Center for Nanophase Materials Sciences

Oak Ridge National Laboratory

Mike Simonson
Group Leader, Nanoscale Structure and Dynamics

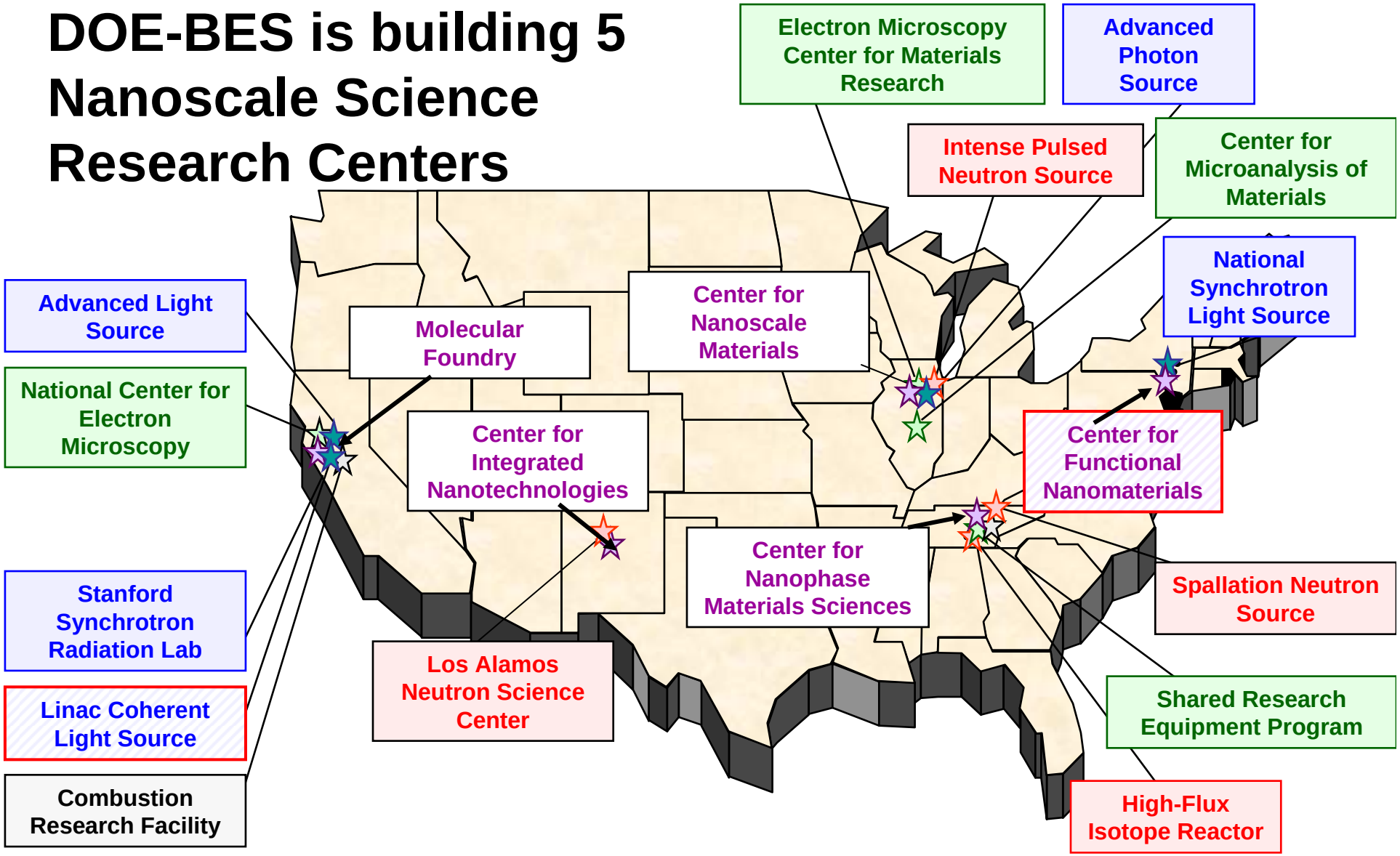
Office of Science
U.S. DEPARTMENT OF ENERGY

Center for Nanopha
Materials Science

DOE's Flagship for the NNI Initiative is the Nanoscale Science Research Centers

- **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
    - New user agreement for nanoscience for collaborative research
  - Proprietary access (internal peer review); full cost recovery required by DOE
- **Co-located with existing user facilities**
  - promote development of these tools for nanoscience and to provide extraordinary characterization and analysis capabilities
- **Conceived with broad input from university and industry user communities**
- **Provide specialized equipment and support staff not readily available to the research community**

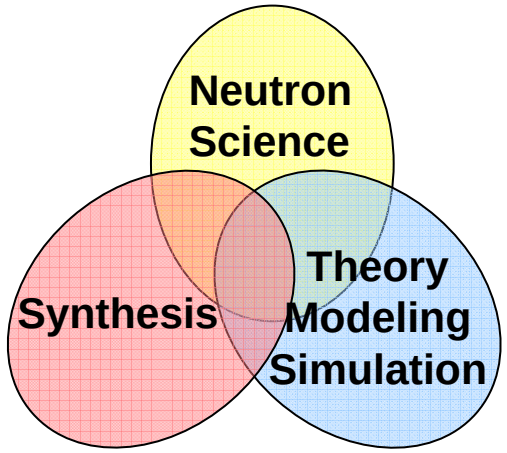
DOE-BES is building 5 Nanoscale Science Research Centers



Under construction

- *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*

CNMS Integrates Nanoscale Science with 3 Synergistic Research Needs



- Neutron Science
  - Opportunity for world leadership using unique capabilities of neutron scattering
- Synthesis Science
  - *Science-driven* synthesis: synthesis as enabler; evolution of synthesis via theory, modeling, and simulation
- Theory / Modeling / Simulation
  - Stimulate U.S. leadership in using theory, modeling and simulation to design new nanomaterials
  - Investigate new pathways for materials synthesis

Nanoscale Science Research Centers – Actual Photos

Center for Functional Nanomaterials (Brookhaven National Laboratory)

Center for Nanoscale Materials (Argonne National Laboratory)

Molecular Foundry (Lawrence Berkeley National Laboratory)

Center for Nanophase Materials Sciences (Oak Ridge National Laboratory)

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

CNMS Scientific Themes

Macromolecular Complex Systems

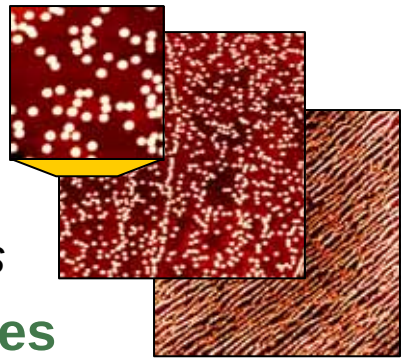
Synthetic (polymeric) and bio-inspired materials

