The Center for Functional Nanomaterials: A User-oriented Research Center

CFN Update

Emilio Mendez

CFN/NSLS Users' Meeting
May 20, 2008

A User-oriented Nanoscience Research Center

Goal: To become a world-class hub of nanoscience research

Staff-driven Research	User-driven Research
Energy-related • interf. & catalysis • photovoltaics • soft & bionanomat.	• training • expert support • collaborations
FACILITIES Technique and Tool Development	

high concentration of state-of-the-art nanoscience tools + top scientists in nanoscience = UNIQUE FACILITY

Outline

- The CFN Project
- Scientific Mission
- Major Facilities
- User Mission
- Staff
- Budget
- Engaging the Science Community
- Future Directions
- Summary

The CFN Project at a Glance

2008

2007

2006

2005

← Operations Review (5/7-9/08)

← READY for OPERATIONS

CD-4b Passed (3/21/08)

← Last equipment received

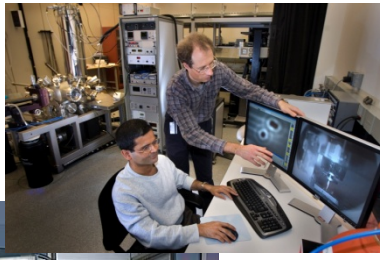
Green Building: Silver

← Ribbon-cutting (05/21/07)

← First equipment received

← CD-4a Passed (03/13/07)

← Construction Starts (CD-3)



Scientific Mission

CFN Staff Research

Scientific Themes

- electronic materials / photovoltaics
- interface science & catalysis
- soft & biomaterials

Cross-cutting Programs

- electron microscopy
- theory and computation

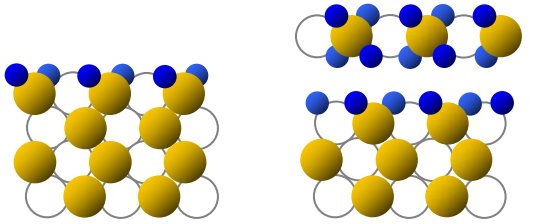
– are relevant to the energy challenge
– address important scientific questions
– are synergistic with BNL's core programs
– take advantage of BNL facilities

Scientific Program

Interface Science / Catalysis

Elucidate microscopic processes and their dynamic nature under reaction conditions

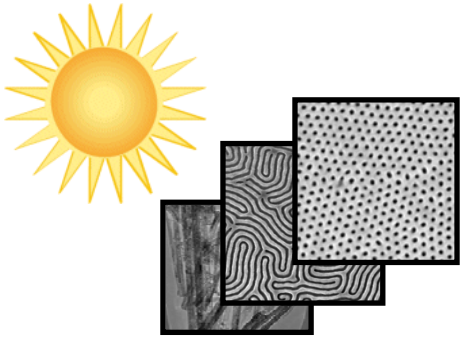
- What is the active phase?
- What determines size-dependent reactivity?
- How is a catalyst affected by its support?



Electronic Nanomaterials

Use nanostructured materials to understand and improve critical steps to photovoltaic/photochemical energy conversion

- How can physical processes in organic photovoltaic devices be optimized by using nanostructured materials?
- How to better capture the solar spectrum?

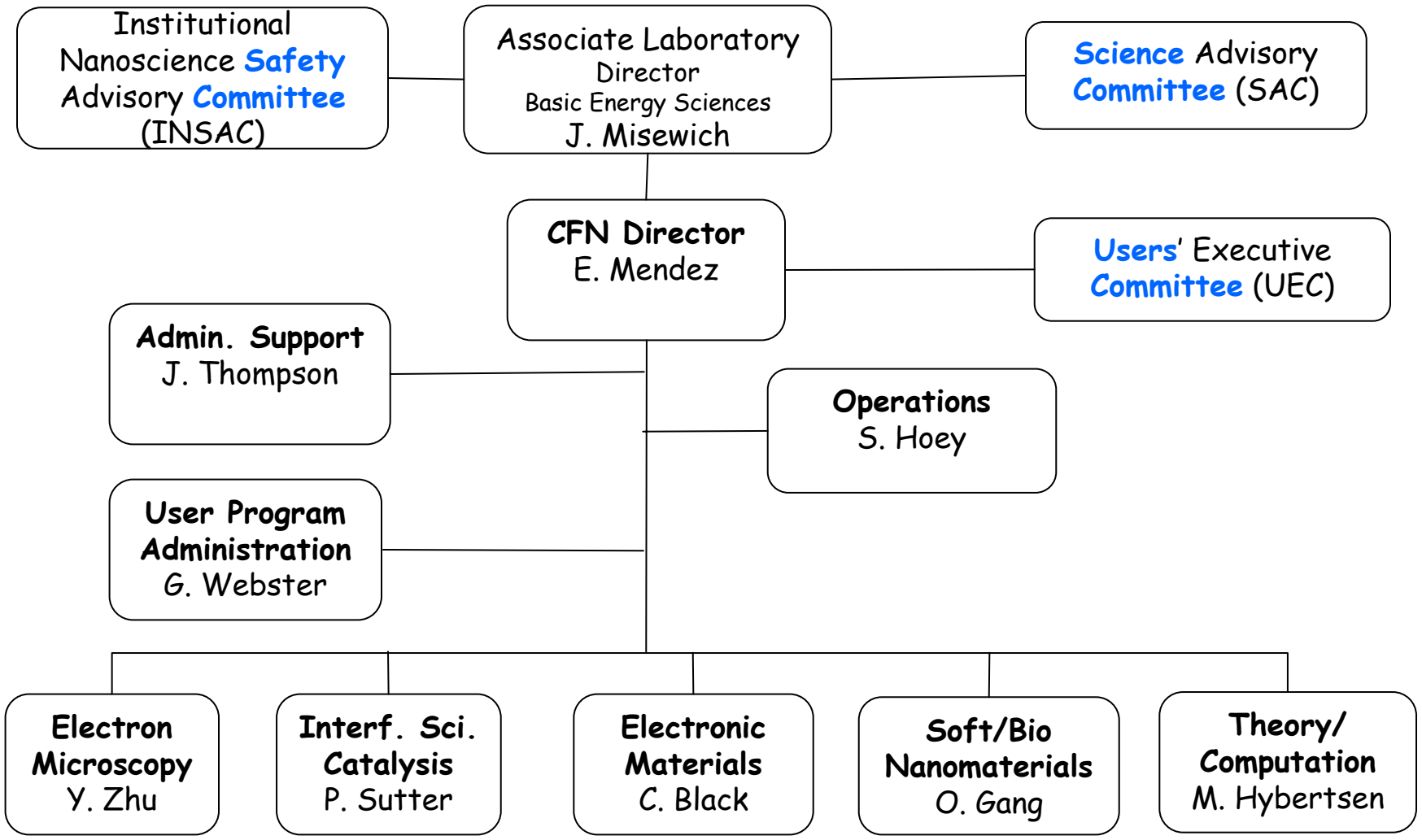


Soft and Bio Nanomaterials

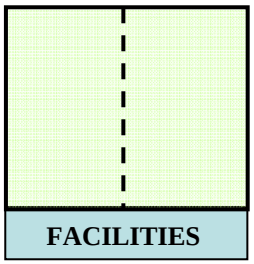
Develop self-assembly methods using hybrid nanoscale systems and explore their functionalities for energy conversion

- How to assemble nano-objects in large well-ordered (2D, 3D) structures?
- How to use hybrid structures for energy/charge transfer?

Organization Chart



Facilities

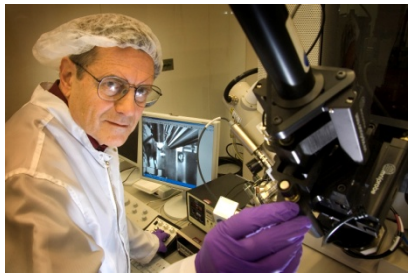


Characteristics

- **State-of-the-art** nanoscience-research equipment
- **Comprehensive** suite of tools
- **Balance** between equipment requiring straightforward training with equipment demanding extensive training and expert knowledge

Materials Synthesis
Nanofabrication
Trans. Elect. Microscopy
Proximal Probes
Ultrafast Spectroscopy
NSLS Beamline(s)
Computer Cluster

