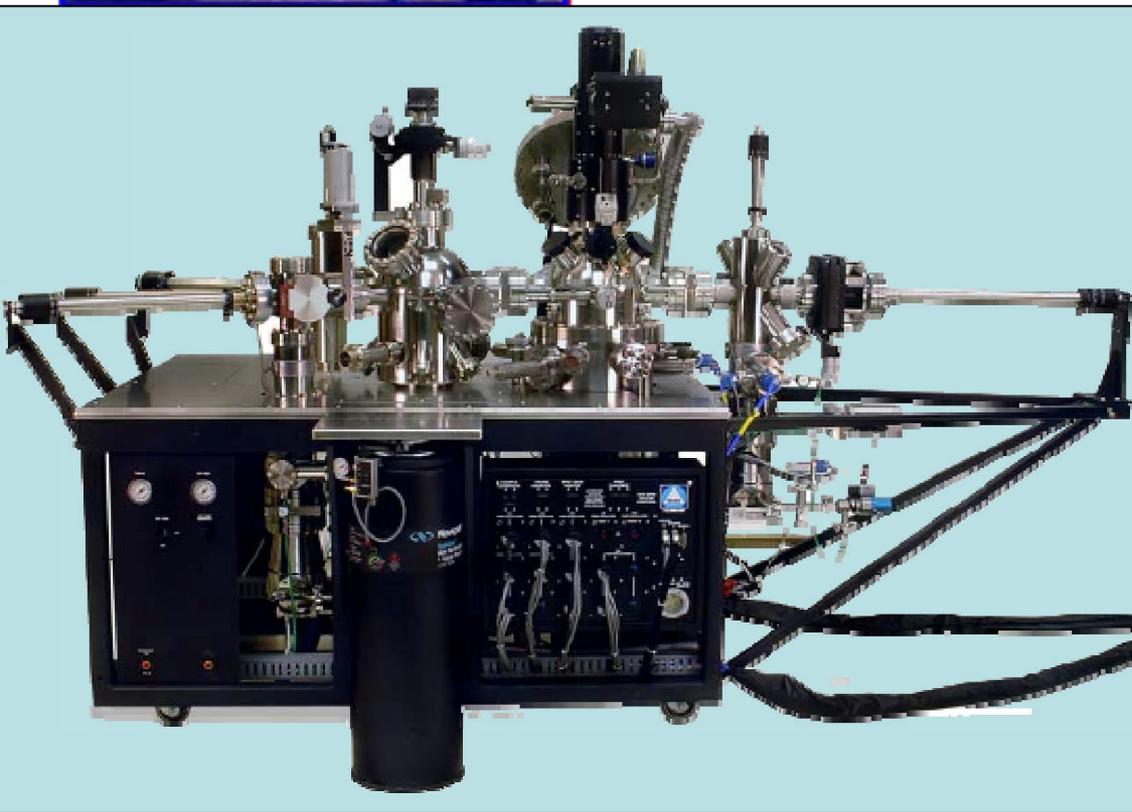
Scientific Drivers

- ❖ Nanoscale electrical transport as a function of temperature
- ❖ Nanofabrication and manipulation
- ❖ Elemental identification



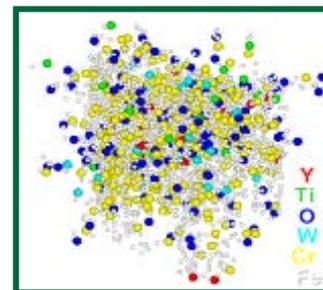
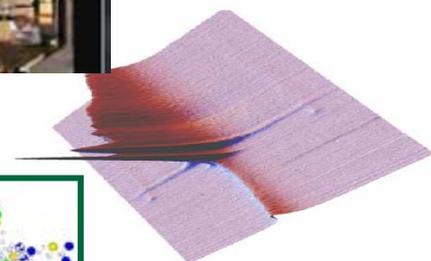
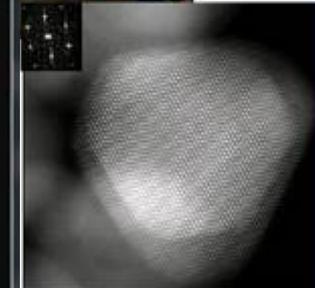
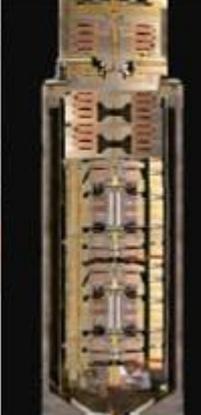
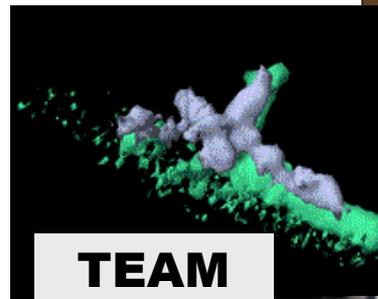
Capabilities

- ❖ Four probe STM
 - Independent operation
 - In situ amplifiers for transport
 - 10-300 K
- ❖ Scanning Electron Microscopy
 - 7 nm resolution
 - Accurate positioning of four tips
 - Electron beam induced current
- ❖ Scanning Auger Microscopy
- ❖ Sample preparation
 - MBE growth
 - Sputtering, cleaving



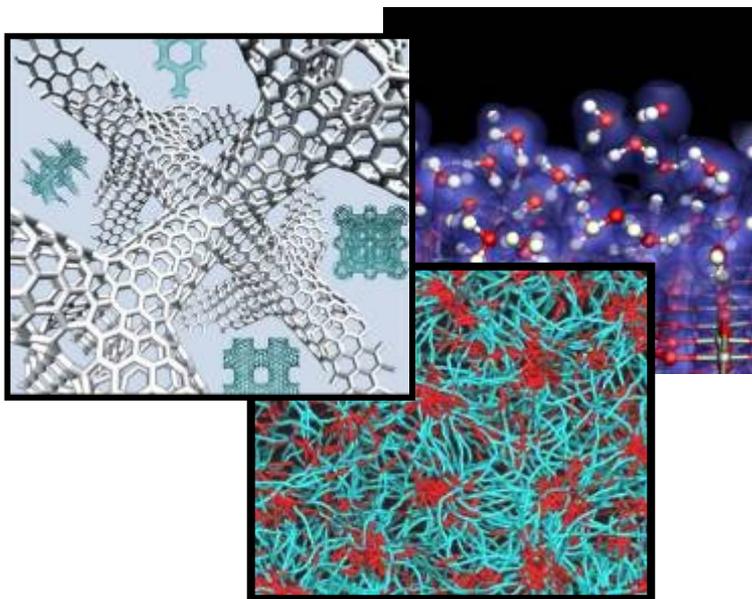
Partnerships with DOE's Electron Microscopy Facilities