Functional Nanomaterials

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

NanoPhysics: Magnetism, Transport, and Scanning Probes

Reduced and variable dimensionality; quantum transport



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

Catalysis and Nano-Building Blocks

Highly selective catalysts; nanoscale synthesis & organization

Nanomaterials Theory Institute: Theory, Modeling, Simulation

Grand challenges of "computational nanoscience"

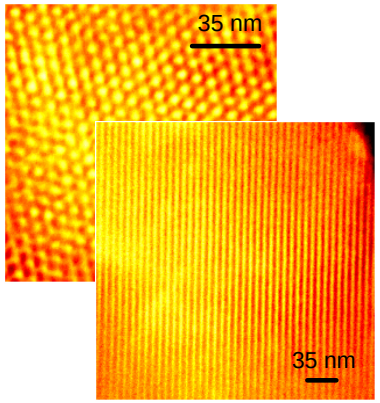
Nanofabrication Research Laboratory and Bio-Inspired Nanomaterials

Controlled synthesis & directed assembly; functional integration of "soft" and "hard" materials

Nanoscale Structure and Dynamics

Neutrons, Electron, and X-ray Characterization

Unique instruments to characterize and manipulate nanostructures; simultaneous imaging and environmental control



Ordered nanoporous silica synthesized using an organic template

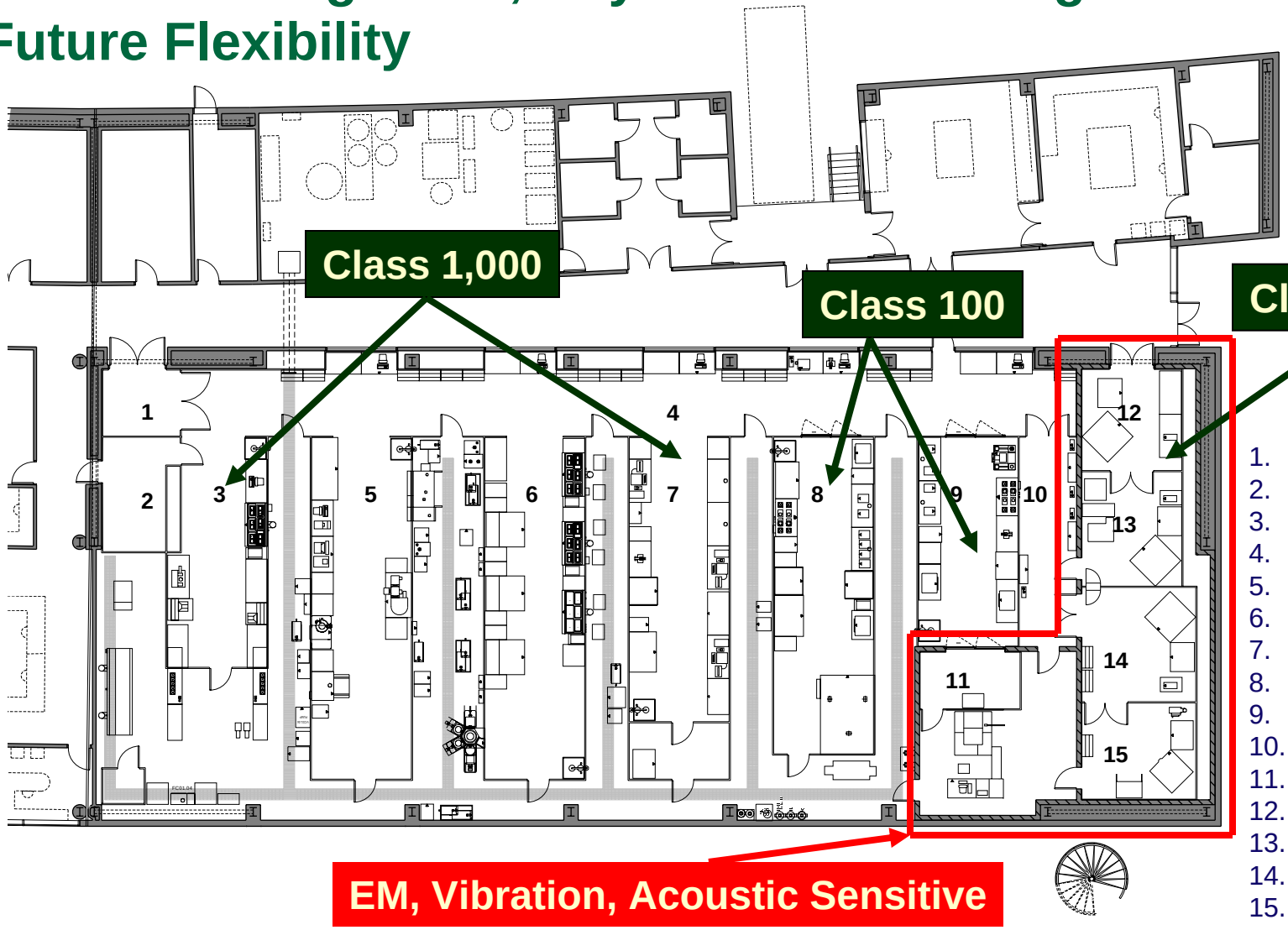
CNMS Project ($65M) was Completed in August 2006

- Construction of an approximately 80,000 sq ft facility
  - 32 laboratories, both wet and dry
  - Laboratories were designed based on best practices for safety for nanoscale research
  - Approx. 10,000 sq ft Cleanroom
  - Office space for up to 190 people (core staff, visiting senior staff, postdocs, graduate students, and users)
  - Common space to promote interactions among all staff and users (CNMS and SNS)
- Initial suite of equipment was included in the line item
  - List based on input from the future user community and the CNMS Advisory Committee
- Building opened for research in the Fall 2005 – Labs have come "on line" as equipment installation/lab set-up were completed
  - Laboratories have Research Safety Summaries, Laboratory Space Managers and Satellite Accumulation Areas and Hazardous Materials Inventory System control areas

Each floor has similar layout: labs across from offices; central interaction space and conference rooms; additional interaction space on upper floor bridges

Ground Floor: Clean room and sensitive instrumentation, including microscopes and scanning probes

CNMS Clean Room – Important for Directed Assembly and Materials Integration; Bay and Chase Design Allows for Future Flexibility