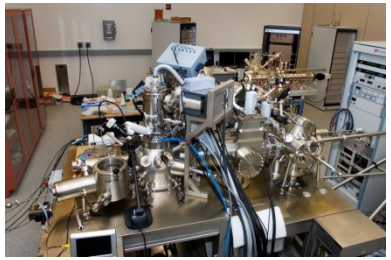
TEM



Dual-beam FIB

SEM

CVD Reactor



Low-T Nanoprobe

Facility Highlights

Electron Microscopy

from general to specialized use

"workhorse" TEM

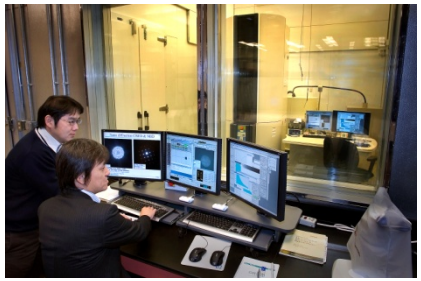
high resolution and ease of use

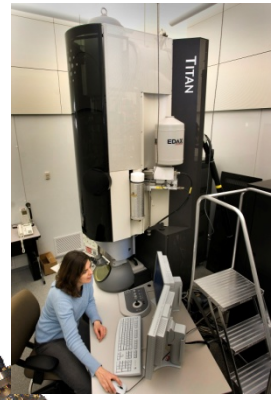
aberration-corrected STEM

high resolution and chem. analysis

aberration-corrected e-TEM

hi. res. and moderate-pressure capability





Nanofabrication

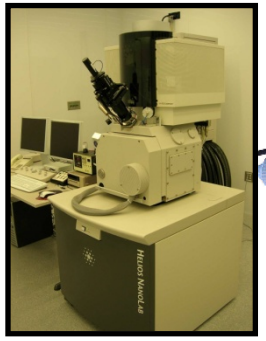
comprehensive suite of tools

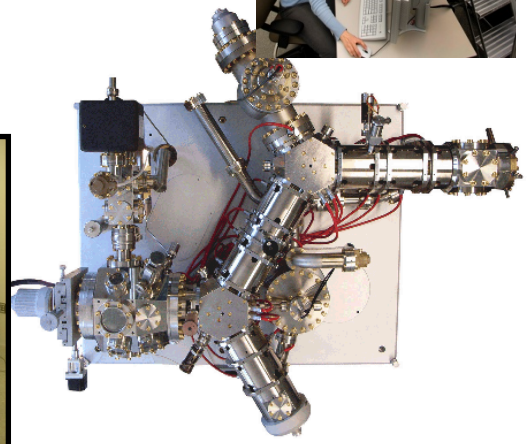
dual-beam system

electron microscopy and lithography

nanoimprinter

large-area patterning of 20 nm features





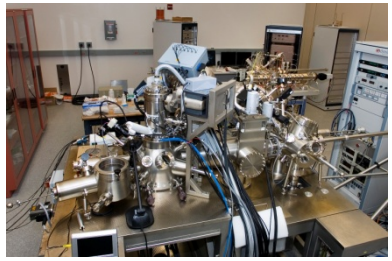
Proximal Probes

low-energy electron microscopy

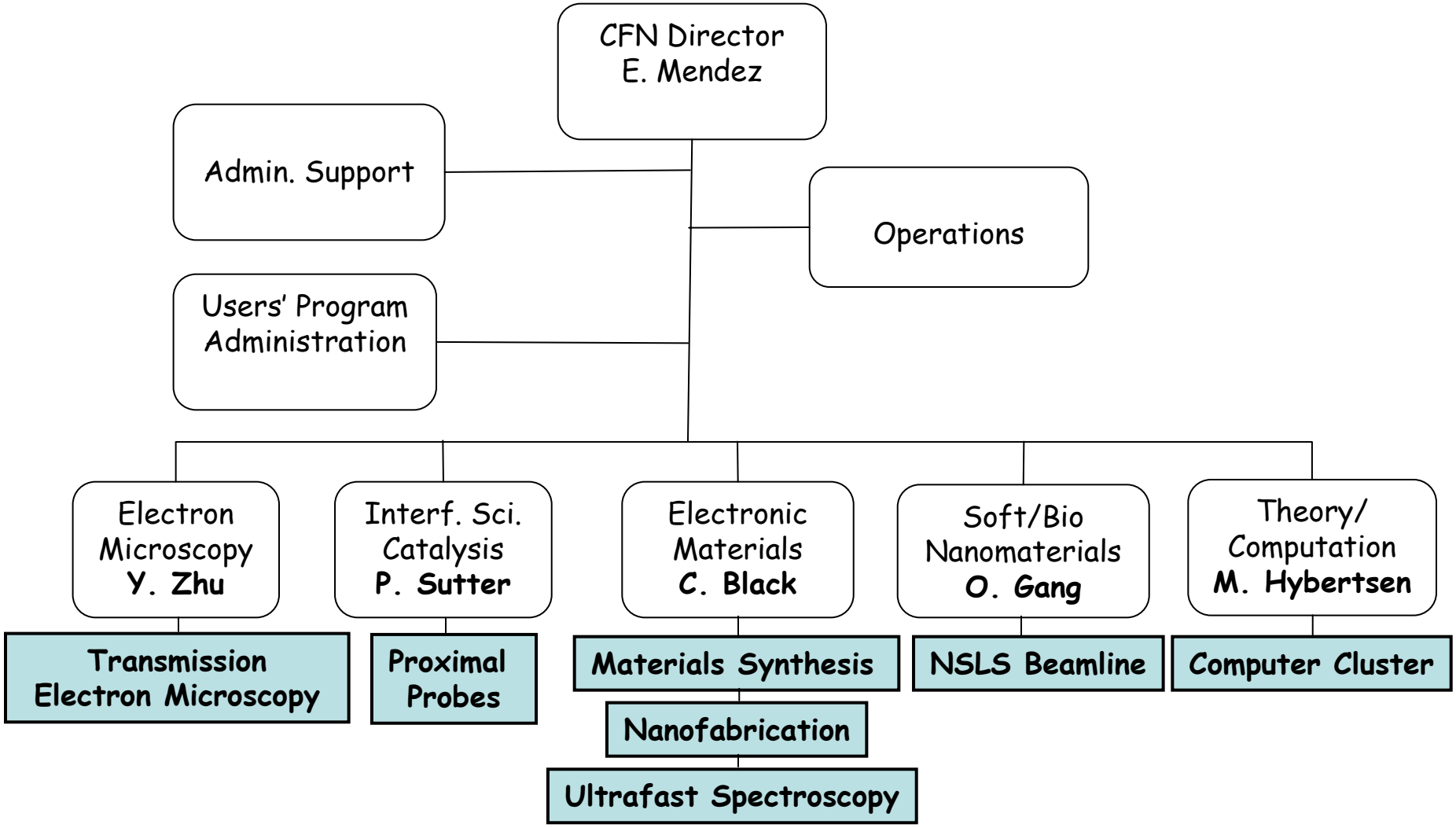
in-situ study of dynamic surface processes

4-point nanoprobe

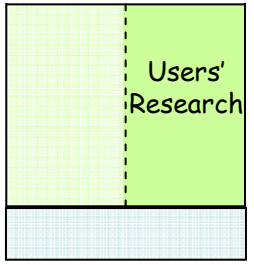
prep. and in-situ study of single nanostructures



Organization Chart and Facilities Management



User Program



CFN Goal: To become a resource for the nanoscience community of the Northeastern US and beyond

Characteristics

- **Free access to facilities and expertise for non-proprietary research**
  academia, companies, national laboratories
- **Based on peer-reviewed proposals**
- **Three proposal cycles per year**
  regular access, rapid access
- **Full-cost recovery for proprietary work**
- **Suitable to a wide range of users' needs**
  from straightforward, one-time measurements
  to complex, extended experiments

Distribution of User Projects

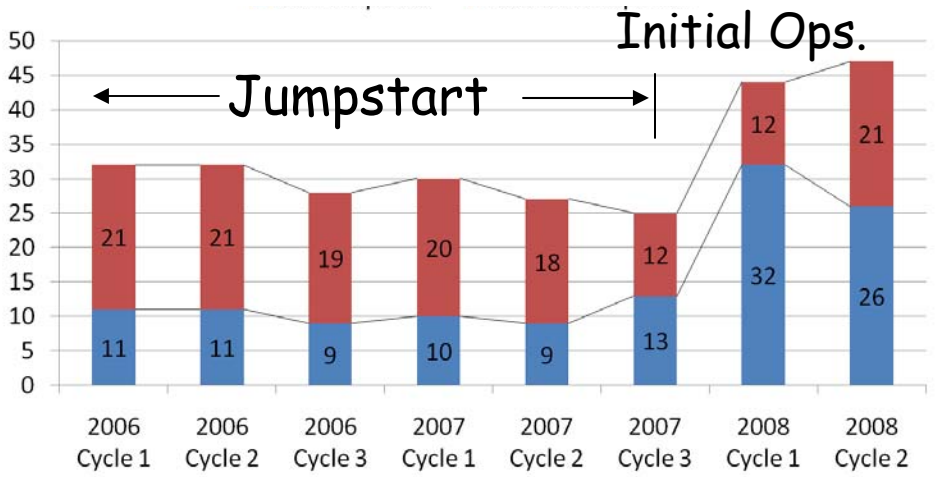
266 proposals received and reviewed (4/30/08)

Facility	# of Proposals Reviewed	# of Proposals Approved	Approval Rate
Electron Microscopy	90	66	73%
Nanofabrication	72	70	96%
Materials Synthesis	39	38	97%
NSLS	32	23	72%
Proximal Probes	20	17	85%
Theory &Computation	8	8	100%
Optical Spectroscopy	5	5	100%
TOTAL	266	227	85%

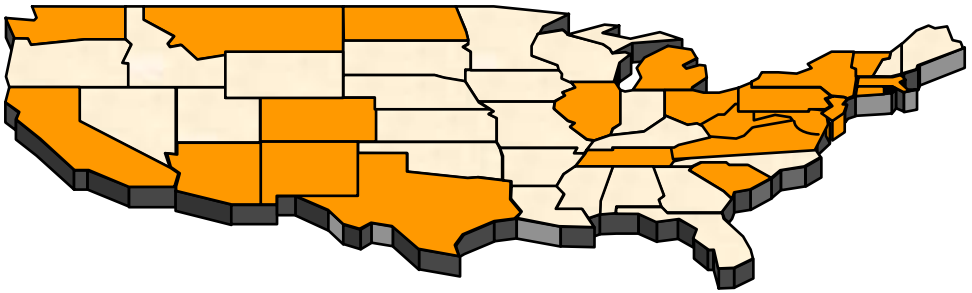
Evolution of User Proposals

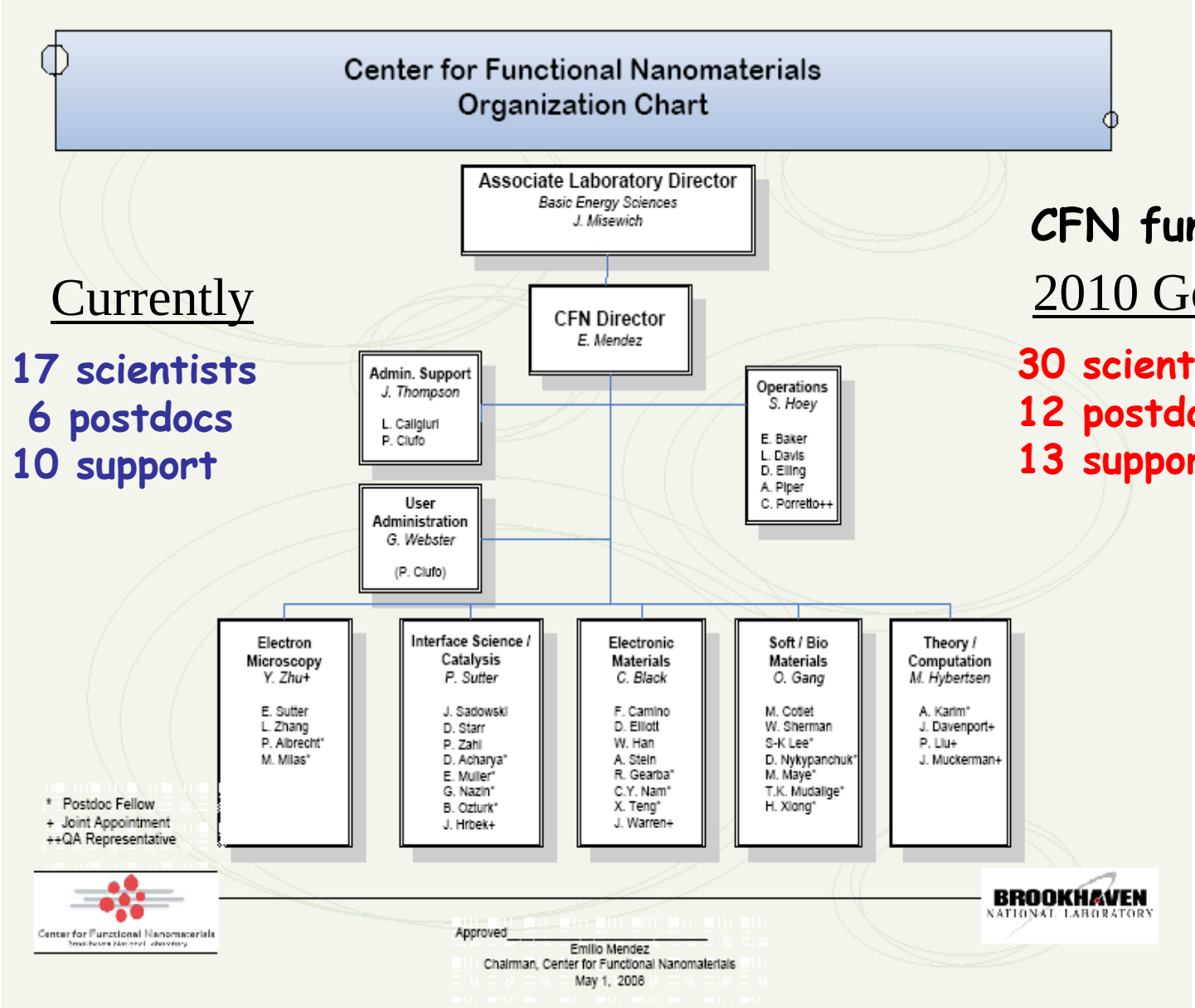
Continuations

New Proposals



266 proposals received so far





Currently

17 scientists
6 postdocs
10 support

CFN funded

2010 Goal

30 scientific staff
12 postdocs
13 support

CFN New Staff

(since July, 07)

