

# *Center for Nanophase Materials Sciences*

## *Scientific Vision and Progress Report*



**Doug Lowndes, Scientific Director**  
**Linda Horton, CNMS Project Director**  
**and Deputy Scientific Director**

**50<sup>th</sup> BESAC Meeting**  
**February 23, 2004**

# Outline

- **CNMS Timeline, Building, and Status**
- **Scientific Vision, Research Themes, Operational Organization**
- **Unique and State-of-the-Art Capabilities for Nanoscience: *Neutrons, Instruments, Theory/Modeling***
- **“Jump Starting” an Outstanding User Program, and Eventual Scope**
- **The Scientific Management and Advisory Groups Team**



## CNMS TIMELINE

CNMS Public | ORNL | SNS |

[About CNMS](#)

[Contacts](#)

[ES&H](#)

[News & Upcoming Events](#)

[Procurement](#)

[Project Controls](#)

[Quality Control](#)

- FY03**      **June 19-20: First SAC meeting**
- FY03**      **July 18: CNMS GROUNDBREAKING**
- FY03**      **July 22: CALL FOR PROPOSALS for “jump start” user-initiated nanoscience research program**
  - Use existing ORNL facilities and expertise
  - Information distributed by E-mail and Web site
- FY03**      **August: Begin CNMS construction**
- FY04**      **February 19: BES Operations Budget Review**
- FY05**      **Complete construction: April 05 (cleanroom Jan 05)**
- FY05**      **Beneficial Occupancy (CD-4a): April 05**  
**Office/lab furnishings in place; begin moving some “jump start” user research into CNMS building**
- FY06**      **First full-year operating budget: *October 05***  
***User operations in all Scientific Theme areas***
- FY06**      **CNMS Project completion: September 06**  
**Initial technical equipment installed & accepted (CD-4b)**

# Building and Support Facilities



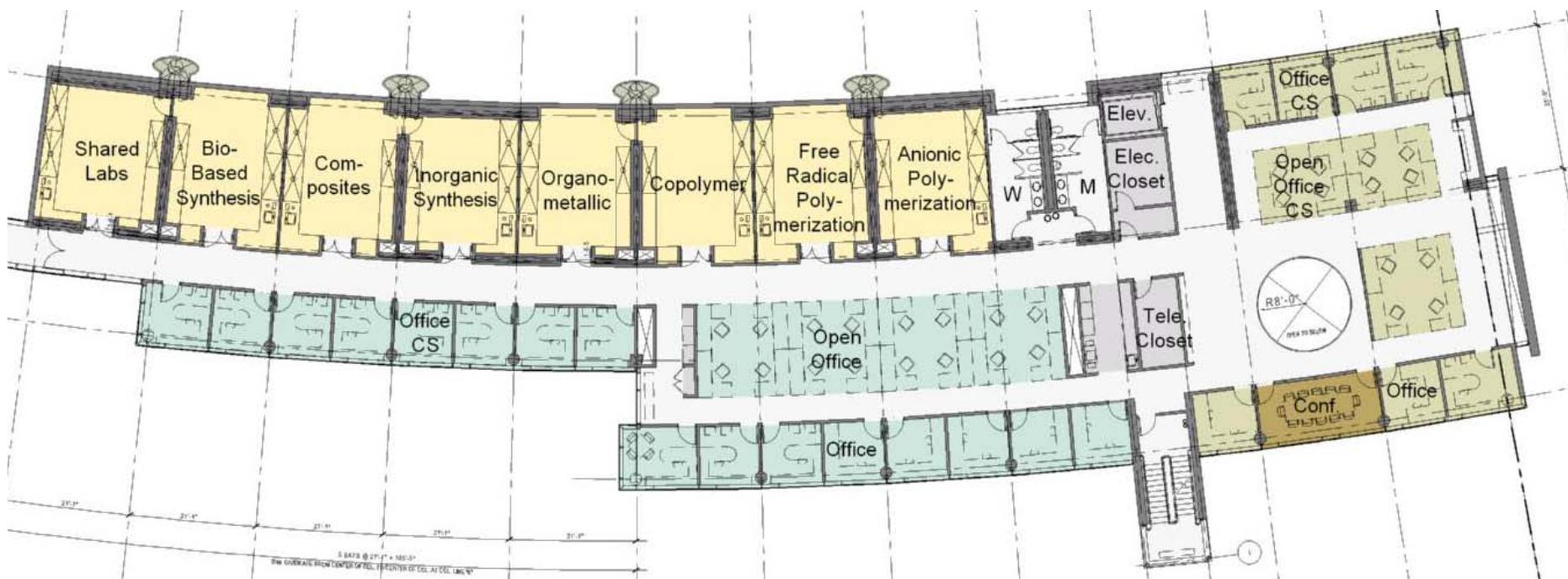
- 80,000 gsf: Four levels + Nanofabrication Research Lab (NRL, ~10,000 sf)
- 32 “wet” & “dry” synthesis / characterization labs (25’ x 20’ modules)
- Office space for 190 staff and visitors: *Immediately opposite labs to maximize collaborative, multidisciplinary, and educational interactions*
- **Nanomaterials Theory Institute:** Offices + lab to access terascale computing facilities of ORNL Center for Computational Sciences (CCS)
- CNMS 1<sup>st</sup> floor (adjacent to NRL): High-resolution scanning probes
- **NRL:** Clean and environmentally controlled rooms; electron microscopes; nanoscale patterning (e-beam writer / lithography); facilities for manipulation and integration of soft & hard materials

# Architecture to Maximize Collaborative, Multidisciplinary and Educational Interactions



Uppermost level (of four) at CNMS

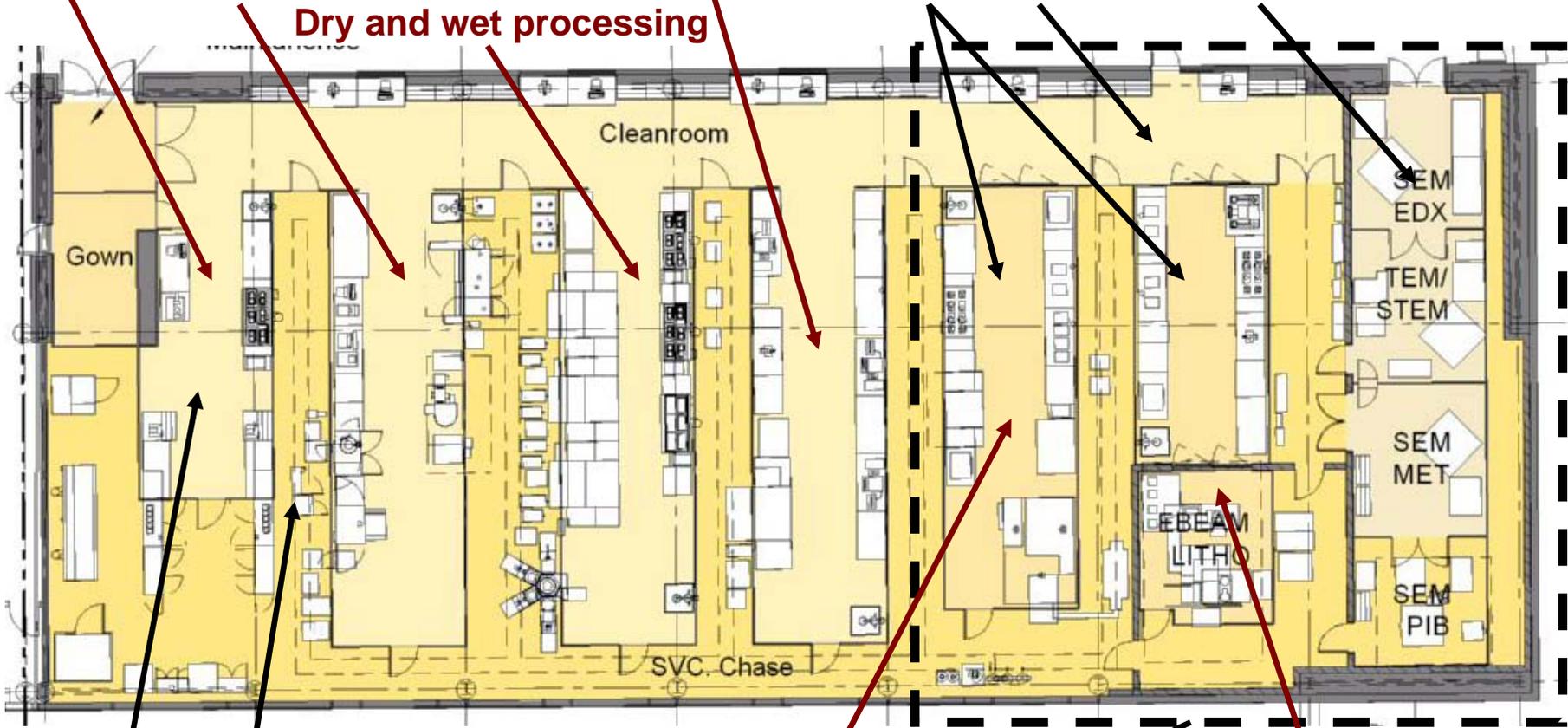
# Architecture to Maximize Collaborative, Multidisciplinary and Educational Interactions