- National Center for Electron Microscopy at LBNL
- Shared Research Equipment Facility at ORNL
- Electron Microscopy Center at ANL
- TEAM Microscope on line at LBNL with 0.5 Angstrom resolution
 - Commercial Aberration-Corrected STEM and TEMs at other facilities
- Local Electron Atom Probe



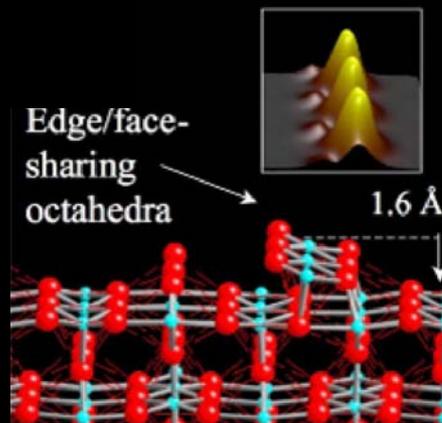
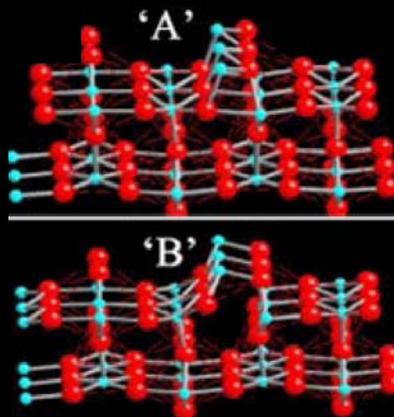
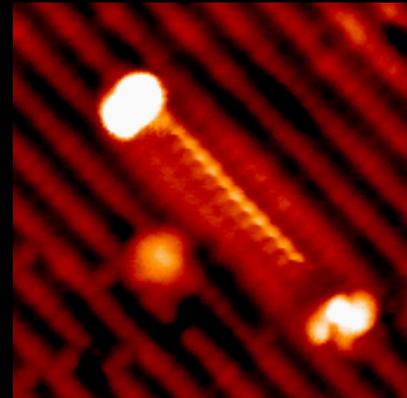
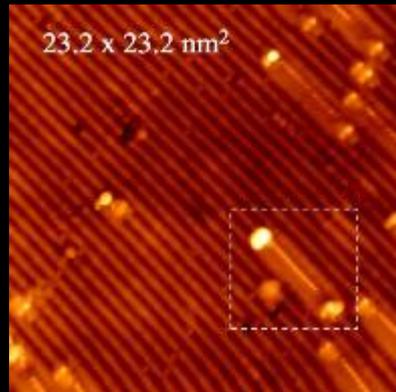
Computation and Visualization

- Access to 250 teraflop Leadership Class Computer
- NSRC Clusters
 - 5 teraflop (CNMS)



Where high-resolution experiment and large scale calculations meet... ..and succeed

High resolution Experiment...



... and first principles theory

Surface Reconstructions of $\text{TiO}_2(110)$ Driven by Suboxides

K. T. Park,^{1,2} M. H. Pan,^{3,4} V. Meunier,^{5,3} and E. W. Plummer^{2,4,3}

(Received 9 March 2006)

Scanning tunneling microscopy and density functional theory are used to develop a new structural model for surface reconstructions driven by Ti interstitials on $\text{TiO}_2(110)$. Ti interstitials form the edge- or face-sharing octahedra that serve as building blocks for (1×1) reconstruction. Thus, contrary to conventional wisdom, the 1×1 periodicity is insufficient to establish the correct surface stoichiometry. Furthermore, in our structural and compositional model the reversible oxidation or reduction between (1×1) and (1×2) is entirely achieved by transfer of the added rows.

Scientific Capabilities of the NSRCs (FY07)

▪ CNMS (ORNL)

- Nanomaterials theory institute
- Bio-inspired nanomaterials
- Macro-molecular systems
- Magnetism, transport, & scanning probes
- Structure/dynamics: neutrons, electrons, x-rays
- Catalysis & nano-building blocks
- Functional nanomaterials

▪ Foundry (LBNL)

- Theory of nanostructured materials
- Biological nanostructures
- Organic nanostructures
- Imaging and manipulation of nanostructures
- Nanofabrication
- Inorganic nanostructures

▪ CINT (SNL/LANL)

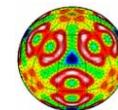
- Theory & simulation
- Soft, biological, & composite nanomaterials
- Nano-electronics, mechanics, systems
- Nanophotonics & optical nanomaterials

▪ CNM (ANL)

- Theory & modeling
- Nano-Bio interfaces
- Electronic & magnetic materials & devices
- X-ray imaging
- Nanofabrication
- Nanophotonics

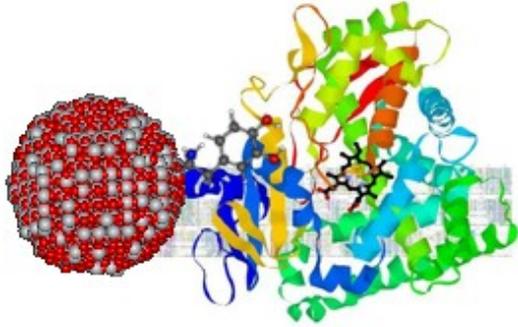
▪ CFN (BNL)

- Theory & computation
- Biological and soft nanomaterials
- Electronic nanomaterials
- Nanocatalysis