- Scientific Staff
  - **David Starr**, Interface Science and Catalysis
  - **Fernando Camino**, Nanofabrication
  - **Mircea Cotlet**, Soft & Bio Nanomaterials
  - **Jurek Sadowski**, Interface Science and Catalysis
  - **Dong Su** (6/08), Electron Microscopy
  - **Qin Wu** (6/08), Theory and Computation
- Technical Staff
  - **Don Elliot**, Nanofabrication
  - **Edward Baker**, General Support
  - **David Elling**, General Support

CFN Staffing Plan

Immediate

- Scientific Staff
  - Electron microscopy
  - Ultrafast spectroscopy
  - NSLS endstation
  - Organic materials synthesis
  - Materials processing
  - Soft-matter theory
- Technical Staff
  - Materials preparation
- Support
  - Information technology
  - User program

Engaging the Scientific Community

- Workshops
  - Bio-inspired Assembly (Users' Meeting '07)
  - Electrical Nanoprobes (Users' Meeting '08)
  - Electron Microscopy (11/07)
  - Nanoscience for High-Tech Industry (11/07)

- Visits / Lectures
  - Stony Brook, U. Conn., Cornell, Delaware, Penn, Columbia, IBM

- Participation in Stony Brook U.
  - Advanced Energy Center

- Focused Collaborations with Stony Brook, Columbia, Cornell
  - catalysis, photovoltaics

Future Instruments & Techniques

Goal: **to be at the cutting edge** as a user-oriented nanoscience research center

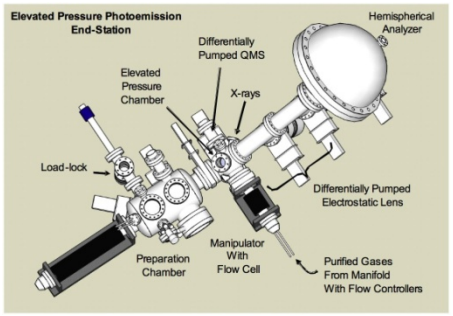
Time horizon: 1 – 5 years
Some, contingent on new resources

High-performance e-beam lithography system

Energy Filter for 'Workhorse' TEM

Aberration corrected LEEM & PEEM

High Pressure Photoemission at NSLS



Reactor STM and high-p STM with IR

New Resources for Users

Web-based Access for Targeted Simulations

- Applications that address recurrent needs
  - DFT calculations for certain class of materials
  - Tool to interpret TEM, STM, x-ray images and data
- Web-enabled data entry, execution and display of results
- Accessible remotely to badged users

Remote Training

- Web-based videos with operational procedures
- Online operation manuals/instructions

Remote Operation of Instruments

Electronic Notebooks for Instruments

Electronic Management of User Proposals

Monographic Courses on Techniques and Instrumentation

Summary

- **User**-oriented **research** center striving to become world-class resource
- Uniqueness from **synergy** between advanced equipment and expert staff
- In full operations since March 21, 2008
- Thriving **Research Program** on **energy**-related materials & processes
- Blooming high-quality **User Program**
- **Staffing plan** well under way
- Planned **facilities enhancement**
- Enthusiastic staff **ready to train/help/work with users**

What a Difference One Year Makes

May, 2007

May, 2008

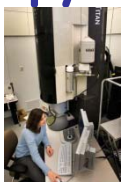
What a Difference One Year Makes

May 18, 2007

May 18, 2008

Back-up Slides

Distribution of Facilities

FACILITIES \ Groups	Electronic Materials	Interface Catalysis	Soft Biomat.	Electron Microscopy	Theory Computat.
Synthesis 	■	■	■		
Nanofabrication 	■				
Proximal Probes 		■	■		
Optical Spectroscopy	■	■	■		
NSLS Beamline(s) 		■	■		
Trans. Elect. Microsc.				■	
Computer Cluster					■

Acquisition of Capital Equipment

High-end e-beam lithography system

System for determination of nanomaterial surface area and porosity

Processing station for air-sensitive organic semiconductor materials

Variable-temperature Hall-effect system

Thin-film growth by atomic layer deposition

Reactor STM

Aberration-corrected LEEM

Single-crystal adsorption calorimetry

Pulsed optical system for for single-photon counting and single-molec.

Coherent anti-Stokes Raman microscope

Near-field optical imaging and tip-induced Raman imaging

Post-column energy filter for JEOL 2100F TEM

Monochromator for FEI e-TEM

Major Instrumentation

High-performance e-beam lithography system (100 keV)

(e.g., JEOL JBX-6300FS)

<u>Characteristics of the instrument</u>

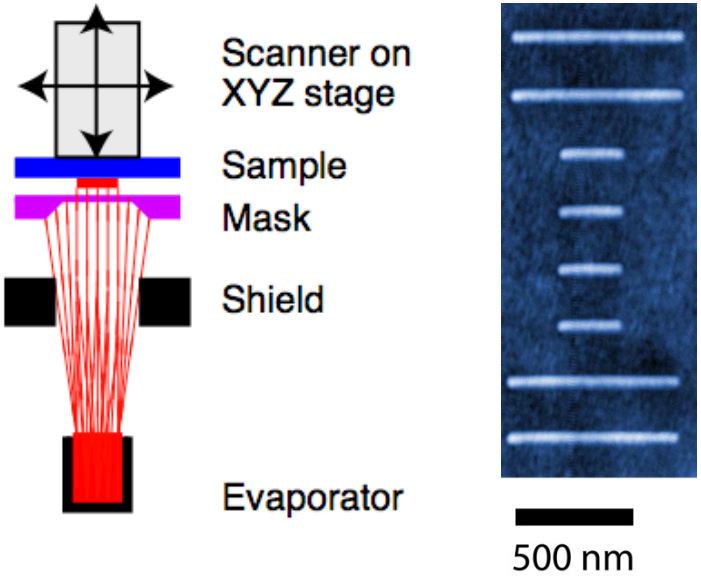
- high resolution (sub-20 nm dimensions)
- large-area patterning
- high placement and stitching accuracy

- will complete suite of nanofabrication tools
- will be located in specially designed space in clean-room area
- will support CFN's User Program as well as internal-research program

Major Instrumentation

Nano-Stencil for In-situ Patterning

- Combination with existing nanoprobe instrument
- Direct nanopatterning in UHV by evaporation through shadow mask (line width < 30 nm)
- Broad range of materials (anything evaporable in UHV) -'sensitive/soft' materials



Target user group: broad range of in-situ studies on functional nanomaterials:
transport, magnetic properties, catalysis

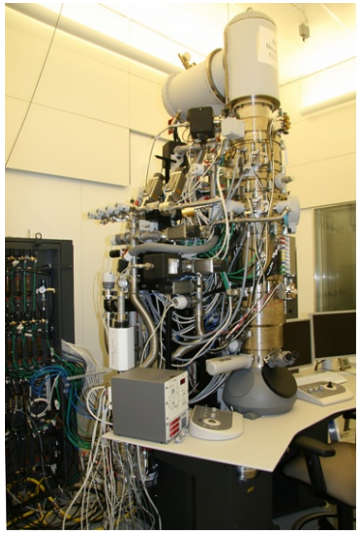
Major Upgrades

Energy Filter for 'Workhorse' TEM

- Will allow users to perform EELS for chemical identification.

Monochromator for e-TEM

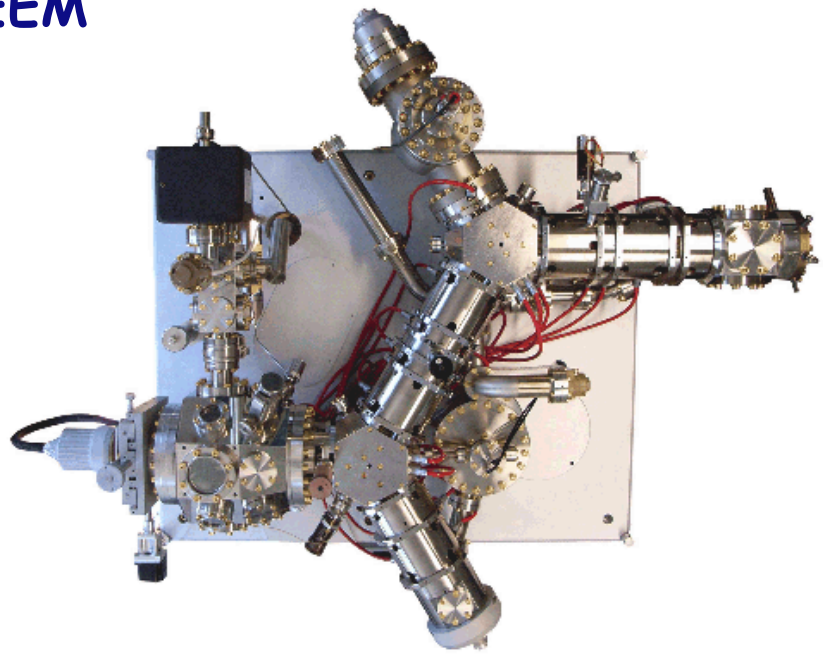
- Will allow users and staff to reduce detrimental electron scattering of gases in in-situ chemical reactions.
- As a result, data quality and energy resolution will improve.