Uppermost level (of four) at CNMS

# Nanofabrication Research Laboratory

Diffusion and LPCVD “Soft”-”hard” materials integration  
PVD and CVD films  
Dry and wet processing  
Class 100, 1000, & 100,000 clean areas



Bay/chase configuration

PHOTOLITHOGRAPHY

E-BEAM LITHOGRAPHY

Electromagnetic, vibration, acoustical (EVA) controlled area

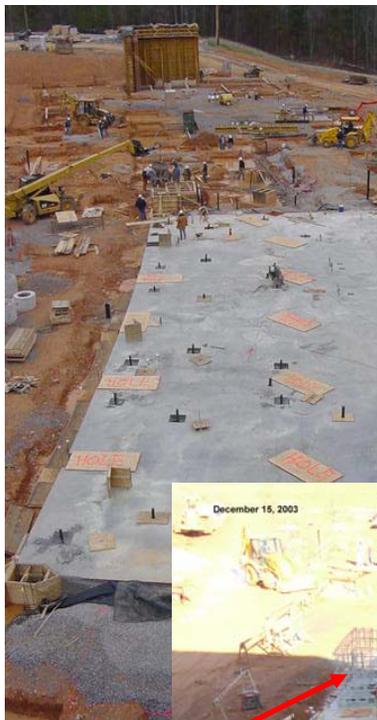
# CNMS: September....



**OAK RIDGE NATIONAL LABORATORY**  
**U. S. DEPARTMENT OF ENERGY**



# CNMS: December....



**Steel!!**



**Rebar cage**

**“The pit”**



**OAK RIDGE NATIONAL LABORATORY**  
**U. S. DEPARTMENT OF ENERGY**



# CNMS: February....



**First elevated  
concrete slab**

# CNMS on the SNS Site: Feb 19, 2004



**OAK RIDGE NATIONAL LABORATORY**  
**U. S. DEPARTMENT OF ENERGY**



*Vision and Plan for*  
**OUTSTANDING**  
**SCIENCE**



## *Vision and Plan for Outstanding Science*

# Center for Nanophase Materials Sciences

- **Co-located** with the Spallation Neutron Source (**SNS**) and the Joint Institute for Neutron Sciences (**JINS**) on ORNL's "new campus"

- **JINS**: Meeting rooms, classrooms, and support facilities for research visitors and students

- **SNS**: Provides access to *unique neutron scattering capabilities for nanoscience*

- **CNMS**: Provides urgently needed capabilities for *materials synthesis and characterization; nanofabrication; theory and modeling; and nanomaterials design*



***The CNMS Concept:  
Exploit scientific synergies  
to accelerate discovery in nanoscale science***

# Our Vision and Plan for the CNMS

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Create *an environment* to accelerate discovery and drive technological advances



- Nanoscale science is highly integrative: Bring together
  - the best *ideas* and the best *instruments*
  - a highly *interactive* and *multidisciplinary* user research community
- Developed in partnership with the national scientific community

# How Will we Create this Environment?

[1] **Exploit synergies with two rapidly emerging ORNL strengths, for national scientific leadership**

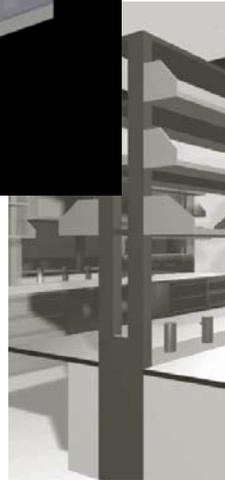
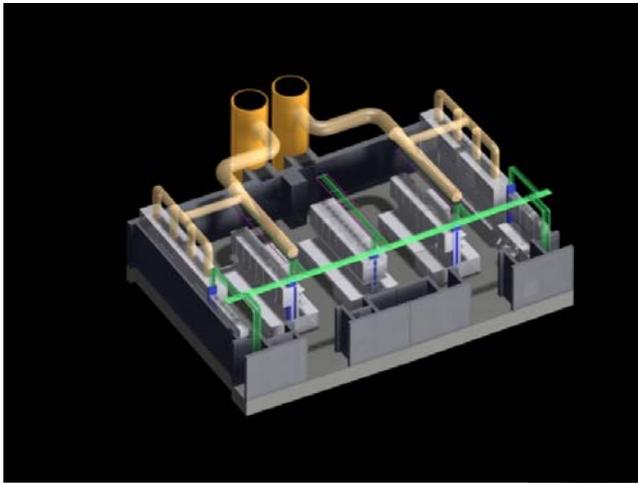
- **NEUTRON SCATTERING** (Spallation Neutron Source)  
Support development of neutron scattering **techniques and sample environments** needed to understand nanoscale phenomena
- **LEADERSHIP COMPUTING** (Center for Computational Sciences)  
Address grand challenges of **computational nanoscience** through the CNMS' **Nanomaterials Theory Institute**

[2] **Address the need for a new generation of nanoscience instruments**

- **COMBINE** nanoscale imaging with **simultaneous** sample-characterization and manipulation capabilities

**Operate CNMS to reliably deliver these unique capabilities to the national user community**

**CNMS supports**  
**a *FOCUSED* research agenda**  
**with a *HIGH* level of demand**



*Directly Engaging the Scientific Community*

2<sup>nd</sup> CNMS Planning Workshop

***Breakout Sessions***

**Purpose and Results**

- Define Candidate Research Focus Areas and Equipment Needs

**Greatest Challenges to Scientific Understanding**

**Greatest Opportunities for New Technology**

- Desired CNMS mode(s) of operation

# Scientific Themes for CNMS

CNMS' research is organized under **seven related scientific themes**, selected to address **grand challenges to understanding** and **nanotechnology needs**