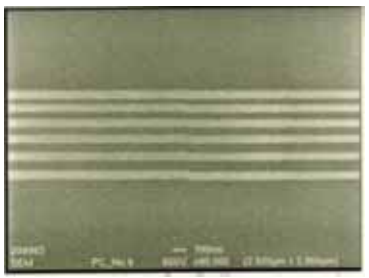


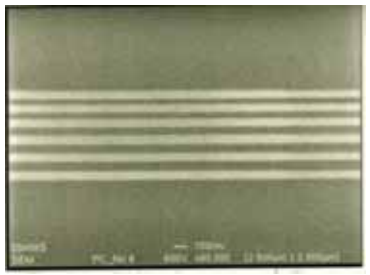
Nanofabrication Research Laboratory

Direct Write Electron Beam Lithography (DWEBL) System

sub-5 nm minimum spot
sub-20 nm minimum line width
1 mm field

- **Scientific Driver: Nanofabrication**
  - Patterns of arbitrary shape and size
  - Dimensions as small as 5 nm
  - Fabricated on any flat substrate sensitive to electron irradiation, or coated with e-beam resist
- **Capabilities:**
  - 100 keV thermal field emission source
  - Substrate handling capabilities for small pieces, membrane structures, and whole substrates up to 8–inch diameter
  - Laser interferometer with sub–nanometer resolution to permit highly accurate mechanical positioning
  - Substrate–height sensor to permit dynamic corrections to the beam focus



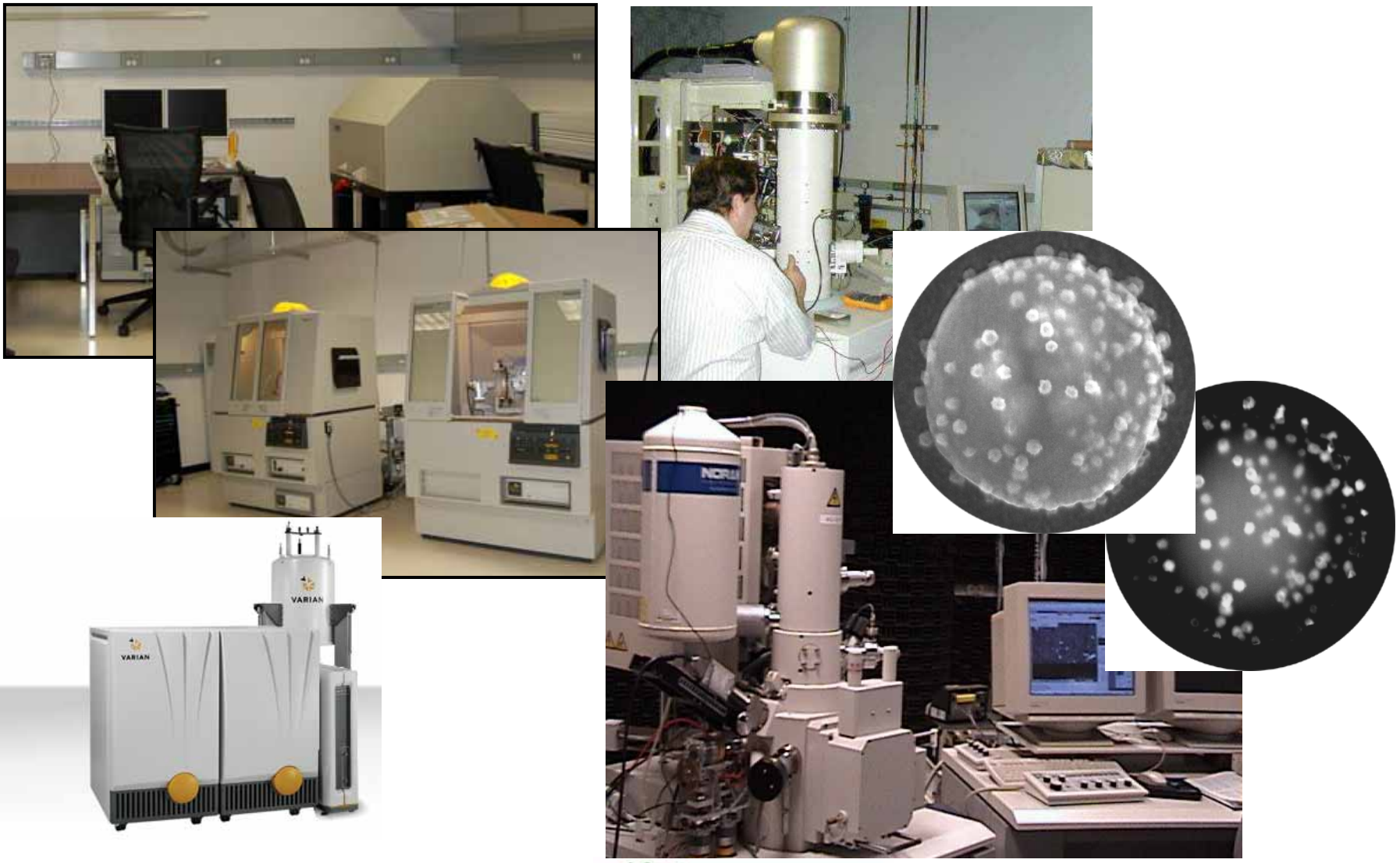


Focused Ion Beam (FIB) / Scanning Electron Microscope (SEM) (Dual–Beam System)

FEI Nova 600 Nanolab

- **Scientific Drivers**
  - Materials can be patterned using the FIB, or synthesized specimens can be thinned to highlight specific regions of interest, and immediately imaged with high resolution using the SEM
- **Capabilities**
  - Electron columns operate simultaneously to permit sample observation during FIB processing
  - Substrates may range from small pieces up to 50mm
  - Can be equipped with a TEM sample–preparation stage to permit simple interfacing between this instrument and other microscopy tools

Key Characterization Equipment



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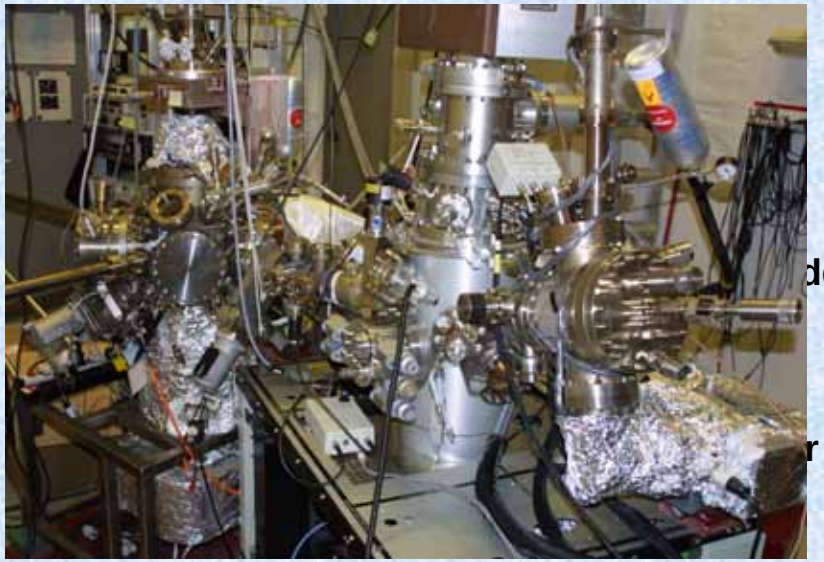
UHV Scanning Probes