Major Upgrades

Aberration corrected LEEM & PEEM

- Prototypes of aberration correctors operational
- Calculated resolution <3 nm at 8 x greater image intensity
- Fast switching between corrected and non-corrected modes
- Upgrade of existing Elmitec LEEM system



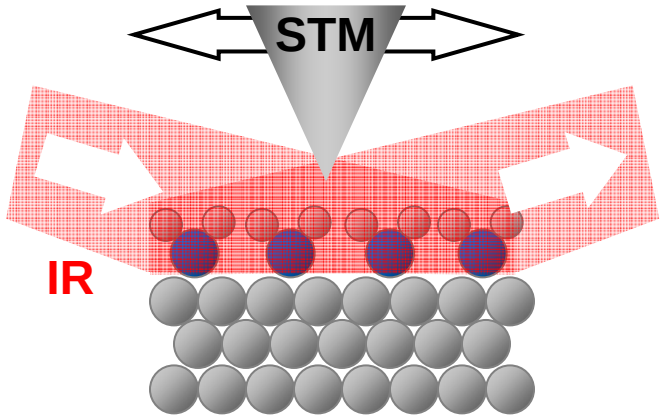
Target user science: in-situ surface microscopy at highest spatial resolution; synchrotron PEEM

CFN science connection: active phase of catalysts under reaction conditions

New Techniques

Reactor STM

- Small-volume catalytic flow reactor with integrated STM
- Samples prepared and and characterized in UHV
- Simultaneous STM imaging and mass spectroscopy of products



High-pressure STM & IR spectroscopy

- Simultaneous microscopic and spectroscopic information
- Integration of existing instruments

Target user group: scientists working on catalysis, corrosion, H storage

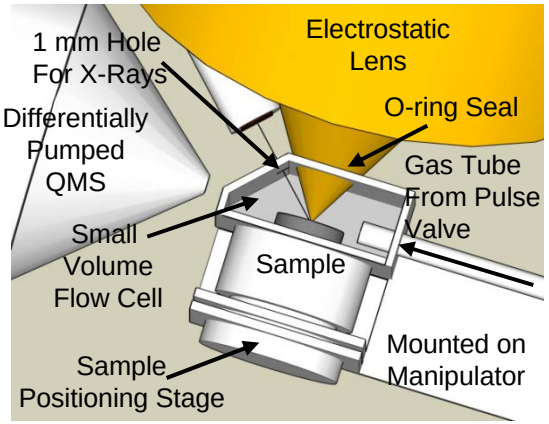
CFN science connection: active phase of catalysts under reaction conditions

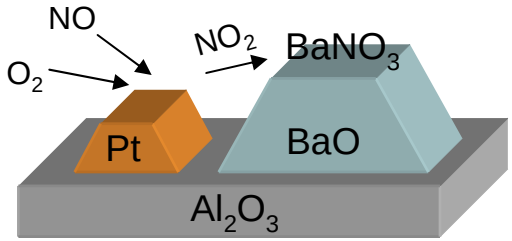
From New Techniques to New Science

High Pressure Photoemission at NSLS-II

Example: NOx Storage Catalyst

- Depends on inter-conversion of BaO to BaNO3
- What are the dynamics of this process?
- Needs time resolution on the ms timescale



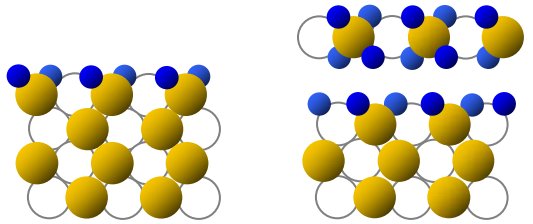


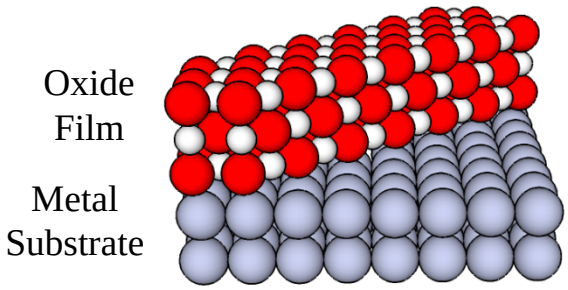
Interface Science & Catalysis

Contribute to rational catalyst design, by elucidating microscopic processes, their dynamic nature under reaction conditions, and their tuning by nanoscale phenomena

Scientific Questions

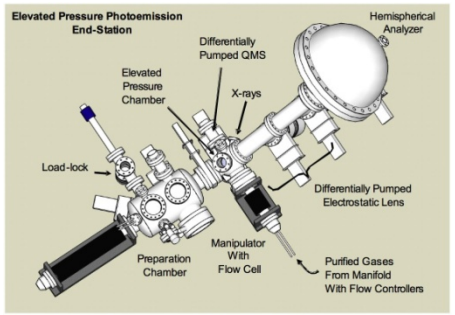
- What is the active phase of a catalytic system under reaction conditions?
  time, pressure, temperature
- What determines size-dependent reactivity?
  S/V ratio, electronic structure, active sites
- How is a catalyst affected by its support?
  oxide thickness





Approach

- Model systems with *atomic-scale precision*
- *In-situ* observation of dynamic restructuring under reaction conditions
- Development of new experimental techniques and tools.



Electronic Nanomaterials

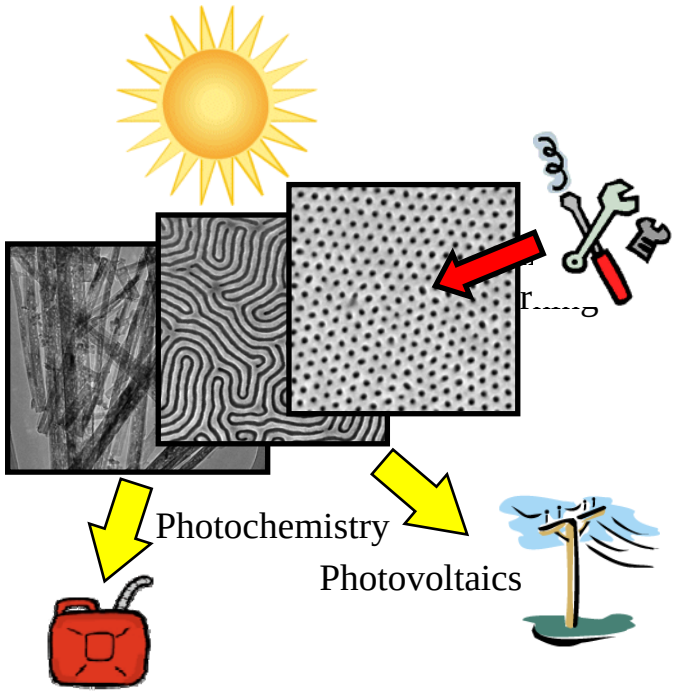
Use nanostructured materials with precisely defined dimensions to understand and improve critical steps to photovoltaic/photochemical energy conversion

Research Directions

- Nanostructured PV devices based on low-cost materials
- Assembly methods for large-area patterning at nm length scales
- Nanostructured materials for photochemistry

Scientific Questions

- How can physical processes be optimized?
  light collection, exciton diffusion and recombination
- How to use self-assembly materials and methods to direct structure?
- How to better capture the solar spectrum?



Soft & Bio Nanomaterials

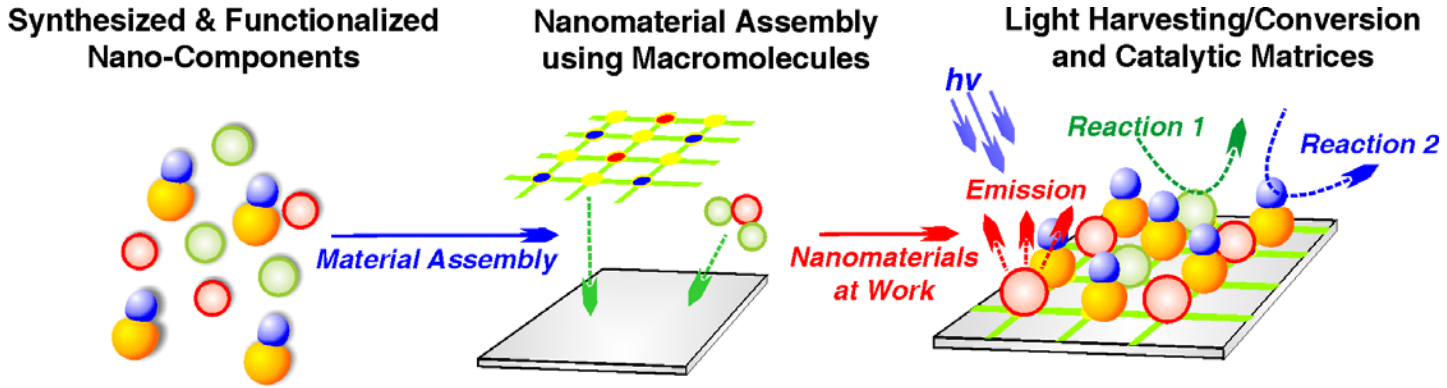
Develop methods for assembly and characterization of hybrid nanoscale systems and explore their functionalities for energy conversion

Scientific Questions

- How to assemble nano-objects in large well-ordered (3D) structures?
- How does addressable recognition compete with entropy and non-specific interactions?
- How to use hybrid structures for energy/charge transfer?

Approach

- Exploit properties of DNA and proteins
- Use in-situ structural and optical characterization

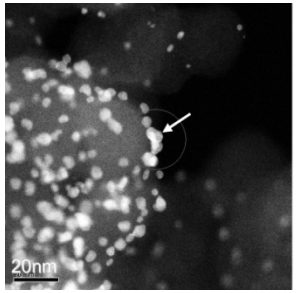


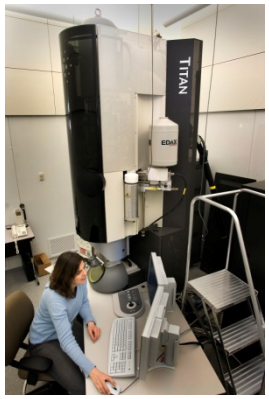
Electron Microscopy

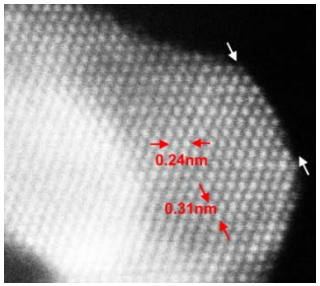
Exploit most advanced TEMs for quantitative microscopy and to address questions relevant to the CFN themes

Scientific Questions

- How are individual atoms arranged?
  nanocrystals, aperiodic objects
- What are chemical species, bonding states?
  chemical mapping
- What is structure-property response under various environments?
  temperature, pressure







Pd/Pt nanoparticles

Approach

- Apply capabilities to basic and applied science
  solid-liquid phase diagrams at the nanoscale
  catalysis (water gas shift reaction)
- Develop new techniques
  strain mapping, diffractive imaging

Theory & Computation

Characteristics

- Strong coupling to the CFN scientific themes
- Strong interaction with experimental research
- Complementary materials theory and methodology development

Research Plans

- Assembly and Pattern Formation

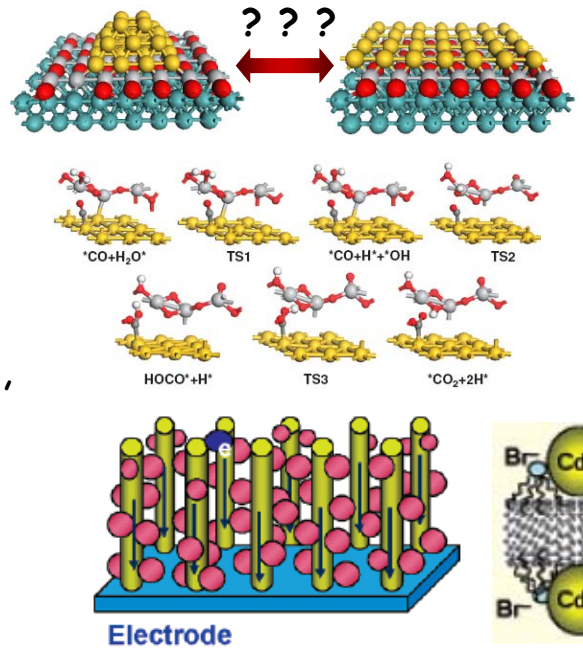
  how do interface interactions control structure and properties?