## Macromolecular Complex Systems

Synthetic (polymeric) and bio-inspired materials

## Functional Nanomaterials

Nano- tubes, wires, dots, composites; artificial oxide film structures

## Nanoscale Magnetism and Transport

Reduced and variable dimensionality; quantum transport

## Catalysis and Nano-Building Blocks

Highly selective catalysts; nanoscale synthesis & organization

## Nanomaterials Theory Institute: Theory, Modeling, Simulation

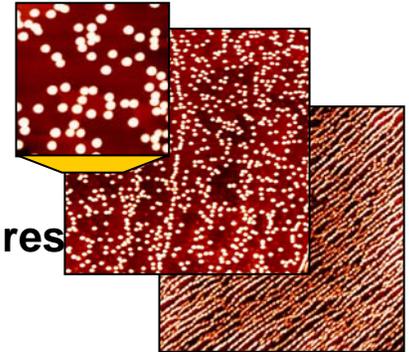
Grand challenges of “computational nanoscience”: Multi-scale modeling; nanomaterials design; virtual synthesis

## Nanofabrication Research Laboratory

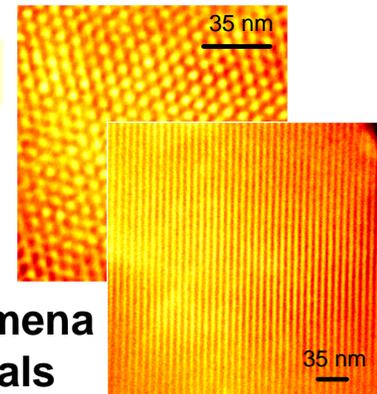
Controlled synthesis & directed assembly; linking nanoscale phenomena to the macroscale; functional integration of “soft” and “hard” materials

## Nanoscale Imaging, Characterization, and Manipulation

Unique instruments and methods to characterize and **manipulate** nanostructures, with **simultaneous imaging and environmental control**



AFM images of Fe nanodots and nanowires on flat and stepped NaCl surfaces (edge length 750 nanometers)



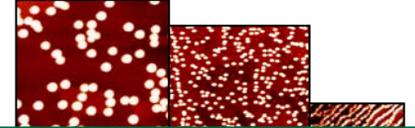
Ordered nanoporous silica synthesized using an organic template

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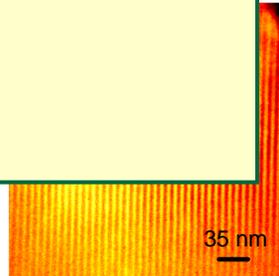
**CNMS' 7 Themes are not independent,**

**but are scientifically and operationally related**

Controlled synthesis & directed assembly; linking nanoscale phenomena to the macroscale; functional integration of “soft” and “hard” materials

## Nanoscale Imaging, Characterization, and Manipulation

Unique instruments and methods to characterize and **manipulate** nanostructures, with **simultaneous imaging and environmental control**



*Ordered nanoporous silica synthesized using an organic template*

*UNIQUE and  
State-of-the-Art  
Capabilities for  
Nanoscience*



# New Research Capabilities: Unique Instruments

## **TWO CLASSES of INSTRUMENTS for NSRCs**

- **Critical** to support the scientific research agenda, often expensive, but **not unique**
  - NANOSCALE: HR-SEM, E-beam writer, nanomaterials synthesis, ...
  - Can be purchased, together with maintenance contract
- **Truly unique**, offering **new research capabilities**
  - Currently **unavailable** to national community (new, complex, only “ $\beta$ -users”)
  - CRITICAL to ADVANCE NANOSCIENCE: **Simultaneous**
    - ▶ Imaging, combined with
    - ▶ Properties measurements
    - ▶ Sample manipulation, e.g. deposition, .....
    - ▶ Assembly
    - ▶ Environmental control
  - Critical to **attract forefront user nanoscience** and **scientists**

## Recommendations from the SAC

- **Recommend:** Begin immediately to highlight and develop new *capabilities that will be world-class*. These are *the most significant draw for new users* and help stimulate new research opportunities.
- **Recommend:** The external community represents a significant force driving development of new facilities or enhancing capabilities at many other national user facilities. *Every effort should be made to engage external users* in the development of new capabilities.

# Nanoscale Imaging, Characterization and Manipulation

***New techniques and instruments for imaging, characterization and manipulation of soft and hard materials, with environmental control***

- **Neutron and X-ray Scattering**

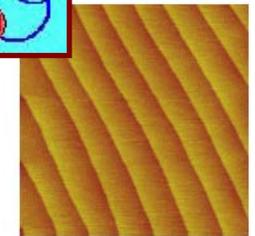
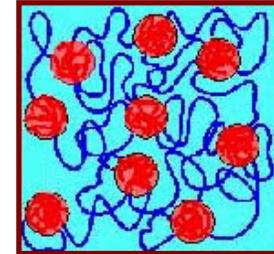
- Specialized scattering techniques and environments for nanoscience

- **UHV Scanning Probes**

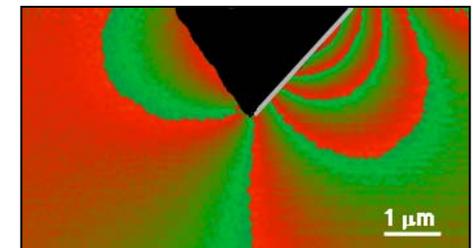
- UHV scanning probe microscopies for magnetic and quantum transport properties in nanostructured materials

- **Electron Microscopy and Spectroscopy**

- Combine imaging with characterization and/or manipulation methods
- Special sample environments (soft materials); in situ spectroscopy; integrated use of ambient scanning probes



0  $\mu\text{m}$  3  $\mu\text{m}$



***Provide technicians, staff, and budget to make UNIQUE instruments RELIABLY available to the national user community***

## *Vision for Outstanding Science*

# Neutron Techniques are Exquisitely Suited for Soft Materials

## • **Nanoscale Challenges to Synthesis and Understanding**

- Control of self-assembly and nanoscale structure
- Understanding how morphology, symmetry, structure, and phase behavior relate to function
- New approaches for rational design and fabrication of soft and hybrid materials

## • **Neutron scattering opportunities**

- **SANS** for nm-scale shape, location, and evolution
- **Reflectometry** for molecular-scale structure near surfaces and materials interfaces
- **H/D contrast** for component-by-component imaging on all nanometer length scales
  - > Dilute and concentrated systems
  - > “Fillers” to control block copolymer properties
  - > Proteins within complexes (“Machines of Life”)
  - > Selective migration of components to surfaces
  - > Interdiffusion in solutions
  - > Atomic-level details for MD simulations



See: John Ankner and Hartmut Zabel, “Applications of Neutron Reflectivity Measurements to Nanoscience”