Magnetic Characterization



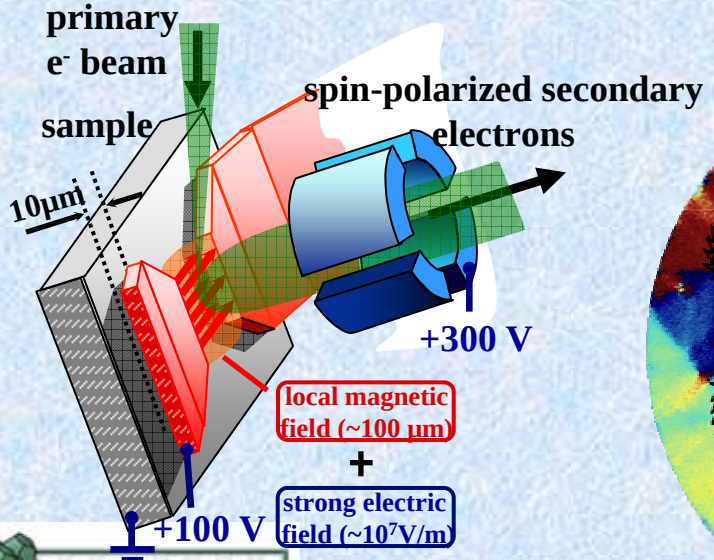
Scanning Electron Microscopy with Spin Polarization (SEMPA)

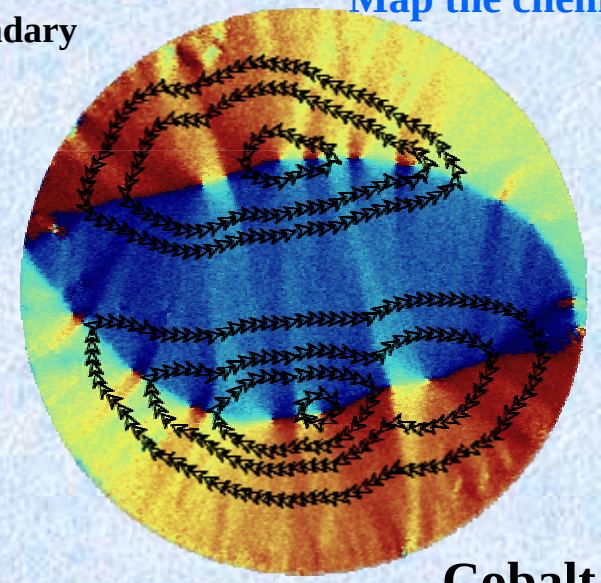
Scientific Drivers

- Direct imaging of magnetic domains
- Spin switching and spin dynamics in a field
- Correlation between chemical and magnetic inhomogeneities

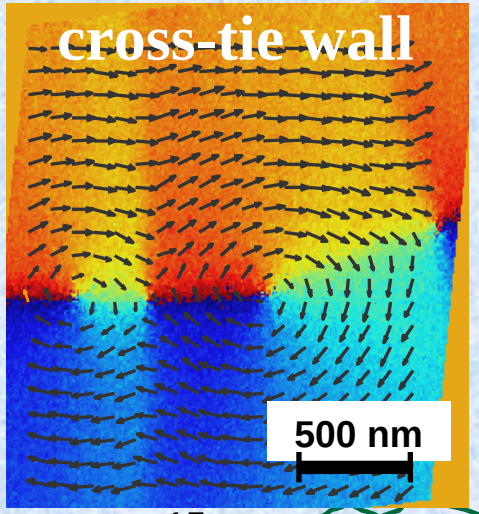
Capabilities

- Scanning Electron Microscopy
  - 300 mT in-plane magnetic field
  - 50-1000 K
- Scanning Auger Microscopy
  - Map the chemical composition





Cobalt film



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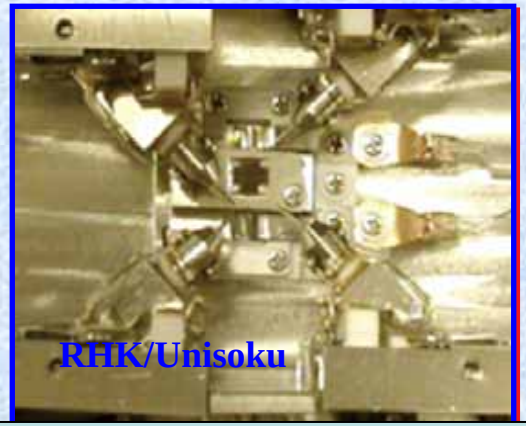
Transport and Manipulation