- Catalysis

  what are the active phases in a reaction, and can they be controlled?

- Electronic Properties

  how does energy and electron transfer occur in light-gathering systems?



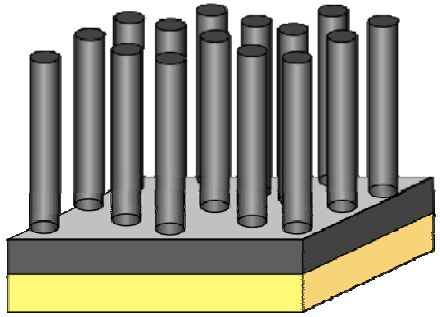
New Research Directions

Nanostructured Materials for Chemical Energy Storage

Technological Challenge

Improve battery energy- and power-density; maintain cyclability

intermetallic Li alloys have large storage capacity but volume increase limits cyclability

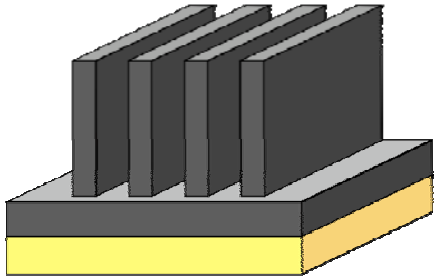


self-assembled pillar array

Advantages of Nanostructured Electrodes

- Increased surface area
- Stress relief to accommodate volume expansion
- Shorter Li diffusion lengths

  increased power delivery capacity
- Self-assembly approaches amenable to flexible material choices



anisotropically etched fin array

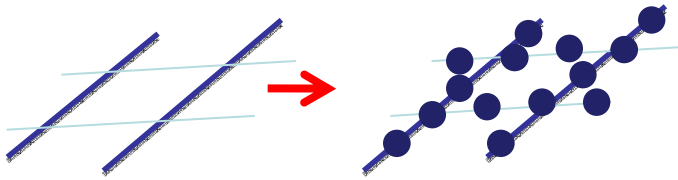
New Research Directions

Metallization of Macromolecular Structures

Technological Challenge

Increase materials density and incorporate different inorganic particles

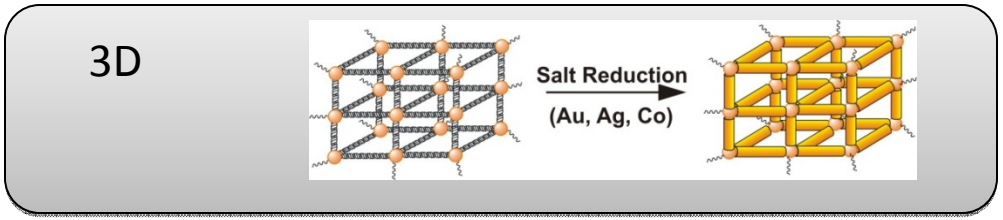
catalysis, batteries, fuel cells



macromolecular template metallization

Approach

Exploit CFN expertise in macromolecular structures

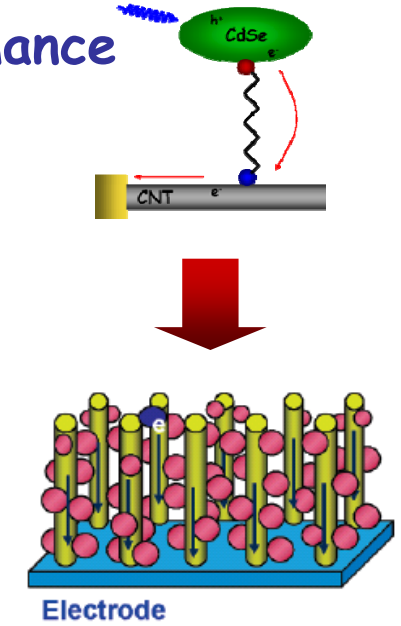


Plan

- N*on-specific* metal (Ag, Au, Co) deposition using chemical reduction of metal ions
- *Site-specific* deposition for placement of particles
- Development of in-situ structure characterization during metallization process and during function

New Research Directions

From Nanoscale Kinetic Processes to Device Performance



Challenge

Extend theoretical model of local kinetic processes to a multiscale device

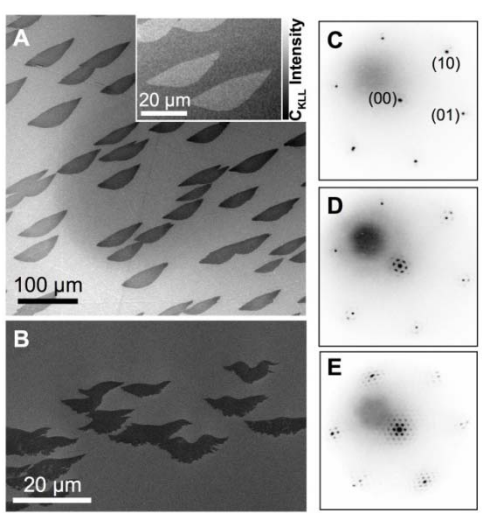
- CNTs for photovoltaics, sensing, …
- QD-organic hybrid photovoltaic concepts

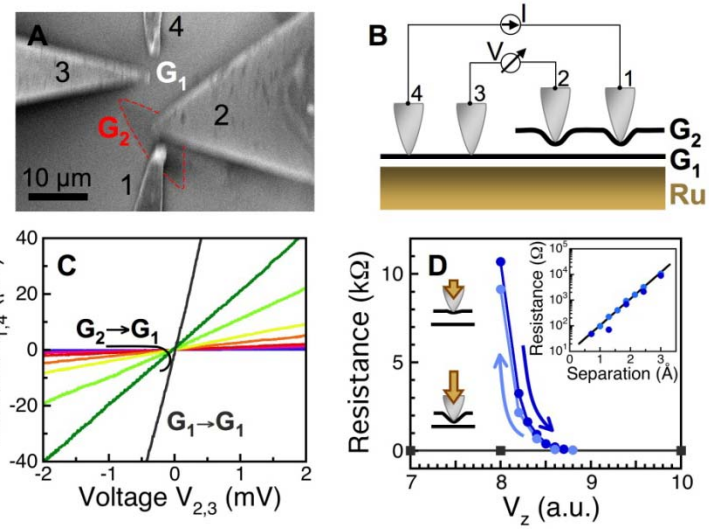
Plan

- Exploit CFN's expertise in quantum description of local kinetic processes
- Collaborate with external groups
- Hire new staff member, if feasible

Seeding New Directions

Epitaxial Graphene on Transition Metals: from Catalyst Template to Electronic Material?





(P. Sutter et al., Nature Mat., 2008)