*Vision for Outstanding Science*

# Unique and State-of-the-Art Measurement Capabilities for CNMS

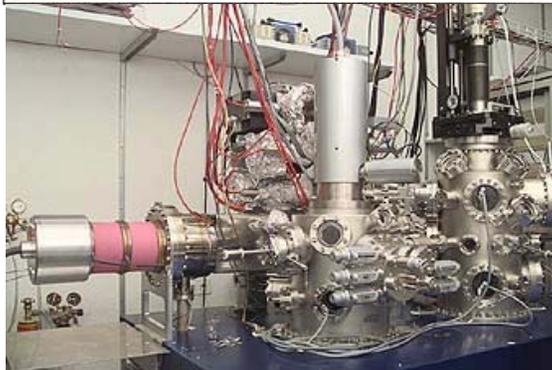
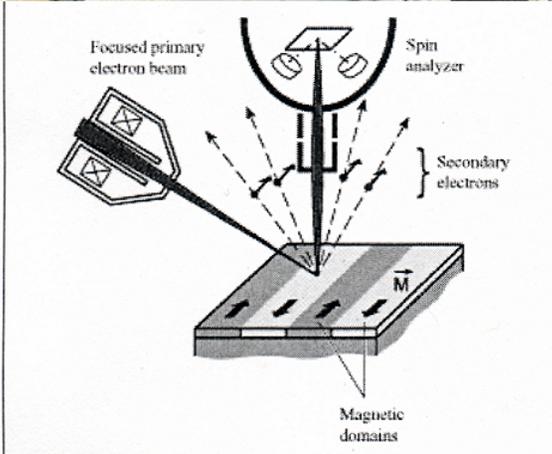
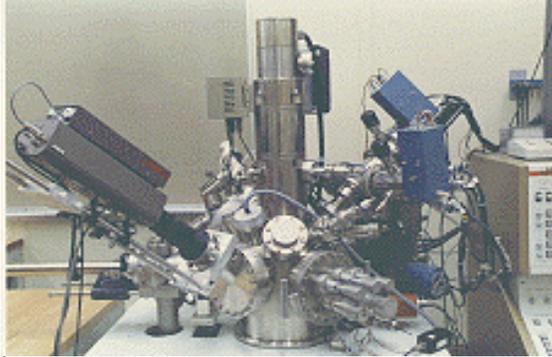
## ***Atomically-Resolved Imaging, Manipulation and Spectroscopy***

- In-Field SEMPA (SEM with polarized analysis of current)
- Low-Temperature, High-Field STM (the “Ultimate STM”)
- Four-Point-Probe STM
- Scanning Nano-SQUID
- Suite of Scanning Probe Laboratories (CNMS ground floor)

## ***In Situ Diagnostics of Nanomaterials Synthesis***

- Spectroscopic Diagnostics Facility for Nanomaterials & Film Growth
- Continuous Compositional Spread (rapid nano-catalyst evaluation)

# High-Resolution Scanning Electron Microscope for (Spin)-Polarized Analysis (SEMPA)



## • Scientific Drivers

- **Direct high-resolution (nanometer-scale) imaging of magnetic domain structure**
- Investigation of spin-switching and spin dynamics under magnetic field
- Correlation between chemical and magnetic inhomogeneities with SAM and SEMPA
- Elemental analysis of nanostructures via scanning Auger microscopy
- **Areas Impacted: Nanoscale magnetism  
Complex oxides**

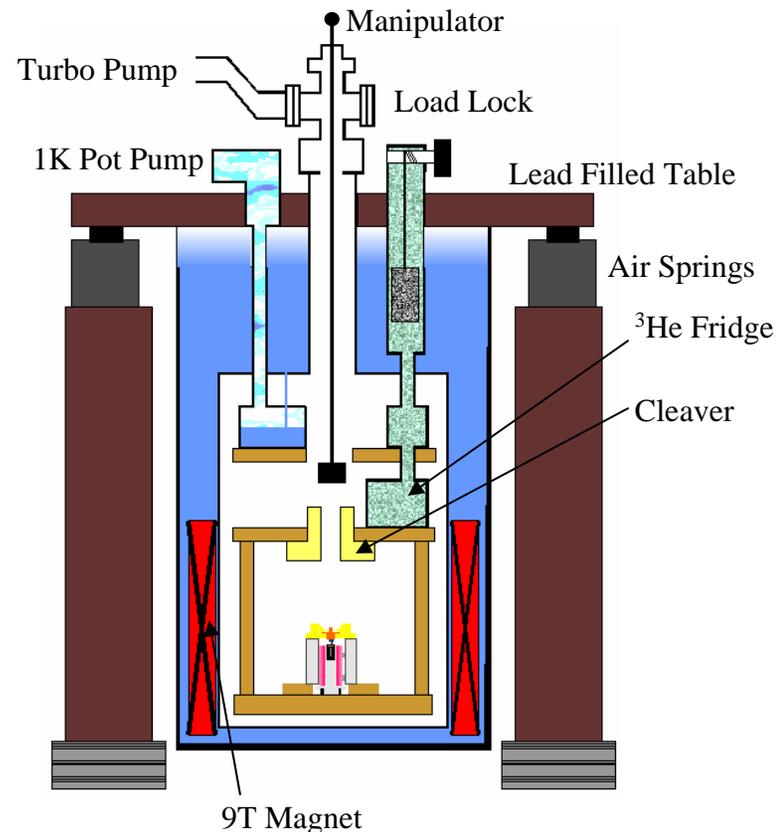
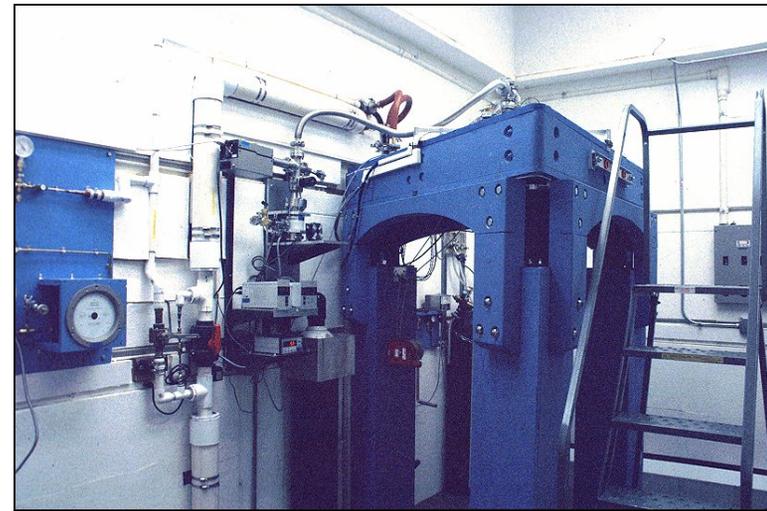
## • Proposed Capabilities

- UHV sample environment and sample preparation system;  $T = 50 \text{ K} \rightarrow 1000 \text{ K}$
- True UHV electron column with **15-nm spatial resolution**
- Spin detector based on the spin-polarized LEED detector
- Operates with in-plane magnetic field of 300 mT (to be improved to 800 mT)

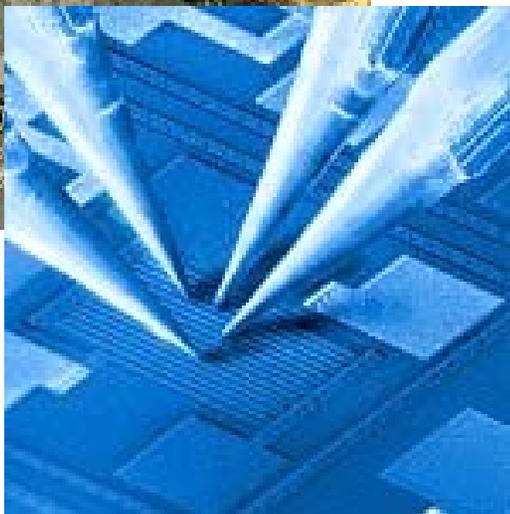
# The "Ultimate STM"

- Single-atom or -molecule spectroscopy
- **Atomically-resolved spectroscopy maps**  
(Requires vertical resolution of  $<0.0001$  nm,  $\sim 100X$  better than commercial instruments.)
- k-space **mapping of electronic structure**
- The temperature and magnetic field range to study the quantum response of nano-objects
- Optical access to the sample in magnetic field, for probing and exciting atoms or molecules
- Sample rotation (STM) in the magnetic field
- Flexibility to convert this STM to a magnetic scanning microscope with atomic resolution