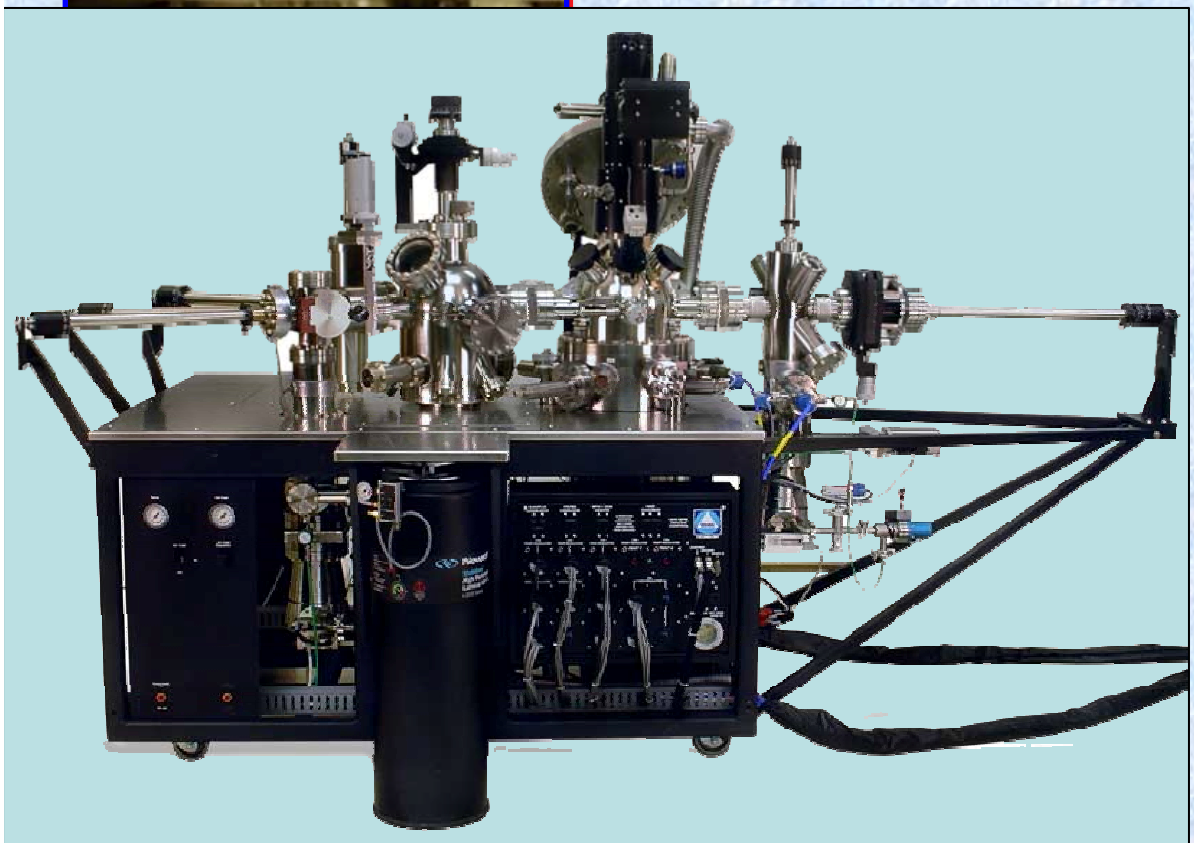
4 probe STM with SEM/SAM



Scientific Drivers

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

H. H. Weitering, UTenn



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

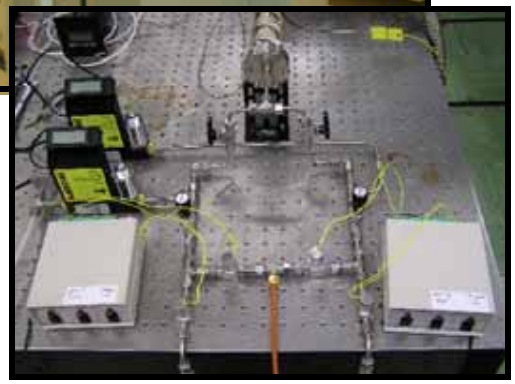
Sample preparation
- MBE growth
- Sputtering, cleaving

UT-BATTELLE

Functional Nanomaterials: Laser-based Synthesis and Characterization

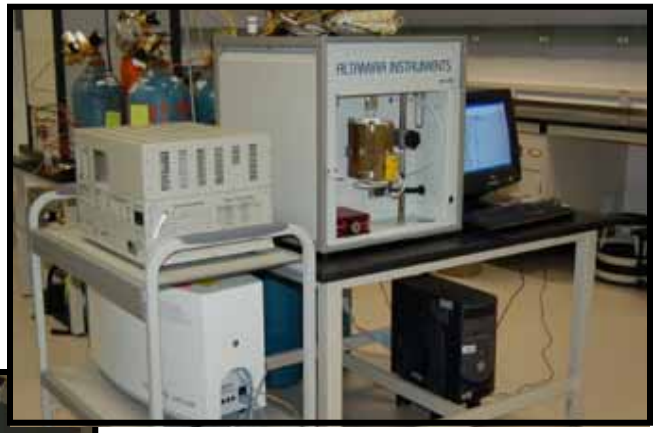
Nanocatalysis: Synthesis and Catalytic Characterization

Synthesis capabilities



Atomic layer deposition

Characterization resources: instruments for volumetric gas adsorption, temperature programmed reaction, and dynamic FTIR spectroscopy

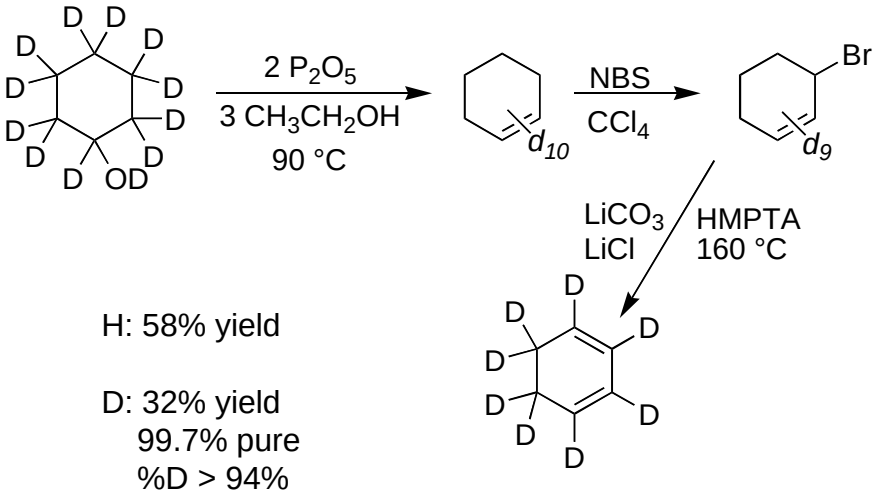


Macromolecular: Synthesis and Characterization

Unique Deuteration Capabilities Available

- **Two NSF Workshops (2003) recommended deuteration facilities convenient to the beam line**
- **CNMS provides synthetic staff trained in organic and polymer synthesis (Bonnesen, Hong) for preparation of well defined deuterated small molecules, monomers, and polymers**