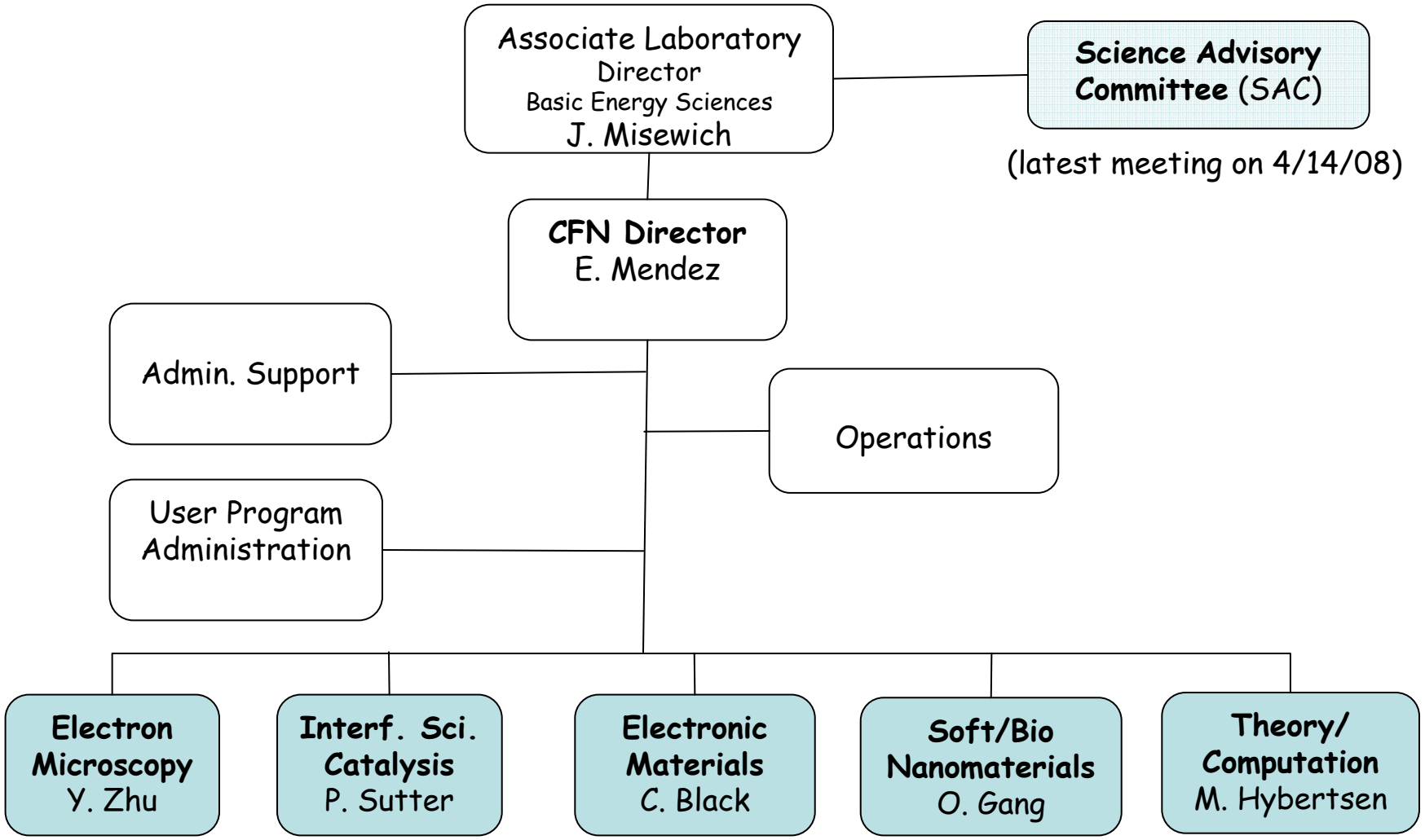
Original goal

develop moiré template for bottom-up synthesis of metal nanoparticles

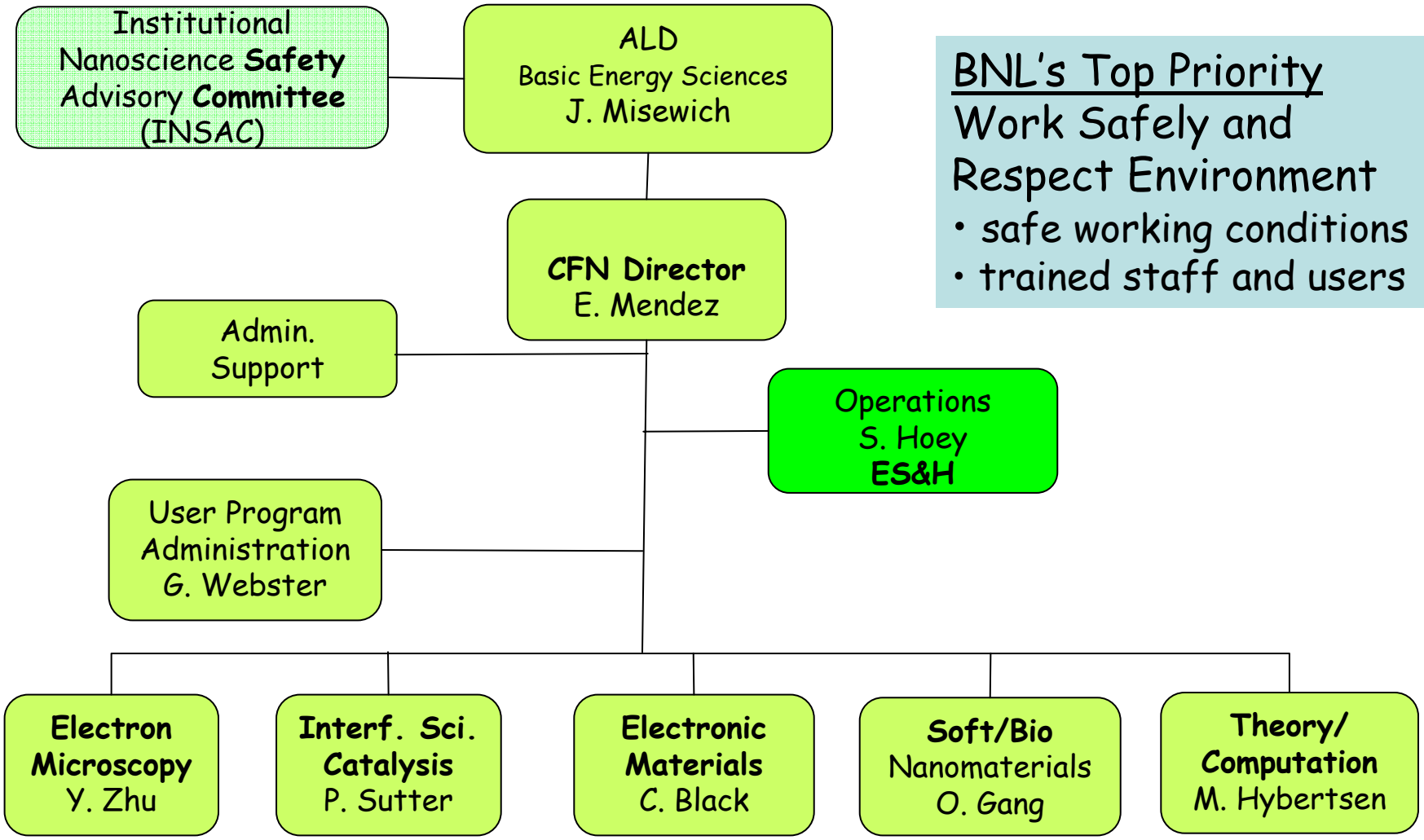
Potential pathway to epitaxy of large-area device quality graphene

- nature of graphene/Ru interface?
- influence of substrate on electronic structure?
- decoupling of additional graphene layers?
- electronic transport in graphene/Ru?

CFN Research



Safety at the CFN



BNL's Top Priority

Work Safely and Respect Environment

- safe working conditions
- trained staff and users

CFN Staff

- Vast majority have full-time CFN appointment
- Current number of FTEs: 46
- 7 scientists and technicians hired in last 10 months
- 2 more scientists to start in June, 2008

	CFN full time	CFN part time	**CFN FTE support**	Other Support (LDRDs, FWPs)
Scientists	14	2.75 FTE	13.75	3.00
Technicians	3	—	3	—
Postdocs	16	—	6	10
Operations	3	—	3	—
Administration	3	0.5	3.5	—
Total	39	3.25	29.25	13
Others	—	3.75	3.75	—
Grand TOTAL	**39**	7	**33**	**13**

CFN Staffing Plan

Medium Term (2010)

- Scientists
  - Photovoltaic devices
  - Catalysis chemistry
  - Hybrid nanodevices
  - Dynamics of excited nanosystems, theory
- Technicians
  - Electron microscopy
- Support
  - General administration
  - User and external programs

Development of a User Project

1. Potential user contacts facility leader (or delegate)
   to discuss project
2. User submits proposal during one of the three annual cycles
3. Proposal is evaluated for
   feasibility: safety/environment, capabilities, resources
   scientific/technical merit (by three external experts)
4. If evaluation is positive, time is allocated and schedule discussed
5. User performs experiment(s)
6. If necessary, user requests more time in subsequent cycles
7. User provides feedback about facilities
8. Ideally, user becomes an active member of the users' community
   and participates in town-hall meetings and annual Users' Meeting

SAC Members

Paul Fleury
Yale University

Jens Norskov
Tech. Univ. Denmark

Frans Spaepen
Harvard University

Ruud Tromp
IBM Watson

James Yardley
Columbia University

Jingguang Chen
University of Delaware

Giulia Galli
UC Davis

Tom Lubensky
University of Pennsylvania

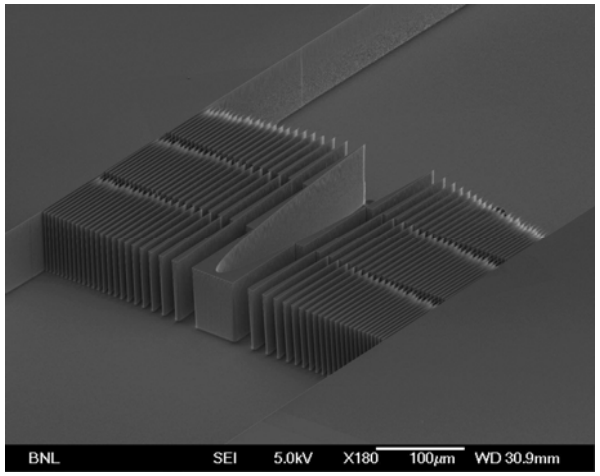
Ali Yazdani
Princeton University

Clare Grey
Stony Brook University

Examples of User Projects

Fabrication of Kinoform Lenses for Hard X-ray Focusing

K. Evans-Lutterodt (NSLS/BNL) and A. Stein (CFN)

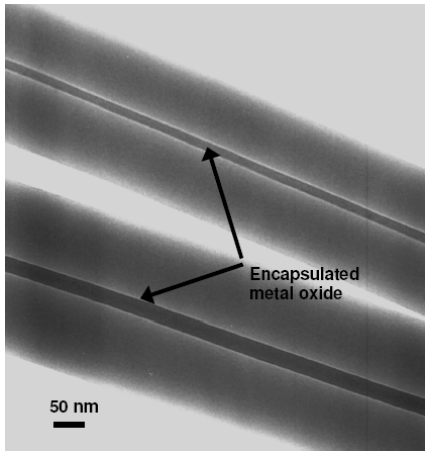


Lenses fabricated using electron beam lithography and deep silicon etching.

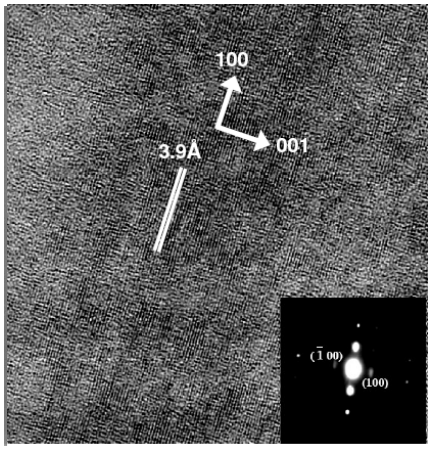
K. Evans-Lutterodt et al. Phys. Rev. Lett. **99**, 134801 (2007)

Electrospun Single Crystal MoO3 Nanowires for Sensing Probes

P. Gouma, K. Kalyanad., and A. Bishop (Stony Brook University)



TEM image of a as-spun polymer-metal oxide nanocomposite



HRTEM image of a MoO3 nanowire on a Si3N4 grid, showing growth direction

P. Gouma et al., J. Mater. Res. 21, 2904 (2006)

Proposal's Evaluation Criteria

1. **Highly innovative** nanoscience project; could launch a new nanaoscience application or impact an outstanding problem. CFN facility is crucial.
2. **Original project** with potential for important contribution to nanoscience and publication in leading journals. Nanoscience facility is essential.
3. **Very solid project**, not groundbreaking, but near cutting-edge and likely to produce significant results. Nanoscience facility tools are needed.

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4. Routine project in a well-worked area of research. Results unlikely to create excitement in the field. Nanoscience facility is needed.
5. Project of doubtful scientific /technical value. There is no clear need for nanoscience facility.