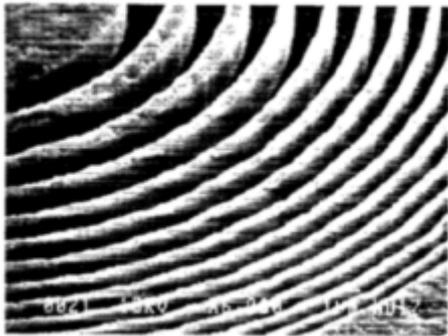
Center for Nanoscale Materials: Six Integrated Themes

NanoBio Interfaces



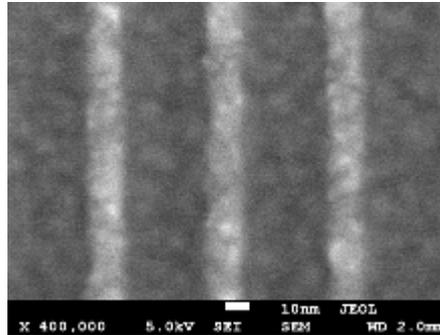
Create bio-inspired materials and processes for energy transduction

X-ray Microscopy



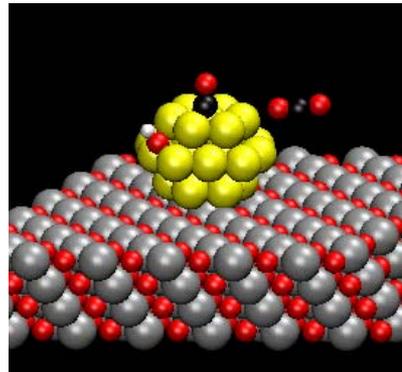
Create images of the nanoworld with hard x-rays

Nanofabrication



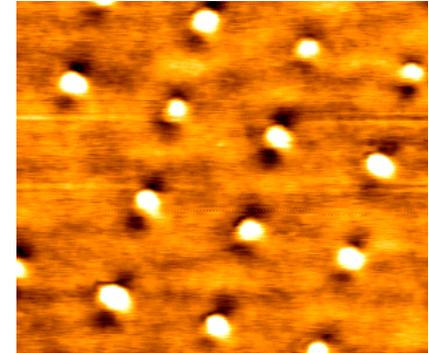
Discover new paths for nanostructured materials, including below 10 nm

Theory & Modeling



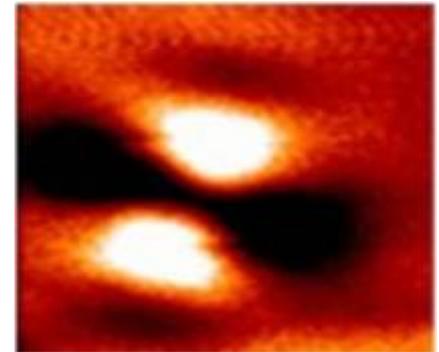
Towards the 'virtual fab lab'

Nanophotonics



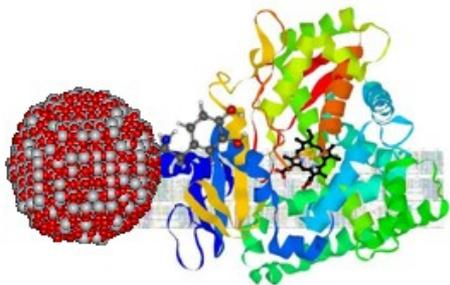
Understand and control optical energy pathways

Electronic & Magnetic Materials

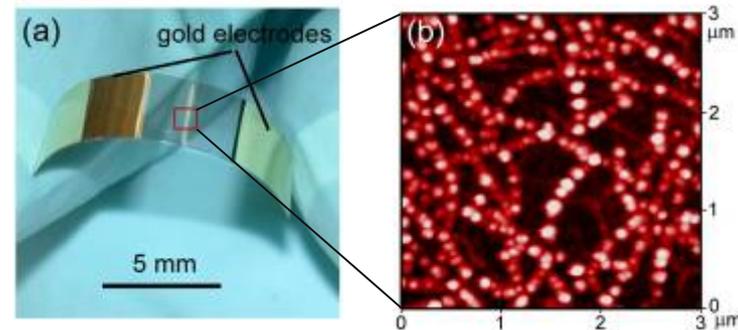


Understand and control charge and spin-based materials for energy and information transport

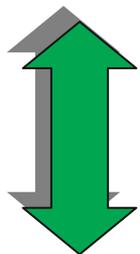
An Integrated Facility for Nanoscience Research



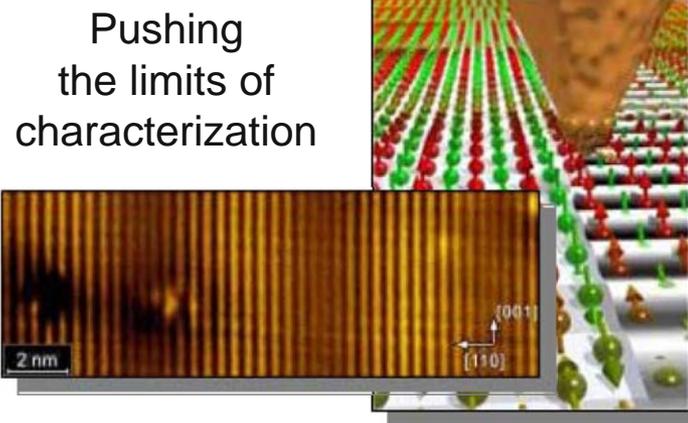
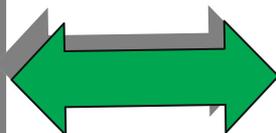
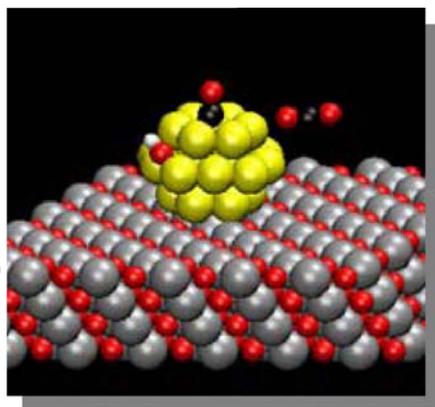
Materials creation:
Synthesis & assembly for control
of processes and function