- $300\text{mK} < T < 150\text{K}$
- $B_{\text{max}} \sim 9.0$  Tesla
- Sample exchange from RT
- Cryogenic UHV Sample Cleavage



# Four-point Probe STM with SEM: Manipulation & Transport in Nanoscale Systems



- **Scientific Drivers**

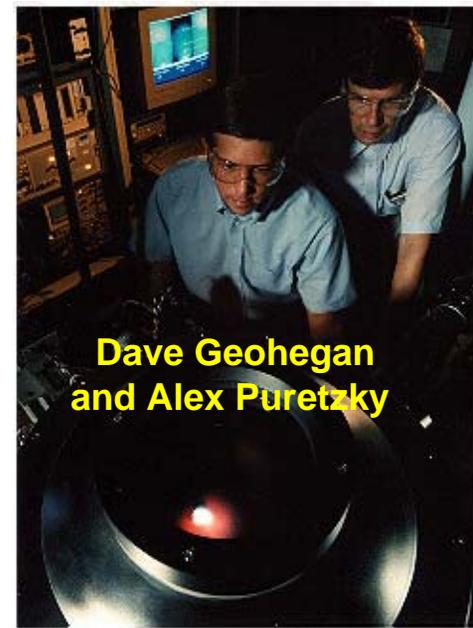
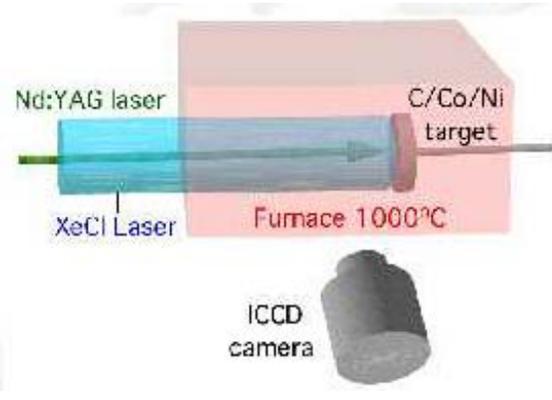
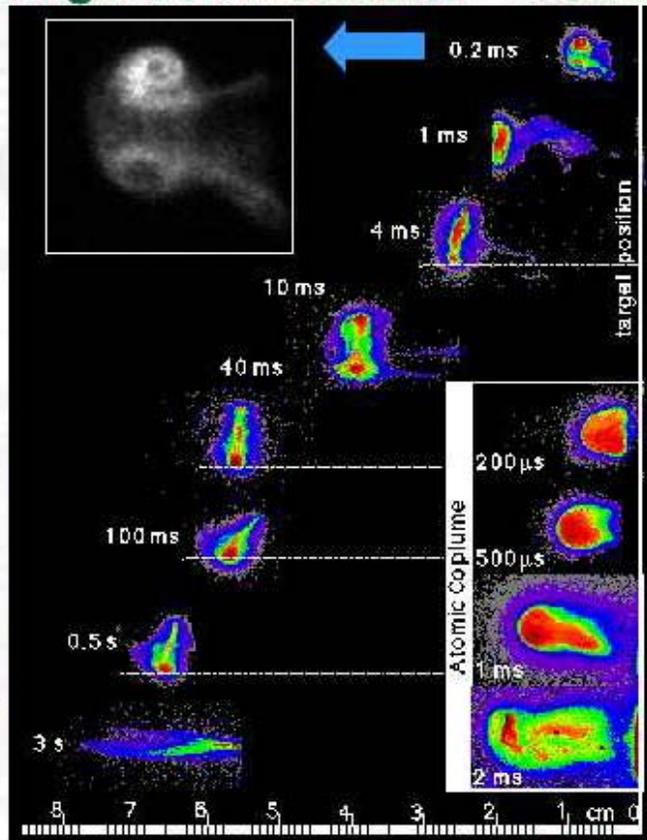
- Temperature-dependent quantum **electrical transport of nanoscale objects on surfaces**
- **Manipulation** of individual nano-objects
- **Fabrication** and **characterization** of nanoscale devices
- Spintronics / spin injection / spin transport

- **Proposed Capabilities**

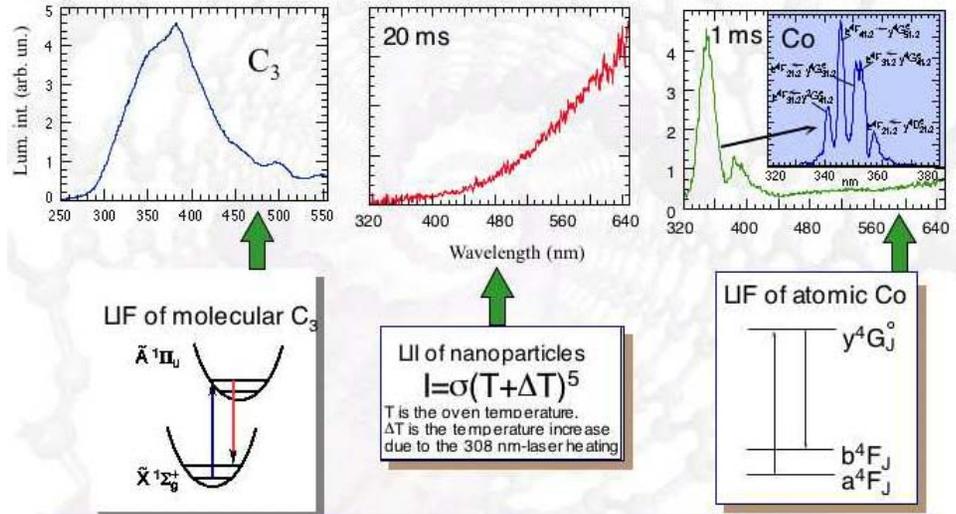
- **Four probes operate independently**, tip separation < 100 nm
- Integrated SEM with resolution < 10 nm permits accurate positioning of four tips relative to each other and to nanofeatures of interest
- 20 K < T < 600 K
- UHV-capable ( $5 \times 10^{-11}$  Torr)
- Integrated sample preparation / handling
- **Nanofabrication: STM tip-stimulated chemical vapor deposition (CVD)**