Recent Scientific Accomplishments

2007-08

› 40 papers in high-impact journals
- 3 Nature (1 cover)
- 1 Science
- 1 PNAS
- 2 PRL
- 2 APL
- 6 Nanoletters
- 3 JACS

9 invention disclosures
- BNL inventor of the year

Sci.Am 50 Award 2007

LETTERS

Dispensing and surface-induced crystallization of zeptolitre liquid metal-alloy drops

PETER W. SUTTER AND ELI A. SUTTER*

Activity of CeO_x and TiO_x Nanoparticles Grown on Au(111) in the Water-Gas Shift Reaction

J. A. Rodriguez,[3]* S. Ma,[1] P. Liu,[2] J. Hrbek,[1] J. Evans,[3] M. Pérez[3]

PRL 99, 134801 (2007) PHYSICAL REVIEW LETTERS week ending 28 SEPTEMBER 2007

Using Compound Kinoform Hard-X-Ray Lenses to Exceed the Critical Angle Limit

K. Evans-Lutterodt,* A. Stein, J. M. Ablett, and N. Bozovic†
Brookhaven National Laboratory, Upton, New York 11973, USA

A. Taylor and D. M. Tennant‡
Lucent Technologies, 600 Mountain Avenue, Murray Hill, New Jersey 07974, USA
(Received 9 January 2007; published 28 September 2007)

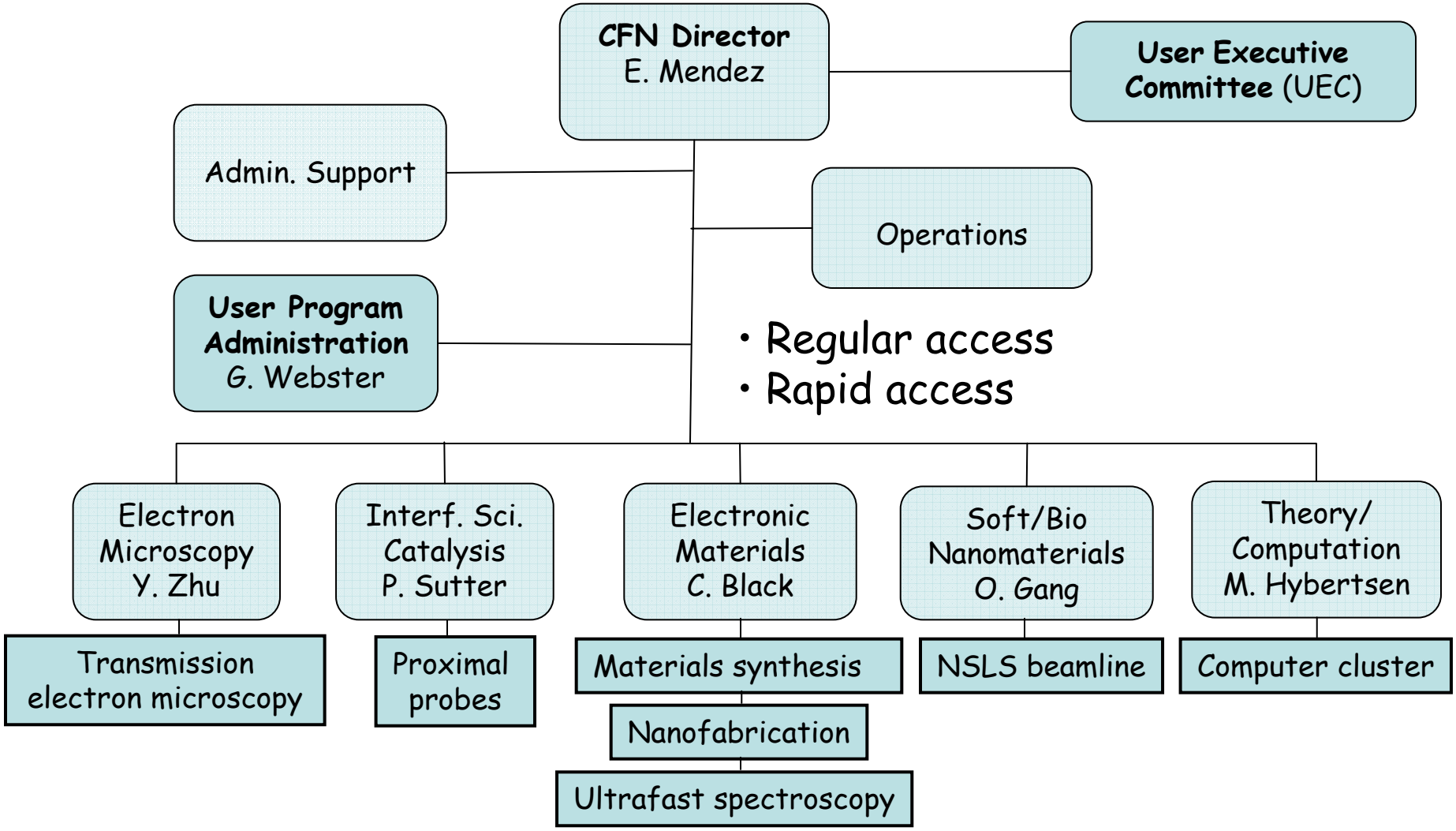
doi:10.1038/nature06560 nature

LETTERS

DNA-guided crystallization of colloidal nanoparticles

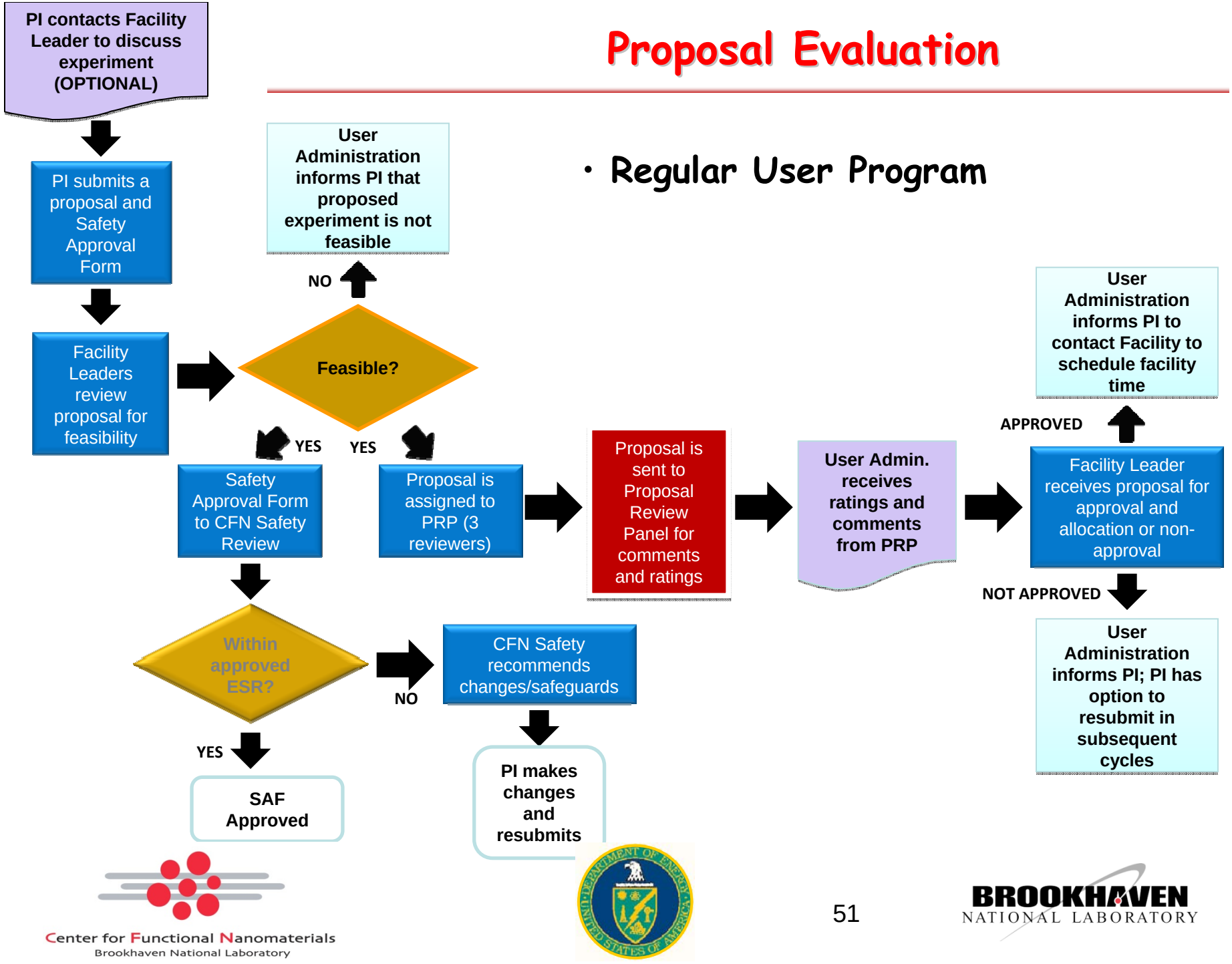
Dmytro Nykypanchuk[1]*, Mathew M. Maye[1]*, Daniel van der Lelie[2] & Oleg Gang[1]

The CFN User Program



Proposal Evaluation

- Regular User Program



Center for Functional Nanomaterials
Brookhaven National Laboratory

BROOKHAVEN NATIONAL LABORATORY

User Access

- ***General Users*** (Tof Carim, BESAC Meeting, 2/22/08)
  - ***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***
  - ***Access based on peer merit review of submitted proposal, evaluated by external Proposal Review Committee and/or by Scientific Advisory Committee***
  - ***Enhance capabilities of and/or contribute to operation of facility, with benefits to the general user community***
  - ***Defined and limited period of reserved time or preferential access***
- ***Large majority of time must remain available to general users***
- ***A very limited amount of time may be allocated directly at the discretion of the NSRC Director or management for rapid access***
- ***Collaboration with facility scientists is an important potential benefit to users, but is not required. Facility staff may provide input on feasibility and time needed but do not select or approve proposals.***

Who Is a User of the CFN?