Nanofabrication of novel architectures
and devices



Virtual Fab Lab
(theory):
Guiding the search,
understanding
functionality



Pushing
the limits of
characterization

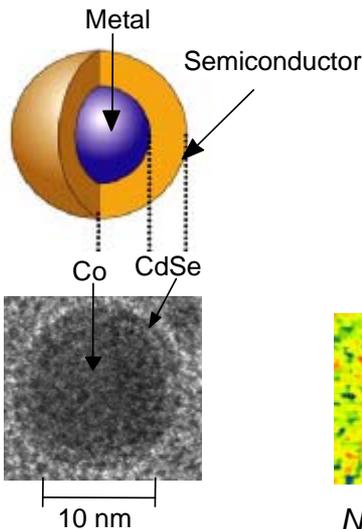


CINT's focus is on Nanoscience Integration

The science of nanomaterials integration

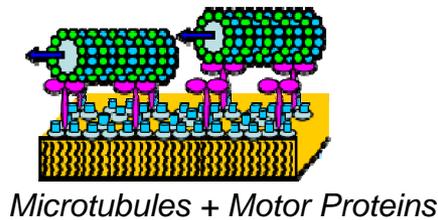
Combining diverse nanomaterials together into composite structures and systems from the nano to microscale to discover, understand, and design new properties and performance of materials.

Bifunctional materials



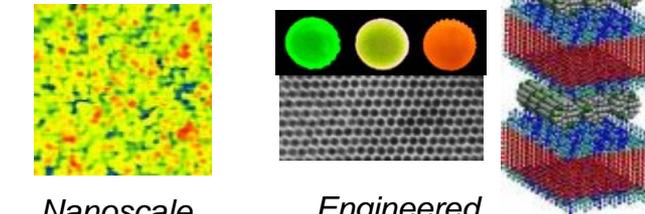
Combining ferromagnetic & semiconducting behavior

Directed assembly



Microtubules + Motor Proteins

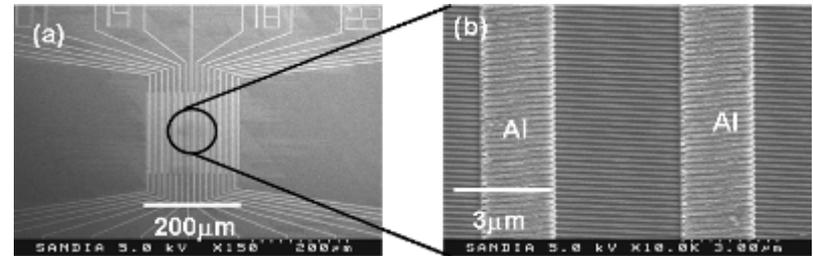
Nanocomposite materials



Nanoscale inhomogeneities

Engineered nanocomposites

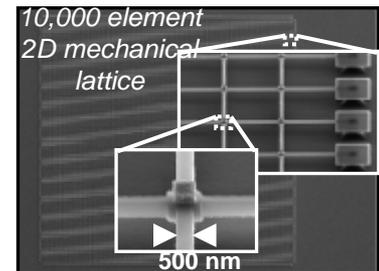
Active nanosystems



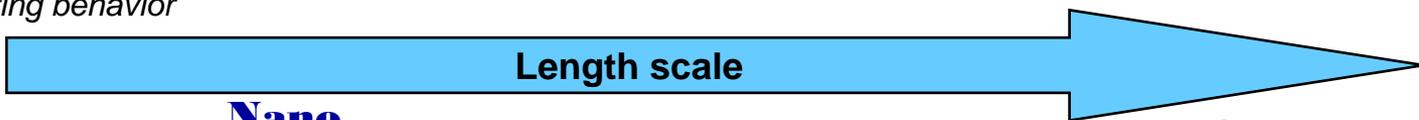
Nanowire arrays



Switchable metamaterials



Nanomechanical arrays



Nano

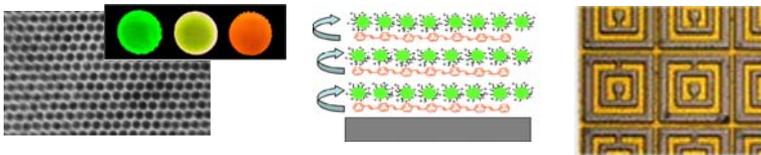
Micro



CINT's capabilities reside in four Science Thrusts

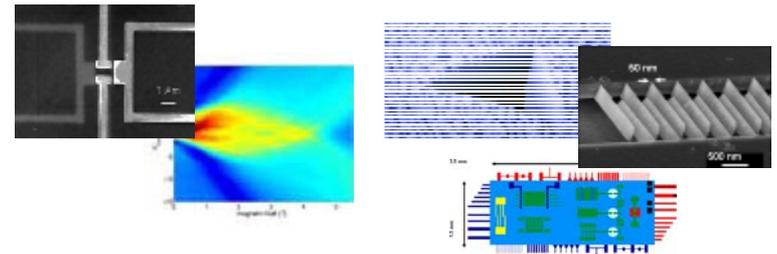
Nanophotonics & Optical Nanomaterials

Synthesis, excitation and energy transformations of optically active nanomaterials and collective or emergent electromagnetic phenomena (plasmonics, metamaterials, photonic lattices)



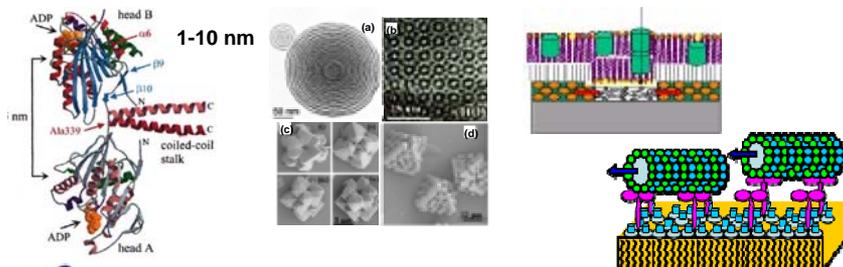
Nanoscale Electronics, Mechanics & Systems

Control of electronic transport and wavefunctions, and mechanical coupling and properties using nanomaterials and integrated nanosystems