# Vision for Outstanding Science In situ Spectroscopic Diagnostics of Nanomaterials Growth

## Imaging and Spectroscopy Diagnostics of SWNT Growth



## Laser-Induced Emission Spectra of C/Co/Ni Plume at 1000° C During Nanotube Growth

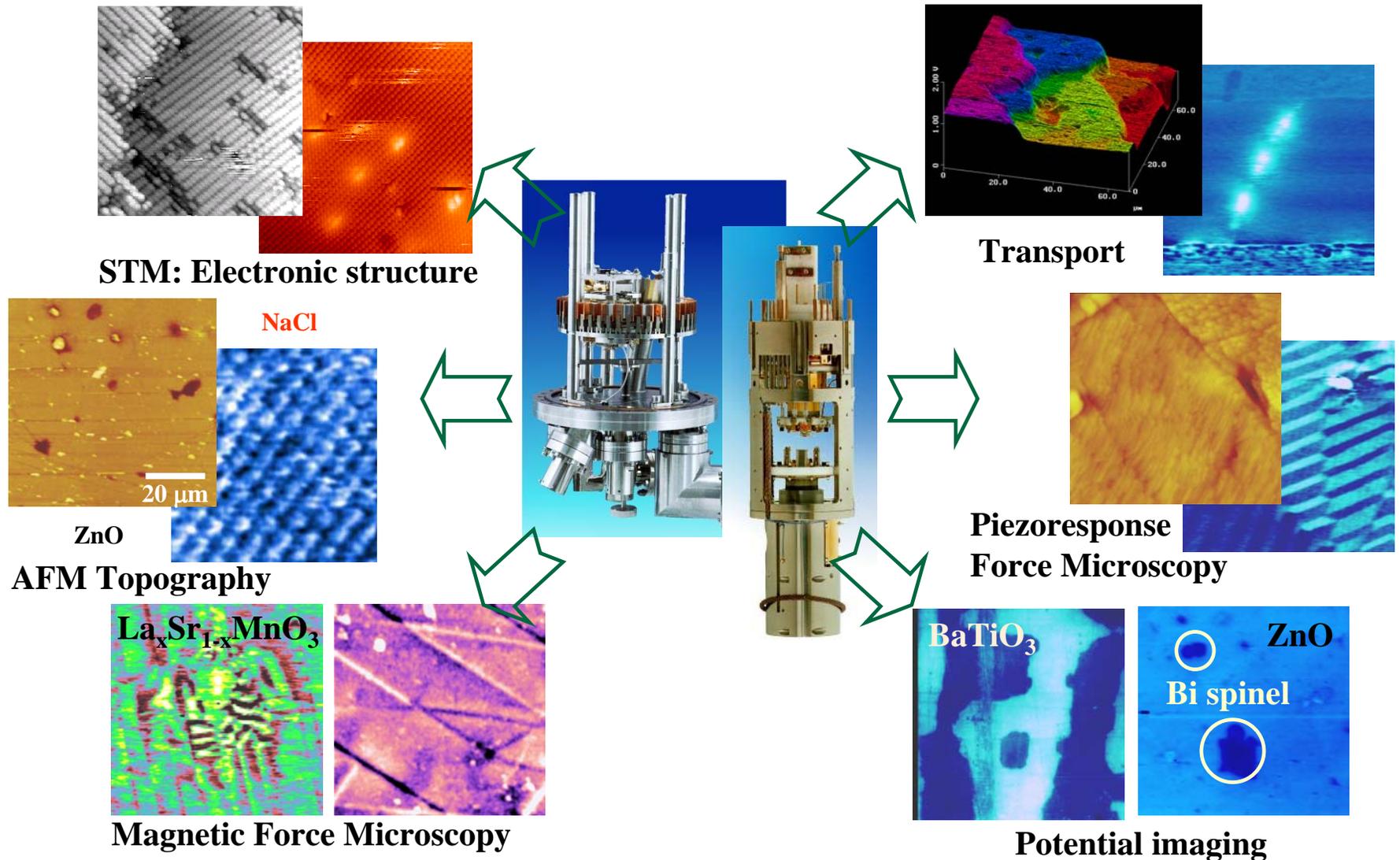


Using 308 nm-laser-induced emission we can monitor ground state species of  $C_3$  and Co and probe carbon nanoparticles in the C/Co/Ni plume.

# Spatially Resolved Characterization:

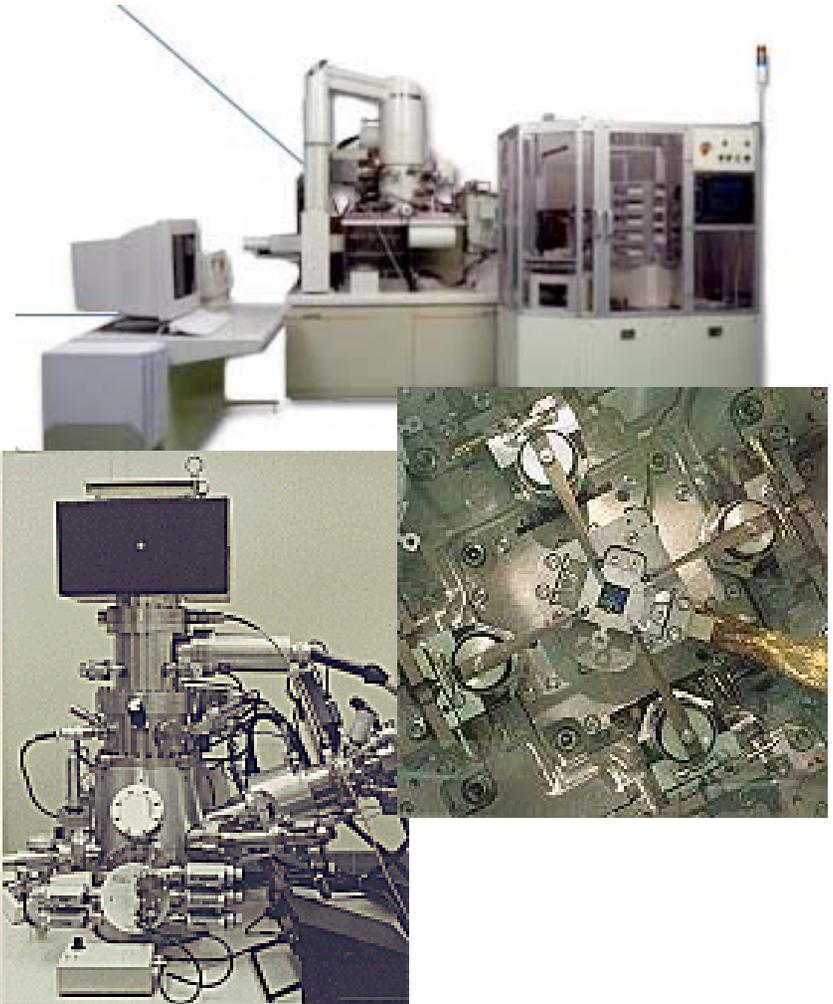
Atoms, Spins, Charge and Transport with Atomic Resolution

CNMS ground floor: Scanning Probe Laboratories Suite



# Ordering of CNMS Technical Equipment has Begun

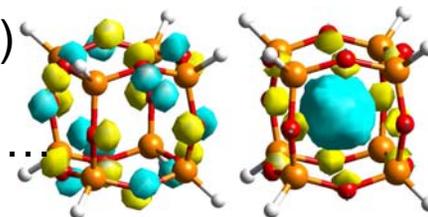
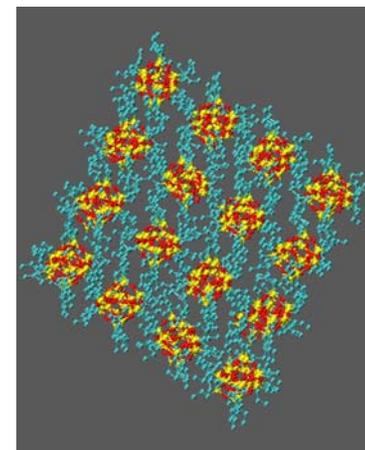
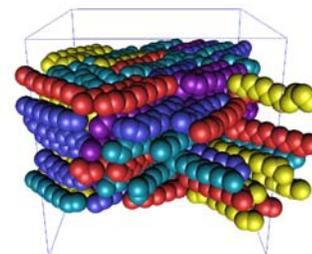
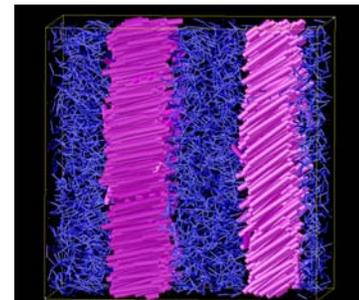
- **E-beam lithography system (order placed)**
- **SEMPA – order placed**
- **4-probe STM/SEM (bids under review, order expected in March)**
- **2 other pieces of clean room equipment are ordered and will be delivered in FY04**
  - **Used/refurbished equipment!**