Someone outside the CFN who …

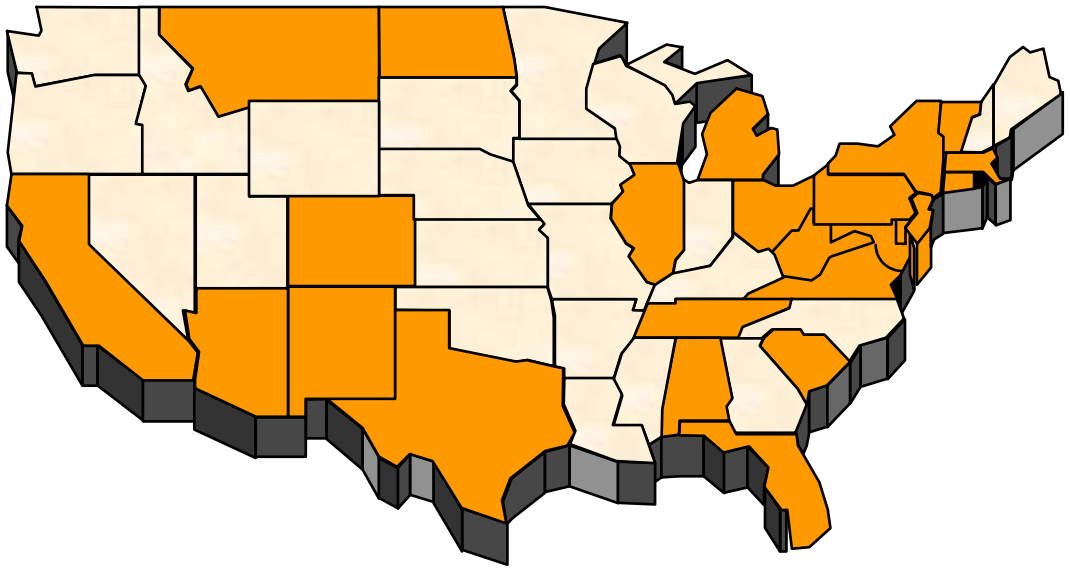
- Performs a standard measurement or process in a CFN facility, typically for one or two days, and interacts mostly with professional or technical staff.

- Performs a complex experiment that requires elaborate preparation and last one to two weeks (possibly repeated), and interacts with technical and scientific staff.

- Performs complex experiment during summer or sabbatical period and interacts extensively with scientific staff.

- Collaborates with scientific staff in joint scientific project that uses the CFN facilities as well as complementary facilities in home institution.

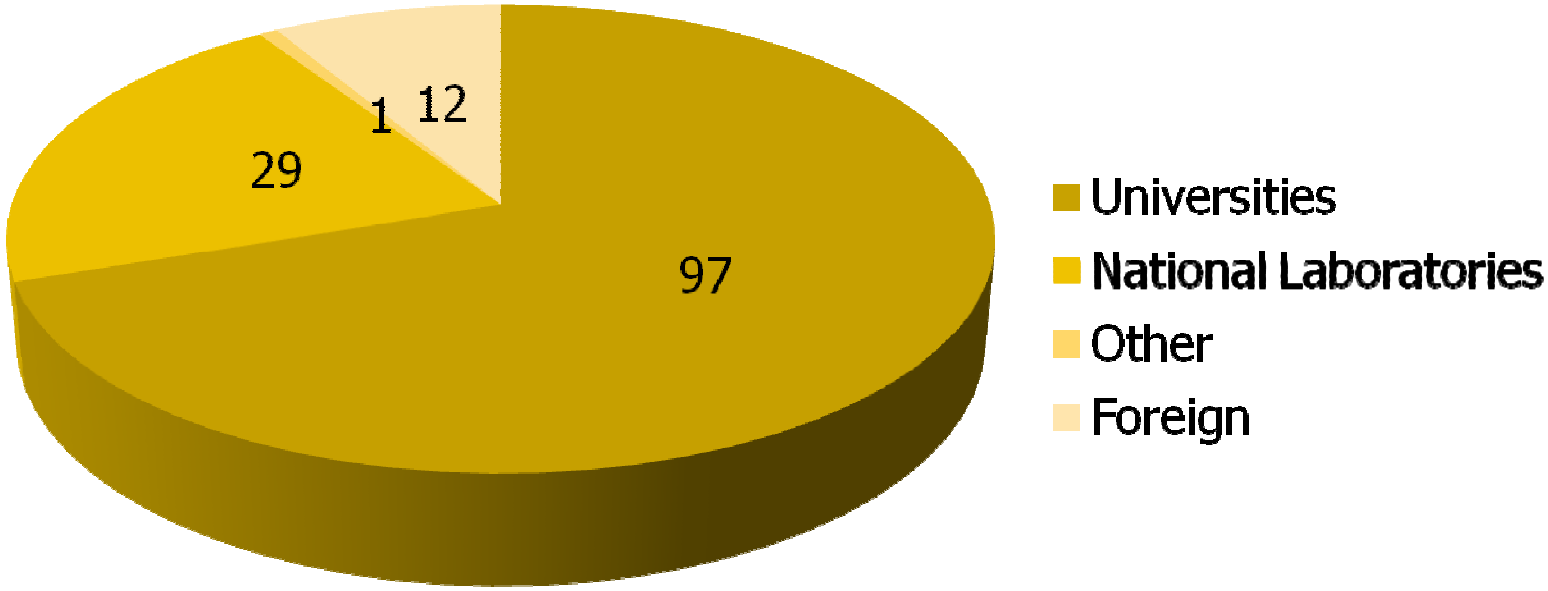
Geographic Distribution of Proposals



Canada
France
Germany
Hungary
Israel
Italy
Thailand
UK

Alabama – 1	Florida – 1	Montana – 2	Ohio - 2	Virginia – 1
Arizona – 3	Illinois – 2	North Dakota – 2	Pennsylvania - 8	Vermont – 3
California – 3	Massachusetts – 4	New Jersey – 19	South Carolina – 7	Washington – 1
Colorado – 4	Maryland – 4	New Mexico – 3	Tennessee – 2	Wisconsin – 1
Connecticut – 9	Michigan – 3	New York – 150	Texas – 3	West Virginia - 1
Delaware - 7				

Institutional Distribution of Proposals



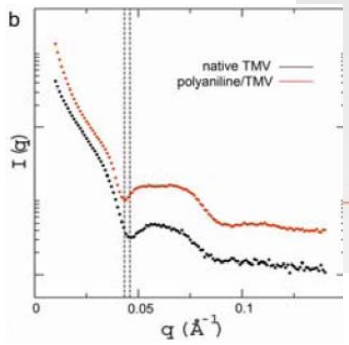
Nanomaterials Safety at NSRCs

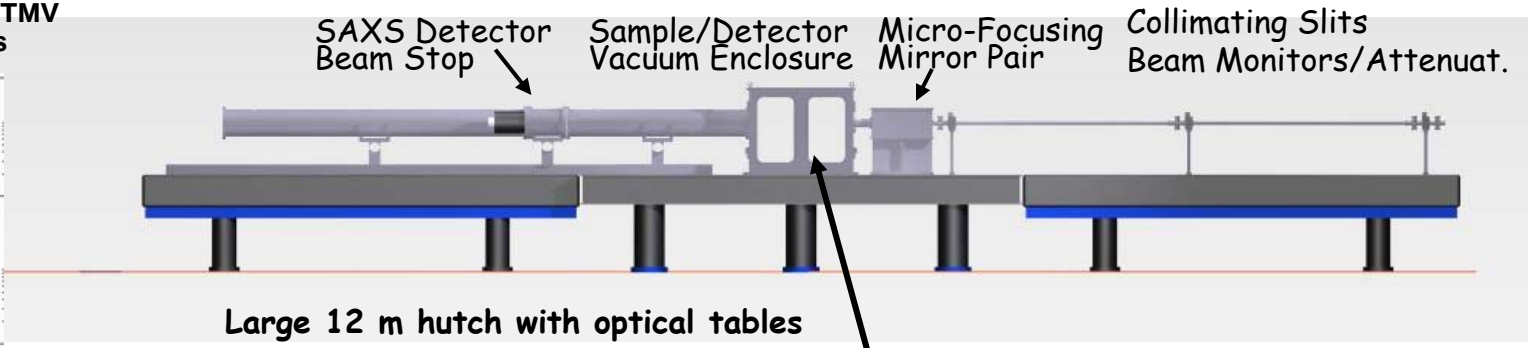
- *NSRCs Collaborated on Design Construction & Operations*

- Shared construction safety experiences
- Share information on Nanosafety
- Share information on User ESH
- 2003: Operations/ES&H people began informal teleconferences with BES-ES&H
- Today - a chartered (by NSRC Directors) activity
- Members are involved in consensus standards development.
- Developed an "Approach to Nanomaterial ESH" best practices document.
- Approach doc is based on a precautionary principle "treat all nanomaterials as hazardous until proven otherwise"

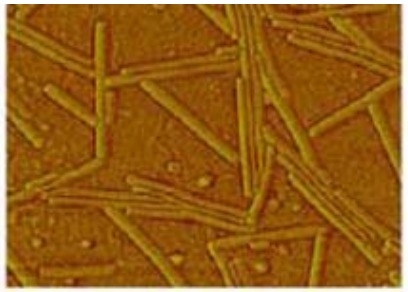
Small/Wide Angle X-Ray Scattering at NSLS Endstation

SAXS profiles of native TMV and TMV nanofibers



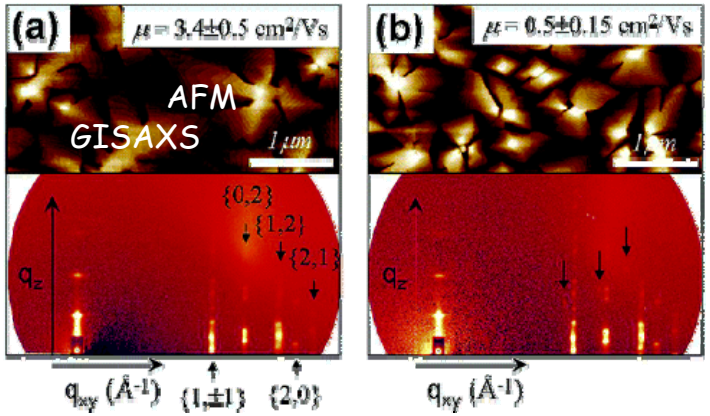


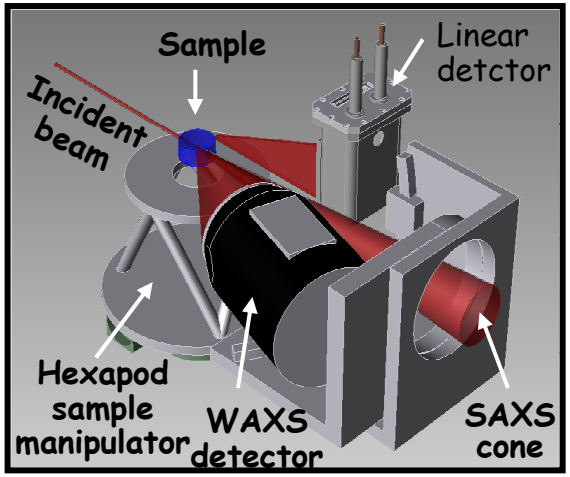
Large 12 m hutch with optical tables



SAXS confirmed coating of Tobacco Mosaic Virus nanorods to promote end-to-tail self-assembly into long (microns) nanofibers

Grazing Incidence SAXS (GISAXS) used to correlate the structure of organic semiconducting films prepared using different surface treatments with their electronic properties, Prof. Yang, *RPI.*





Simultaneous Small and Wide Angle X-Ray Scattering Set-up

Operations and Review Schedules for NSRCs