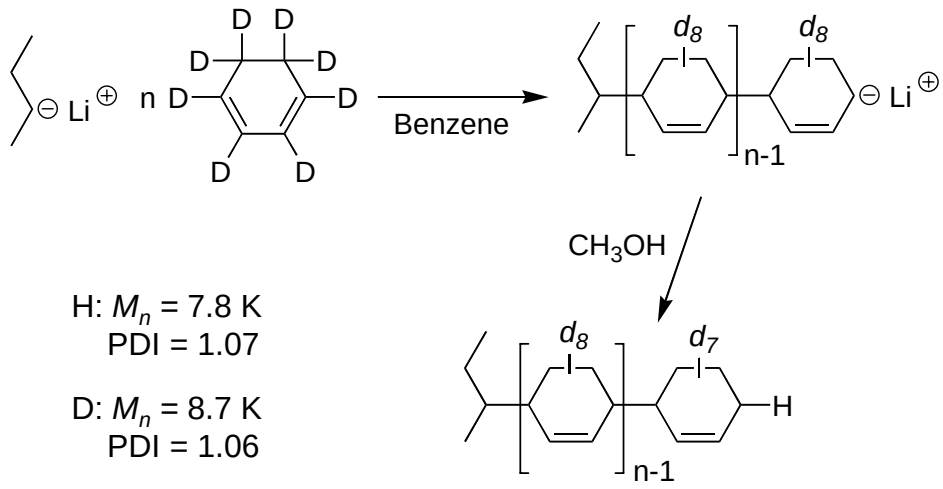
Synthesis of deuterated monomer



H: 58% yield

D: 32% yield
99.7% pure
%D > 94%

Synthesis of deuterated polymer



H: M_n = 7.8 K
PDI = 1.07

D: M_n = 8.7 K
PDI = 1.06

- ***Results of SANS studies using CNMS polymers published in Macromolecules (2006)***
- ***First neutron reflectivity experiments at SNS performed recently using CNMS polymers***

Computation and Visualization

- CNMS 80-dual processor node Beowulf Cluster
  - 4 GB RAM per node;
  - gigabit interconnects;
  - 1.1 teraflop
- Photon visualization cluster
  - 17-nodes, each with
    - 4x Opteron (dual dual-core)
    - 4GB RAM
    - NVIDIA 7800 GT graphics cards
    - 10TB total storage
- 30 million pixel display wall
  - 4×4 LCD panels
  - Each at 1600x1200

CNMS has Synergistic Relationships with other ORNL Capabilities

- Neutron Scattering
  - **High Flux Isotope Reactor Center for Neutron Scattering**
  - **Spallation Neutron Source**
  - **Joint Institute for Neutron Scattering (future)**
- Computational Resources
  - **National Center for Computational Sciences (OASCR)**
- Electron Microscopy
  - **Shared Research Equipment (SHaRE) User Program**
  - **High Temperature Materials Laboratory (EERE)**
- Shared and Affiliated resources with ORNL's BES Materials and Chemical Sciences Programs
- Integrated Proposal under development for ORNL's BES User Facilities
- **Key component of ORNL capabilities for nanoapplications/nanomanufacturing**

Neutron User Facilities at ORNL

Spallation Neutron Source:

The world's most intense neutron source

Suite of leading-edge instruments

High Flux Isotope Reactor:

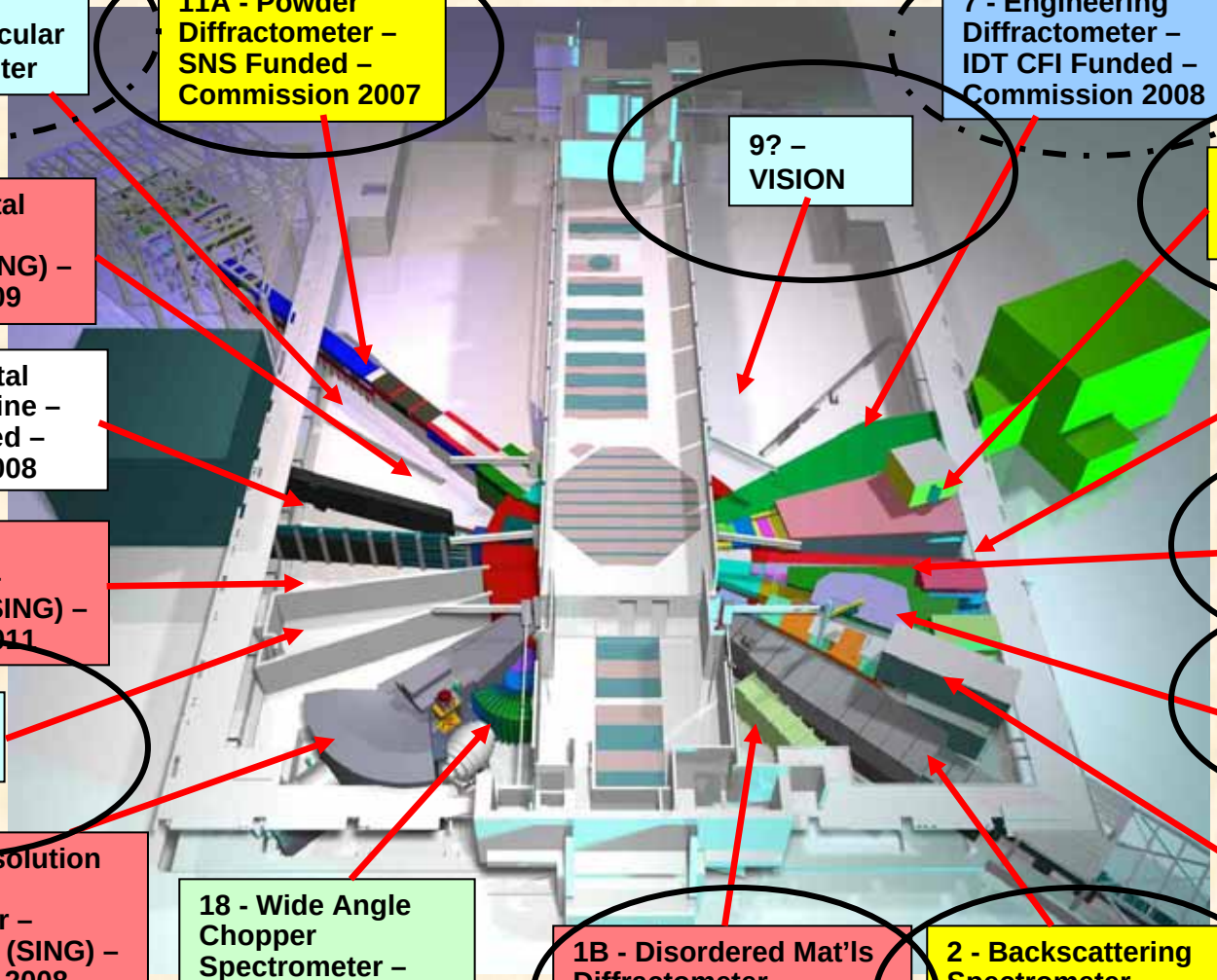
High-power (~85 MW) reactor source

Cold neutrons for nanoscale structures, including biology

SNS Approved Instruments

Nanoscience Research

SNS
UNIV
CANADA
SING
NUC-PH
Other



11B – Macromolecular diffractometer

11A - Powder Diffractometer – SNS Funded – Commission 2007

7 - Engineering Diffractometer – IDT CFI Funded – Commission 2008

9? – VISION

6 - SANS – SNS Funded – Commission 2007

12 - Single Crystal Diffractometer – DOE Funded (SING) – Commission 2009

5 - Cold Neutron Chopper Spectrometer – IDT DOE Funded – Commission 2007

13 - Fundamental Physics Beamline – IDT DOE Funded – Commission 2008

4B - Liquids Reflectometer – SNS Funded – Commission 2006

14B - Hybrid Spectrometer – DOE Funded (SING) – Commission 2011

4A - Magnetism Reflectometer – SNS Funded – Commission 2006

15 – Spin Echo

3 - High Pressure Diffractometer – DOE Funded (SING) – Commission 2008

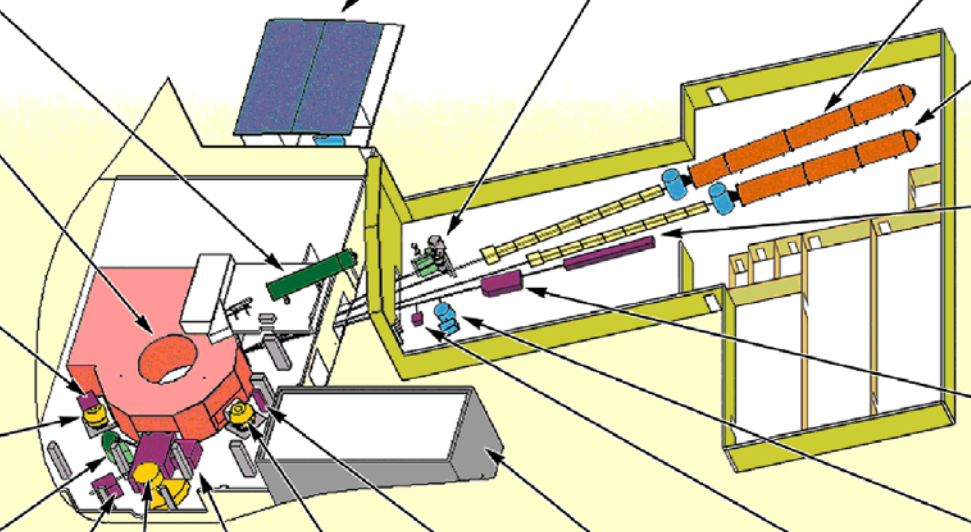
17 - High Resolution Chopper Spectrometer – DOE Funded (SING) – Commission 2008

18 - Wide Angle Chopper Spectrometer – IDT DOE Funded – Commission 2007

1B - Disordered Mat'ls Diffractometer – DOE Funded (SING) – Commission 2010

2 - Backscattering Spectrometer – SNS Funded – Commission 2006

HFIR Center for Neutron Scattering



12-m SANS

Cold Neutron Source

Cold Source Refrigeration Unit

CG-1 STAR
Highly correlated electronic systems, quantum magnetism, molecular and nanocluster magnetic systems

CG-2 SANS
Polymer blends, flux lattices in high-Tc materials, soft materials processing and structure

CG-3 SANS
Biomaterials, pharmaceuticals, polymers

CG-4D Nuclear Physics
Fundamental nuclear physics

CG-4C U.S./Japan Cold Triple-Axis
Highly correlated electronic systems, quantum magnetism, molecular and nanocluster magnetic systems, superconductivity

CG-4B Reflectometer
Thin films, interfaces and surfaces, liquid crystals, biomembranes, surfactants

CG-4A USANS
Colloids, polymers, magnetism

HB-1A Triple-Axis Spectrometer
Low-energy excitations, magnetism, structural transitions

HB-1 Triple-Axis Spectrometer
Polarized neutron studies of magnetic materials, low-energy excitations, structural transitions

HB-2A Powder Diffractometer
Structural studies, magnetic structures, texture and phase analysis

HB-2C WAND
Diffuse-scattering studies of single crystals and time-resolved phase transitions

HB-2D Triple-Axis Spectrometer
Medium-energy excitations in small or weakly scattering samples

HB-2B Residual Stress
Stress and strain measurements in engineering materials

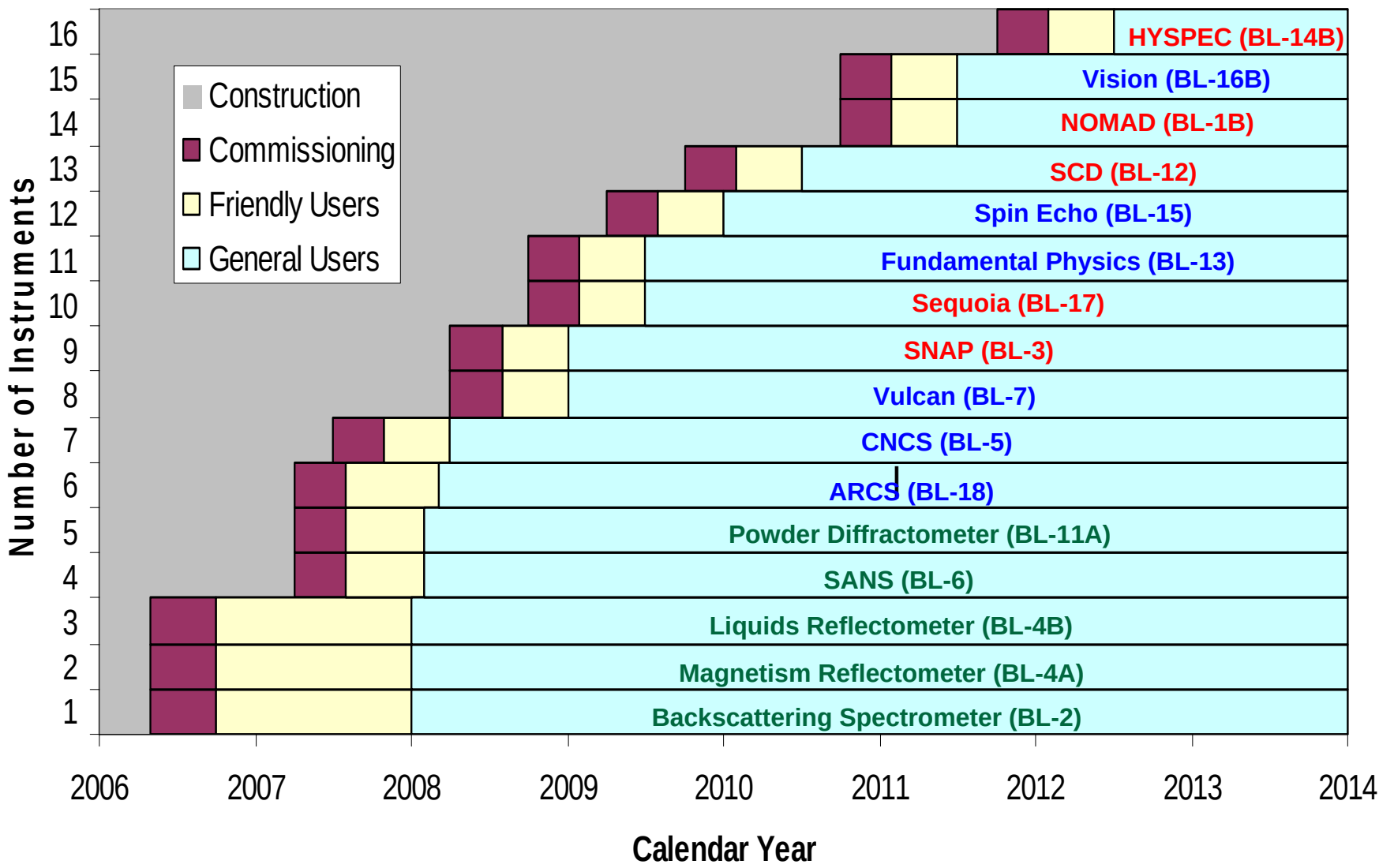
HB-3 Triple-Axis Spectrometer
Medium- and high-resolution inelastic scattering at thermal energies