## Vision for Outstanding Science

# Unique Instruments: Perfecting the “Computational Multiscope”

- **Current capability of computational nanoscience**
  - Excellent clarity for some systems at specific levels of description
    - ▶ Electronic structure scale, atomistic simulation scale, etc.
    - ▶ Analogous to a different lens at each length scale
  - *Moving from one scale to another* requires “changing out the lens”: switching to fundamentally different technique
- **Nanomaterials Theory Institute (NTI) of CNMS**
  - Develop scale-spanning methods and combine with leadership-class computing at ORNL’s CCS
  - Create a “computational multiscope”
    - ▶ Seamless clarity at length and time scales from electronic to macroscopic
    - ▶ Virtual synthesis, virtual experiments, device design,...
- **Enable this vision at the NTI by utilizing expertise of**
  - ~40 theorists and computational chemists, physicists, materials scientists at ORNL (esp. **NTI & CCS user support**)
  - **Users working with** NTI & CCS staff, Guest (visiting) Scientists, ORNL and shared postdocs, graduate students, ...



*“Jump Starting”  
AN OUTSTANDING  
USER PROGRAM*



# Initial User Program and Call for Proposals



The screenshot shows the CNMS website homepage. At the top, there is a green banner with the CNMS logo and the text "Center for Nanophase Materials Sciences", "A Highly Collaborative and Multidisciplinary", and "U.S. DOE Nanoscale Science Research Center". Below this is a navigation menu with links such as "CNMS HOME", "ABOUT CNMS", "FACILITIES", "RESEARCH", "BECOMING A USER", "WORKING AT CNMS", "PUBLICATIONS", "NEWS/HIGHLIGHTS", "UPCOMING EVENTS", "PEOPLE", "CNMS POSTDOCS AND FELLOWSHIPS", "CONTACT US", "CONSTRUCTION", and "PROJECT STATUS". There are also links for "OTHER NSRCS", "OTHER DOE/BES USER FACILITIES", and "DOE BASIC ENERGY SCIENCES". The main content area features a section for "UPCOMING EVENTS" with the text "Computational Nanoscience Workshops August 4-8 and 11-15" and a link "Click here to go to Workshops". Below that is a yellow box for "CALL FOR PROPOSALS" with the text "User-initiated Nanoscience Research Program" and a link "Click here for information about submitting a proposal". To the right of the main content is a vertical navigation menu with "HIGHLIGHTS", "Welcome to the Director", and "Workshops". At the bottom left, there is a logo for the "Office of Basic Energy Sciences" and a photograph of the CNMS building.

## SELECTION OF RESEARCH AREAS

- CNMS Planning Workshops: Expected **strongest user interest**
- **Major strengths** of current ORNL / BES research programs
- **Support for**
  - Controlled synthesis research
  - Broad range of imaging and characterization
  - Theory, modeling, and simulation
- User research initiated in 5 of 7 CNMS Scientific Themes
  - Some aspects of remaining two

## GOAL

- **Vibrant, interactive, productive user community *before* CNMS opens**

# Nanoscience Research with Users

- **Design, Synthesis, and Characterization of Macromolecular Materials**
  - **Materials Focus:** Polymers and biologically-derived or -inspired systems
  - **Grand Challenge:** Design and control macromolecular organization to achieve novel functionalities
- **Controlled Synthesis and Assembly of Functional Nanomaterials**
  - **Materials Focus:** Single- and multi-wall carbon (and eventually other) nanotubes and related composite materials
  - **Grand Challenge:** Understand and control synthesis and functionalization of nanotubes and related structures, to obtain materials with desired physical and chemical properties
- **FY2004: Initiate Nanomaterials Theory Institute's *User Research Focus Laboratories* program**
  - Development and application by users of selected, powerful computational nanoscience techniques, together with world leaders
  - Address key problems / issues *of users' choice* in understanding nanoscale materials and phenomena
  - Users run their applications "hands on" using supercomputers of ORNL's *Center for Computational Sciences (CCS)*

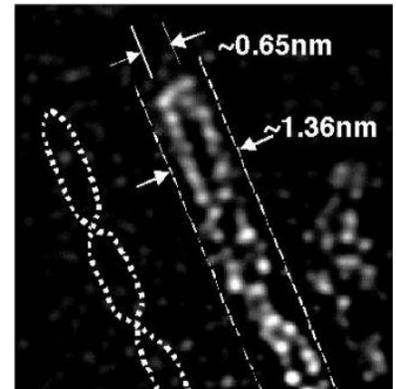
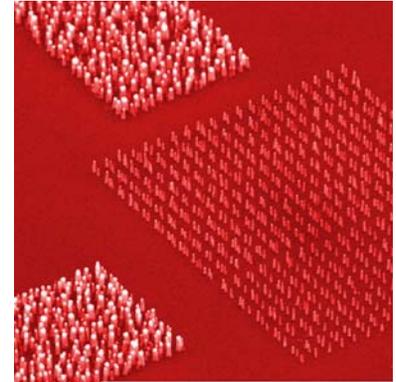
# Collaborative Outreach: Nanofabrication

- **Nanofabrication: Interim nanofabrication lab for users**

- Training / supervision for new users
- Skilled technicians for some tasks / users
- Communication and enforcement of safe-use and clean-use policies

- **Nanoscale Imaging and Characterization**

- Nanoscience user access to:
  - high-resolution electron microscopy
  - analysis instruments
  - scanning-probe instruments
  - SEMPA
- in SHaRE, High Temperature Materials Laboratory (HTML) Materials Analysis User Center, and MPI—Halle (Germany)



*Electron microscopy reveals a double helix chain of iodine atoms inside a carbon nanotube*



# Enthusiastic Response to Call for Proposals

## 71 PROPOSALS RECEIVED

- Most from southern and eastern United States
- 18 states represented



## DISTRIBUTION BY SOURCE

- 50 universities
- 6 industry
- 10 ORNL
  - Some with university collaborators
- 5 foreign
  - Germany, France, China

**71 total**

- 41 proposals selected for support, based on external *PRC* review
  - ~ 10 on proof-of-concept basis
- ***All active user research proposals now listed on CNMS web site***

## 2004 User-Initiated Nanoscience Research Program

### **Tailoring Electrical Properties: PANI/SWNT's Composites**

Principal Investigator: G. B. Blanchet (Material Science & Engineering, DuPont)

Collaborators: D. Geohegan (Oak Ridge National Laboratory)

### **Study of Nanomagnetism in Patterned Structures Using SEMPA**

Principal Investigator: J. Shi (Physics, University of Utah)

### **Direct Growth of Single Walled Carbon Nanotubes with Controlled Structures on Substrates for Device and Sensor Applications**

Principal Investigator: J. Liu (Dept. of Chemistry, Duke University)

### **Optical Manipulation of Carbon Nanotubes: Differential Diffusion Through a New Chirality-Dependent Electric Dipole Response**

Principal Investigator: W. R. Garrett (Physics & Astronomy, University of Tennessee)

### **Fabrication of Magnetic Nanowires and Nanowire Arrays Using Self-Assembling Polymer Templates**

Principal Investigator: M. G. Bakker (Dept. of Chemistry, University of Alabama)

Collaborators: D. Nikles (University of Alabama)