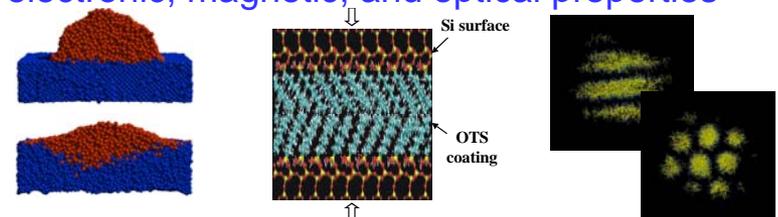
Soft, Biological, & Composite Nanomaterials

Solution-based materials synthesis and assembly of soft, composite and artificial bio-mimetic nanosystems



Theory & Simulation of Nanoscale Phenomena

Assembly, interfacial interactions, and emergent properties of nanoscale systems, including their electronic, magnetic, and optical properties



Center for Functional Nanomaterials

Brookhaven National Laboratory

A User-Oriented Research Center



**Focus on Functional Nanomaterials for Energy-
Relevant Applications**

Scientific Themes

- Interface Science and Catalysis
- Electronic Nanomaterials
- Soft/Bio Nanomaterials

CFN Facilities

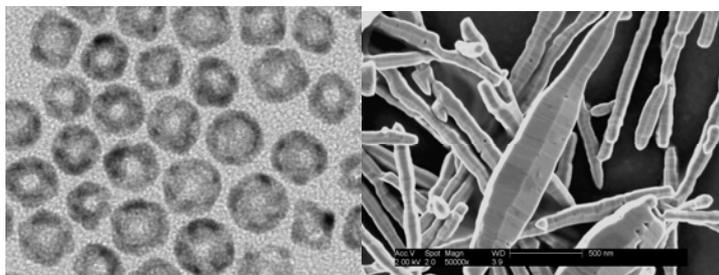
- **Materials synthesis:** Chemical vapor deposition and other synthesis methods for the growth of nanowires and quantum dots; biofunctionalization of nano-objects and surfaces
- **Nanofabrication:** Nanopatterning via optical, electron-beam and nanoimprint lithography; wet or reactive-ion etching, focused ion-beam, thin-film deposition by evaporation and sputtering for materials processing and device fabrication in a class-100 facility
- **Proximal probes:** An array of scanning probe tunneling and atomic force microscopies for advanced surface and interface analysis
- **Electron microscopy:** Most advanced transmission electron microscopy; allows the study of electronic, magnetic and optical properties at the atomic level
- **Optical spectroscopy:** CW and ultrafast spectroscopy tools for the study of optical processes, and their dynamics, in nanomaterials, down to single molecules
- **Dedicated beamline at the NSLS:** Especially designed for small- and large-angle x-ray scattering and ideally suited for the study of soft materials and interfaces
- **Theory & computation:** Staff and computational tools directed to understanding the formation and structure of nanoscale materials and associated electronic, optical and chemical phenomena



Molecular Foundry Facilities

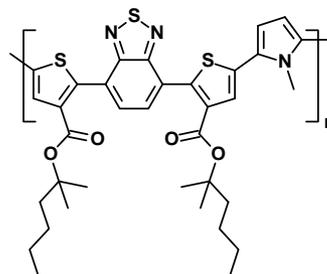


Inorganic



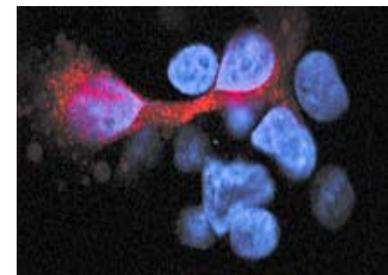
A. Paul Alivisatos

Organic



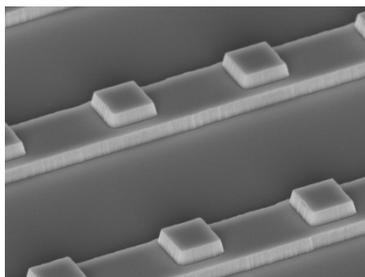
Jean Fréchet

Biological



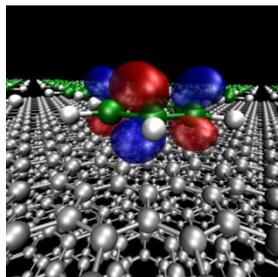
Carolyn Bertozzi

Nanofabrication



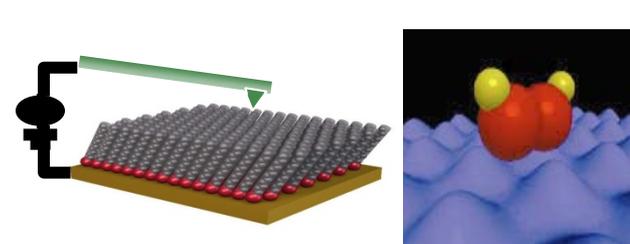
Jeffrey Bokor

Theory



Steven G. Louie

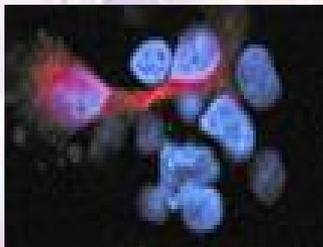
Imaging & Manipulation



Miquel Salmeron

Foundry Themes

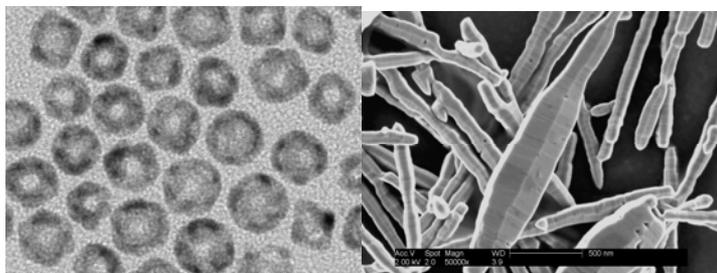
- Multimodal in situ nanoimaging and spectroscopy
- Combinatorial nanoscience
- Single-digit nanofabrication
- Synthesis and characterization of nanointerfaces