HB-3A Four Circle Diffractometer
Small unit-cell crystal structural studies, particularly H-bonding

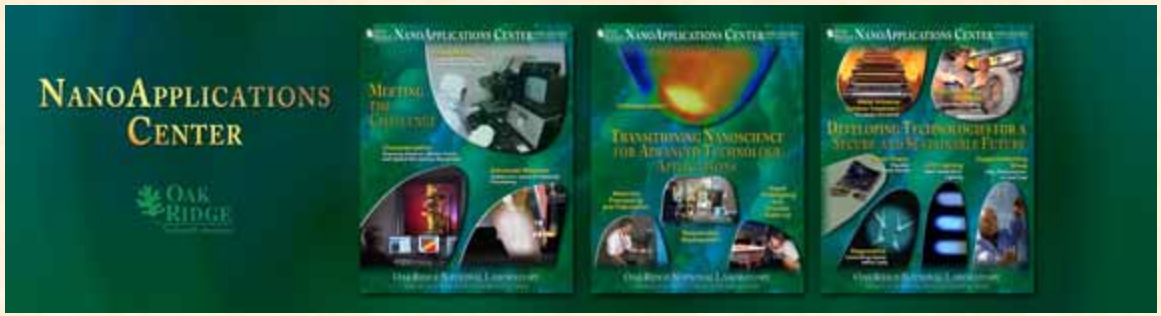
Technical Support and Cryogenic Laboratory

03-02837/dgc

Schedule for Funded Instruments

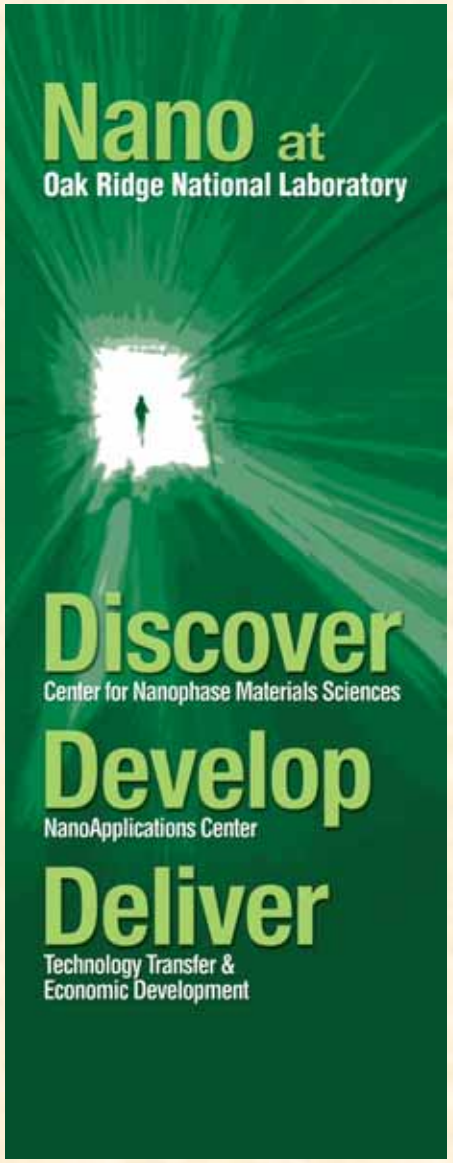


CNMS Contributions to Nanoscale Applications



- **CNMS and basic research programs (DOE SC)**
- **Applied research and development (DOE EERE, FE, NE; DoD, NIH, WFO)**
- **Technology transfer and economic development (Capitalization and commercialization)**

nano.ornl.gov



CNMS User Access Policy

- **Open access based on scientific and technical quality**
  - Well-defined access policies and procedures posted on Web site
- **Flexibility to accommodate spectrum of user modes and needs**
  - General Users: Access to existing tools / support / collaboration
  - Partner Users: Enhance CNMS capabilities
  - Proprietary Users: Access at full cost recovery for sensitive research
- **External, peer-review required for general and partner users**
  - Simple process for both proposers and reviewers
  - Proposal Review Committee entirely external, structured with expertise to review proposals of multidisciplinary collaborations / teams
  - Review guidelines and process described on CNMS Web site
- **Internal ORNL peer-review for proprietary research**
  - Process parallels that for general/partner users
- **Free of charge**:
  - No fees if results published in open, peer-reviewed scientific journals
  - Full cost recovery charged for proprietary access
- **Scheduling and speed**
  - Well-documented scheduling process, once proposal is accepted
  - Web-published turn-around policy for proposal review & experimental access

CNMS SCIENTIFIC THEMES (GROUPS)

Macromolecular Complex Systems

Phil Britt
Jimmy Mays

Functional Nanomaterials

Hans Christen
Dave Geohegan

Nanophysics: Magnetism, Transport and Scanning Probes

Ward Plummer
John Wendelken
Art Baddorf
Jian Shen

Catalysis and Nano-Building Blocks

Steve Overbury
Sheng Dai
Viviane Schwartz

Bio-Inspired Nanomaterials

Mike Simpson
Scott Retterer

Nanomaterials Theory Institute: Theory, Modeling, Simulation

Peter Cummings
Thomas Schulthess
Malcolm Stocks (Sr. Advisor)

Nanoscale Structure and Dynamics: Neutrons, Electrons and X-rays

Mike Simonson
David Joy
Takeshi Egami (Sr. Advisor)

Nanofabrication Research Laboratory (Cleanroom)

Mike Simpson

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