### **Hybrid Composites of Facially Amphiphilic Phenylene Ethynyls and Carbon Nanotubes**

Principal Investigator: G. N. Tew (Polymer Science & Engineering, University of Massachusetts)

### **Hydrogenation of Carbon Nanotubes: Water as a Hydrogen Source**

Principal Investigator: Y.-P. Sun (Chemistry, Clemson University)

### **Directed Assembly of Nanoparticles in Polymers**

Principal Investigator: T. Emrick (Polymer Science & Engineering, University of Massachusetts)

Collaborators: T. P. Russell (Polymer Science & Engineering, University of Massachusetts)

### **Scaffolding of Biosynthetic Enzyme Systems to Nanostructured Electrodes for Controlled Synthesis of Inorganic Materials**

Principal Investigator: D. Morse (Dept. of Molecular, Cellular & Develop. Biology, University of California)

Collaborators: M. L. Simpson (Oak Ridge National Laboratory)

T. McKnight (Oak Ridge National Laboratory)

### **Calculating Time Dependent Effects from a Modified Wang-Landau Density of States**

Principal Investigator: M. A. Novotny (Dept. of Physics & Astronomy, Mississippi State University)

### **Ferromagnetic Domain Structures at Epitaxial Metal/Semiconductor Interfaces for Spintronics**

Principal Investigator: H. H. Weitering (Physics & Astronomy, University of Tennessee)

Collaborators: L. C. Feldman (Vanderbilt University)

J. Shen (Oak Ridge National Laboratory)

### **High Production Rate Nanotube Synthesis Apparatus**

Principal Investigator: M. W. Smith (NASA Langley Research Center)

### **Development of a Nanoscale Solvothermal Processes Laboratory (NSPL) for CNMS**

Principal Investigator: D. J. Wesolowski (Chemical Sciences Division, Oak Ridge National Laboratory)

Collaborators: D. B. Beach (Chemical Sciences Division, Oak Ridge National Laboratory)

D. R. Cole (Chemical Sciences Division, Oak Ridge National Laboratory)

W. A. Hamilton (Chemical Sciences Division, Oak Ridge National Laboratory)

### **Nanostructured Composites as Tunable Dielectrics**

Principal Investigator: M. E. Rogers (Advanced Materials, Luna Innovations, Inc.)

Collaborators: B. Koene (Luna Innovations, Inc.)

P. Stevenson (Luna Innovations, Inc.)

M. Vercellino (Luna Innovations, Inc.)

User Activity (mid-February)

Number of new badges requested: 16

Users actively scheduling or working: > 18

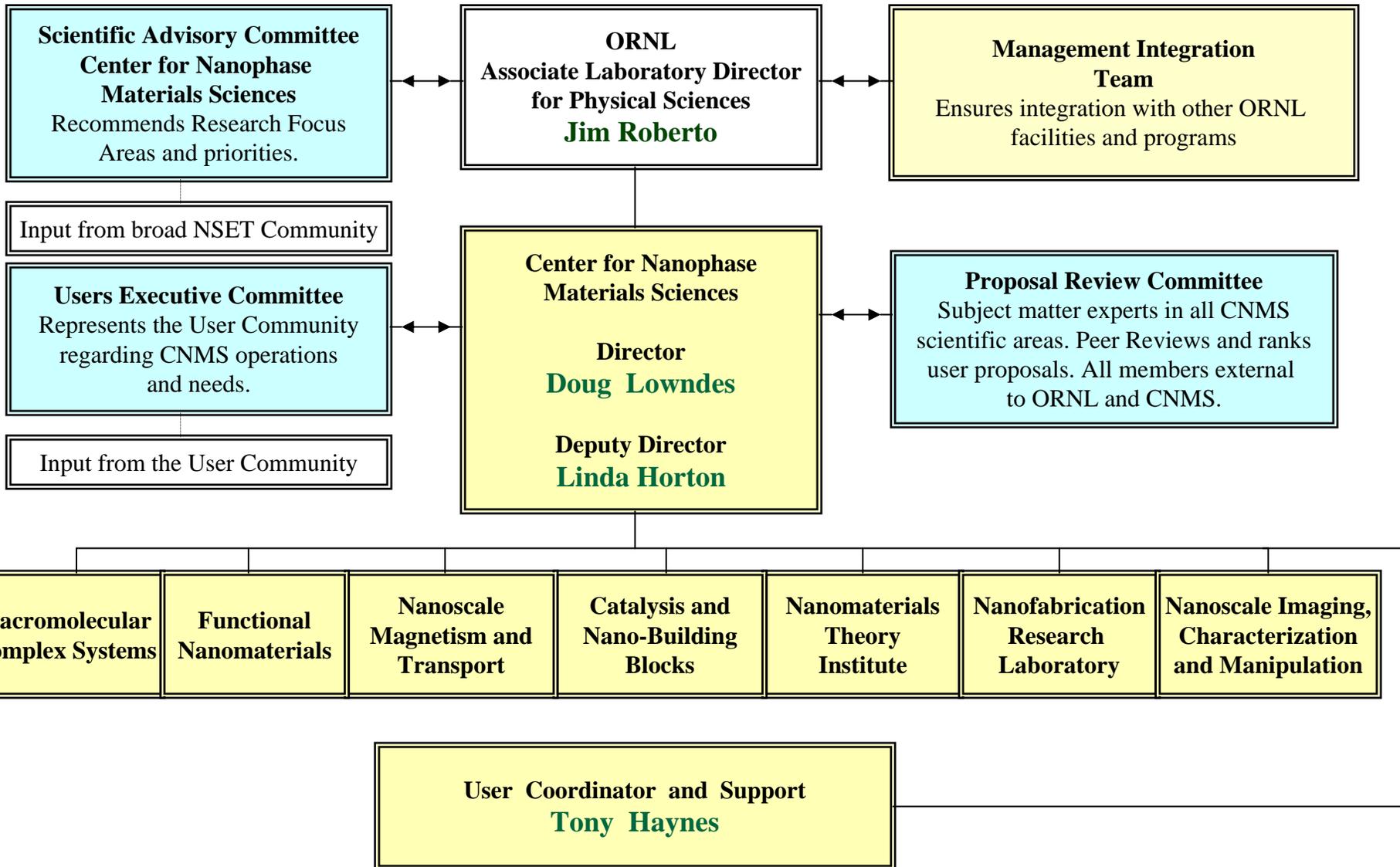
*The Team:*  
*SCIENTIFIC*  
*MANAGEMENT*  
*and*  
*ADVISORY GROUPS*



# CNMS Project Scientific Leadership Team

- **Doug Lowndes**                    **CNMS Scientific Director**
- **Linda Horton**                    **CNMS Project Director and Deputy Scientific Director**
- **Michelle Buchanan**            **CNMS Scientific Thrust Leader for Soft and Hybrid Materials**
- **Ward Plummer**                    **CNMS Scientific Thrust Leader for Complex Hard Materials**
- **Peter Cummings**                **CNMS Scientific Thrust Leader for Theory / Modeling / Simulation  
(Nanomaterials Theory Institute)**
- **Mike Simpson**                    **CNMS Scientific Thrust Leader for the Nanofabrication Research Lab**
- **David Joy**                         **CNMS Co-Leader for Imaging, Characterization and Manipulation**
- **Ian M. Anderson**                **CNMS Co-Leader for Imaging, Characterization and Manipulation**
- **Tony Haynes**                    **CNMS User Coordinator**
- **John Cooke**                    **CNMS Work Proposal Manager**
- **Jim Roberto**                    **ORNL Associate Laboratory Director for Physical Sciences**

# Governance and Operation of the CNMS





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