Molecular Foundry Facilities

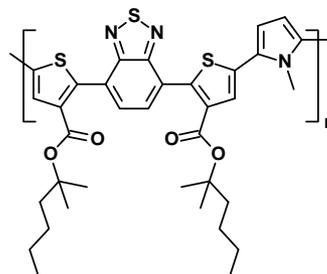


Inorganic



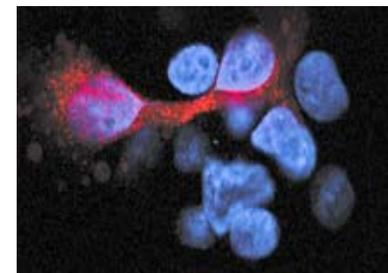
A. Paul Alivisatos

Organic



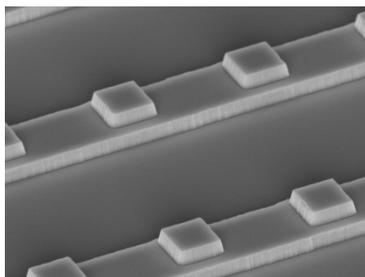
Jean Fréchet

Biological



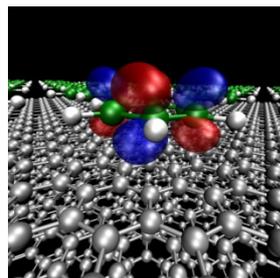
Carolyn Bertozzi

Nanofabrication



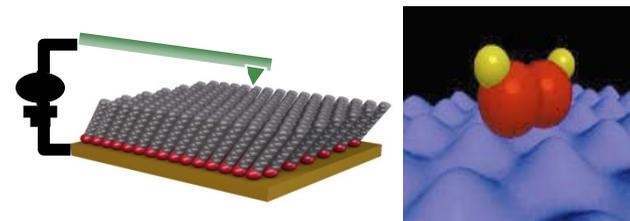
Jeffrey Bokor

Theory



Steven G. Louie

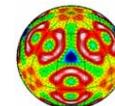
Imaging & Manipulation



Miquel Salmeron

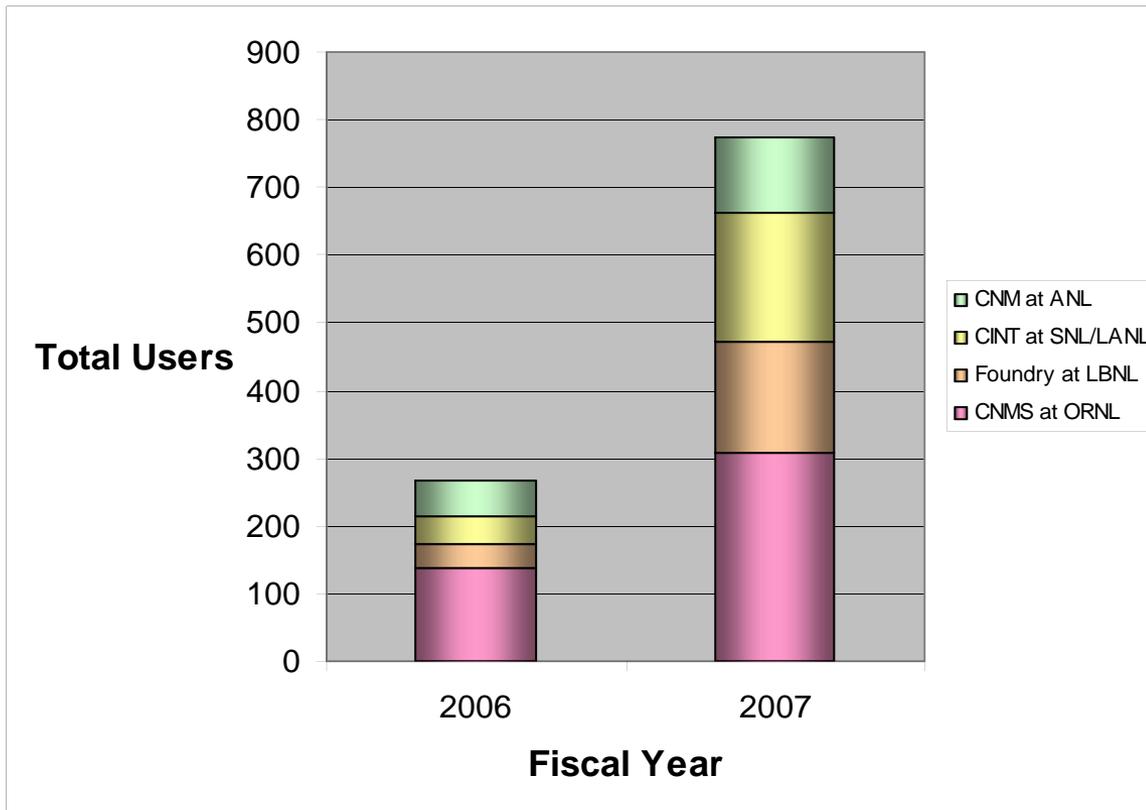
User Definition and Access

- General and Partner Users
 - Access based on peer merit review of submitted proposal, evaluated by external Proposal Review Committee or equivalent
 - Includes on-site (badged), remote, and off-site users
 - Partner Users also enhance capabilities of and/or contribute to operation of facility, with benefits to the general user community and may have defined, limited periods of reserved time or preferential access
- Proprietary Users
 - Access may be based on internal review to preserve confidentiality
 - Full cost recovery required
- A limited amount of time may be allocated directly at the discretion of the NSRC Director or management for rapid access
- Collaboration with facility staff is a potential user benefit, but is not required
- Facility staff may provide input on feasibility and time needed but do not select or approve proposals



User Statistics

Numbers of unique users, including badged users, remote users, and, starting FY 2007, off-site users. FY 2006 data is partial-year as three NSRCs began operations in mid-FY. Totals, FY 2007:

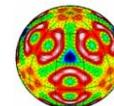


- **Badged Users:**
626
- **Remote Users:**
40
- **Off-Site Users:**
108

Definitions of NSRC Users

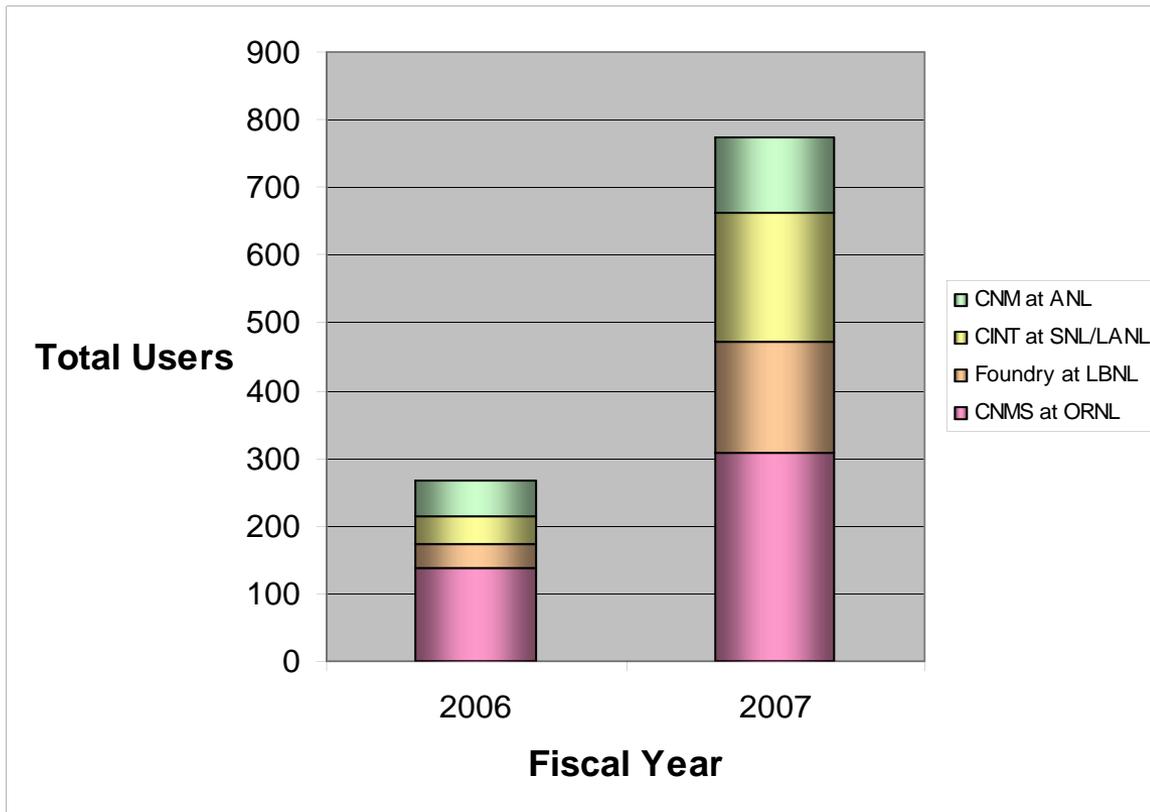
- The primary type of user is a **Badged User**, i.e., a researcher who conducts research within the facility.
- There are two other types of users who conduct research:
 - (1) **Remote User** - a researcher who has been granted authority to remotely produce data (excludes just “looking at data”)
 - (2) **Off-Site User** - a researcher to whom the facility provides custom-manufactured materials, tools, or devices that the facility has unique or unusual capabilities to fabricate (only applies to NSRCs; starting 2007).

Note: Only one user counted per proposal and only if no individual is counted in any of the other user categories under the same proposal.
- For annual totals, an individual is counted as 1 user no matter how often or how long the researcher conducts research at the facility during the fiscal year.



User Statistics

Numbers of unique users, including badged users, remote users, and, starting FY 2007, off-site users. FY 2006 data is partial-year as three NSRCs began operations in mid-FY. Totals, FY 2007:



- **Badged Users:**
626
- **Remote Users:**
40
- **Off-Site Users:**
108

FY07 CNMS User Statistics: 309 Unique Users (~240 so far in FY08)

- ~60% US Academic
- ~5% Industrial (increasing)
- ~25% from ORNL
- ~75% first time users
- >130 Refereed journal publications
 - Nearly 20% in high-impact journals (impact factor >5 and/or top journal in the field)
- 2 Calls for Proposals per FY
 - 8 week turn-around
- <70% proposal acceptance
 - ~50% accepted in first call in FY08
- Rolling Rapid Access

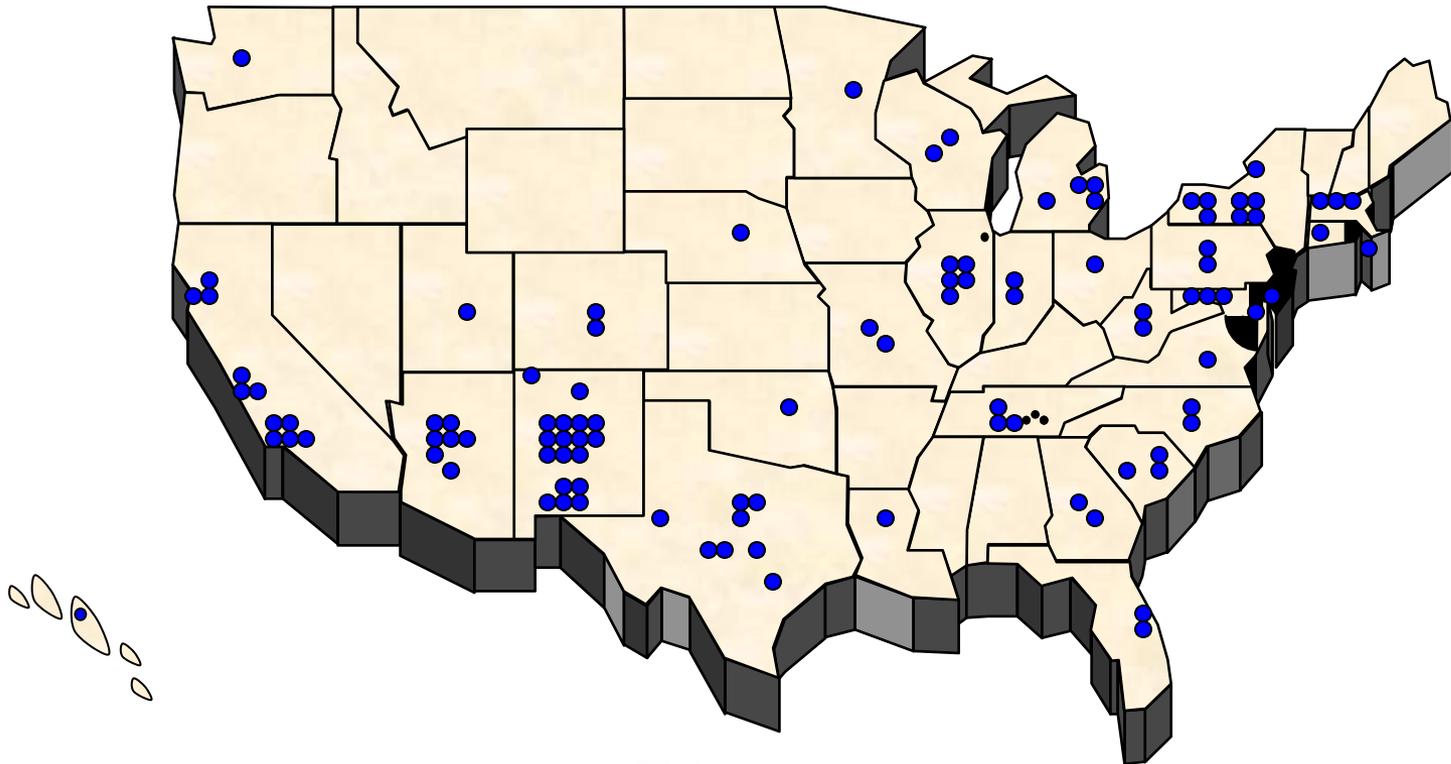
Typical US Geographic
Distribution of User
Proposals



Users come from across the
Globe: ~10% International

User Distribution - CINT

- Also have international users (~10 countries)



Center for Nanophase Materials Sciences Industrial Users

Integration of High Surface Area Electrodes with Microfluidic Structures

Nanotek LLC



“I had a fundamental interest in nanotechnology for many years ... but the real launching pad for me deciding to do this was the CNMS It is absolutely the cornerstone.”

Feasibility Study for Generating Continuous, Patterned Metal Thin-Films on Fragile Surfaces using Buffer Layer Assisted Growth

Hewlett-Packard Labs



“CNMS staff provided the fundamental knowledge and experimental expertise to give us a foot-hold in this new area of materials deposition. Without this user access to CNMS, our own project would have been delayed by months or years.”

Effective Spin Hamiltonian Theory of Fe(Co)-Pt(Rh) Alloy Nanostructures

Seagate Research



High Performance Simulations of Polymer Nanoparticle Composites at Mesoscopic Scale

Tech-X Corporation



Development of Band Excitation Piezo Force Microscopy for Atomic Force Microscopy
Asylum Research Corp.



Ultrafast Laser Nanostructuring
Lexitek, Inc.



User Agreements and Intellectual property

Pre-Competitive NSRC User Agreement

No.

BETWEEN

(" CONTRACTOR ")
under the U.S. Department of Energy Contract No.

AND

("USER")
(Collectively, "the Parties")

ARTICLE I: SCOPE OF SERVICES

CONTRACTOR will make available to USER certain Nanoscale Science Research Centers (NSRC) facilities, equipment, services, information and other material, on a collaborative basis, as described in Appendix A. Appendix A shall set forth the technical scope of work, including deliverables, to be performed pursuant to this Agreement. The scope of work shall not be considered proprietary information and shall be publicly releasable. The Parties agree that an initial abstract of the work to be performed shall be a deliverable under this Agreement.

Access to and use of the NSRC facility is subject to the prior written approval of the Scientific Director of the NSRC facility and available NSRC scheduling therefor. It is understood and agreed that the approval determinations of the Scientific Director of the NSRC facility are final.

ARTICLE II. TERM OF THE AGREEMENT

This agreement shall have a term of ___ months/years from the effective date. The term of this agreement shall be effective as of the date on which it was signed by the last of the Parties.

ARTICLE III: COST Each Party will bear its own costs and expenses associated with this Agreement. No money will be transferred to or from either Party as consideration, in whole or in part, for this Agreement.

ARTICLE IV: ADMISSION REQUIREMENTS

A. **General** - USERS are subject to the administrative and technical supervision and control of CONTRACTOR; and will comply with all applicable rules of CONTRACTOR and DOE with regard to admission to and use of the NSRC facility, including safety, operating and health-physics procedures, environmental protection, access to information, hours of work, and conduct. USER is required to obtain agreements from each of the employees or representatives as necessary to implement the provisions of the Agreement. USER's employees will not be considered employees of CONTRACTOR for any purpose.

- User agreements, authorities, intellectual property, and related issues extensively discussed among DOE-BES, DOE-General Counsel, and NSRCs
- Existing authorities and types of user agreements available for:
 - Non-proprietary, essentially non-collaborative work
 - Proprietary, non-collaborative work (full cost recovery)
- New authority and user agreement template developed for pre-competitive (non-proprietary), collaborative work
- Existing authorities available on case-by-case basis for proprietary, collaborative work (CRADAs, WFO, etc.)

DOE is currently evaluating User Agreements:
Uniformity across all Labs, Simplify, Fewer types – Stay Tuned

An Opportunity: DOE's Experimental Program to Stimulate Competitive Research (EPSCOR) Annual Meeting

- Will be held in Oak Ridge this year
- July 22-25, 2008
- Attended by Representatives from the EPSCOR states
 - *Alabama, Alaska, Arkansas, Delaware, Hawaii, Idaho, Kansas, Kentucky, Louisiana, Maine, Mississippi, Montana, Nebraska, Nevada, New Mexico, North Dakota, Oklahoma, Rhode Island, South Carolina, South Dakota, Tennessee, Vermont, West Virginia, Wyoming, the Commonwealth of Puerto Rico, and the Virgin Islands*
- Opportunity for outreach to future users
- See Judy Trimble for information



www.cnms.ornl.gov

