Industrial Research Program

Industrial Research at NSLS-II
A Workshop to Showcase NSLS-II Capabilities and Create Industry Partnerships
8-9 April 2014, Brookhaven National Laboratory

Jun Wang

U.S. DEPARTMENT OF ENERGY

BROOKHAVEN NATIONAL LABORATORY
BROOKHAVEN SCIENCE ASSOCIATES

Outline

- Motivations
- On-going Efforts at NSLS
- Examples
- Transition Plan
- Summary

Major Activities in Photon Sciences Directorate

- NSLS operations

Shut down end of FY13

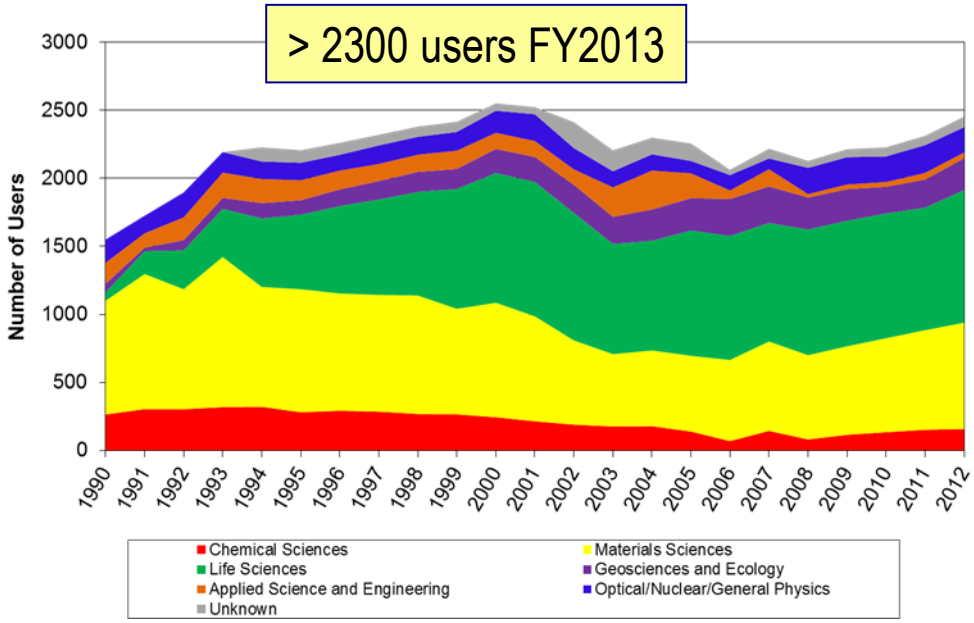
- NSLS-II construction

Commissioning Oct, 2014

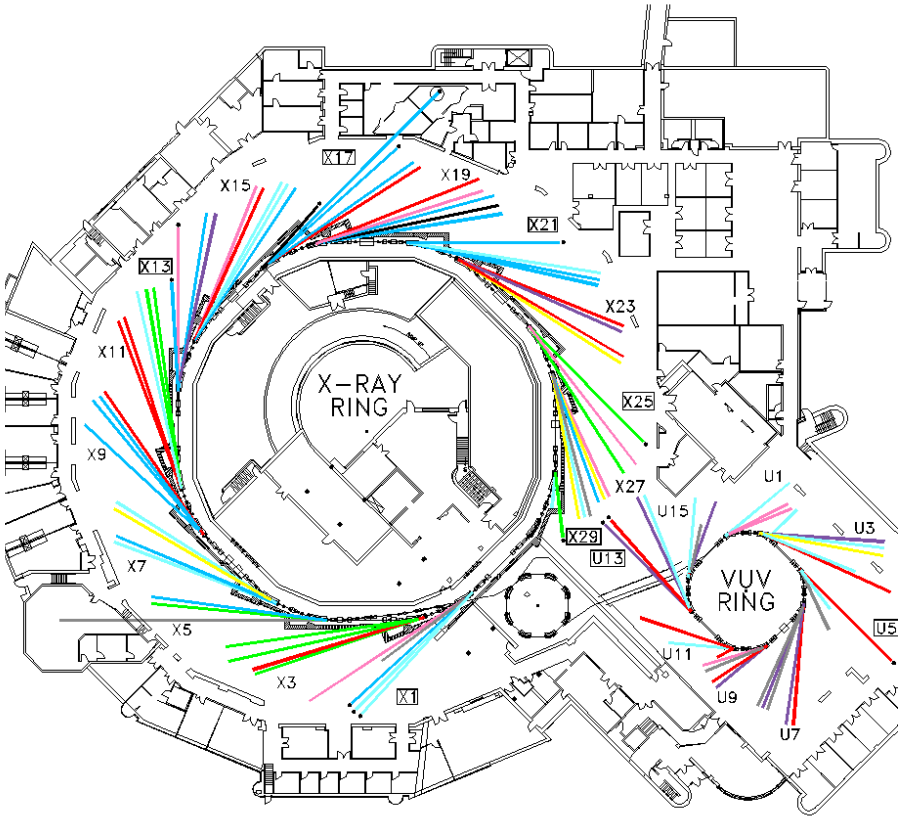
- Developing strategic research thrust areas:
  - Physical sciences: catalysis, energy storage, superconductivity, environment, ….
  - Life sciences: molecular biology, genomics, cell biology, medical research, ….
  - Applied science & industry: engineering materials, systems, devices

National Synchrotron Light Source (NSLS) Facility

- NSLS operates 59 beamlines on X-ray & VUV rings:
  - 48 X-ray beamlines and 11 UV/IR beamlines
- NSLS is a national synchrotron facility – over 2300 visiting scientists (users) per year perform experiments in broad range of scientific disciplines



Statistics provided by Gretchen Cisco, user office



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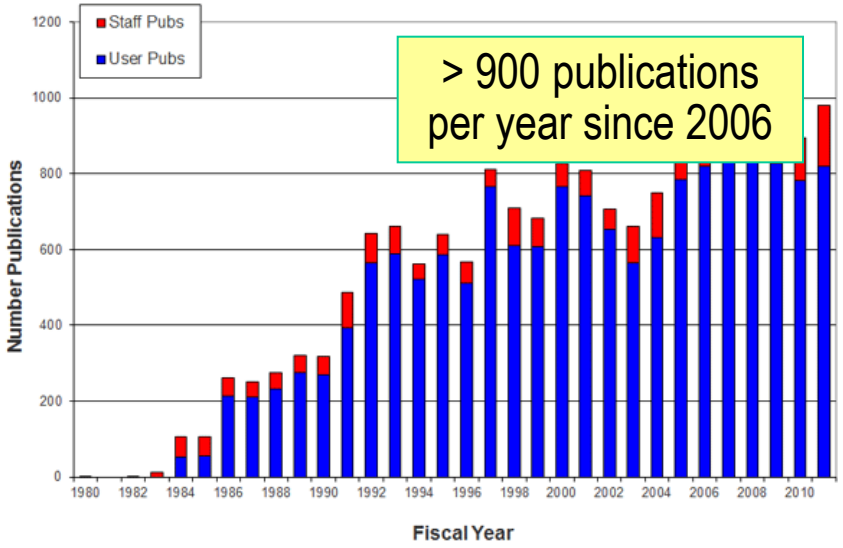
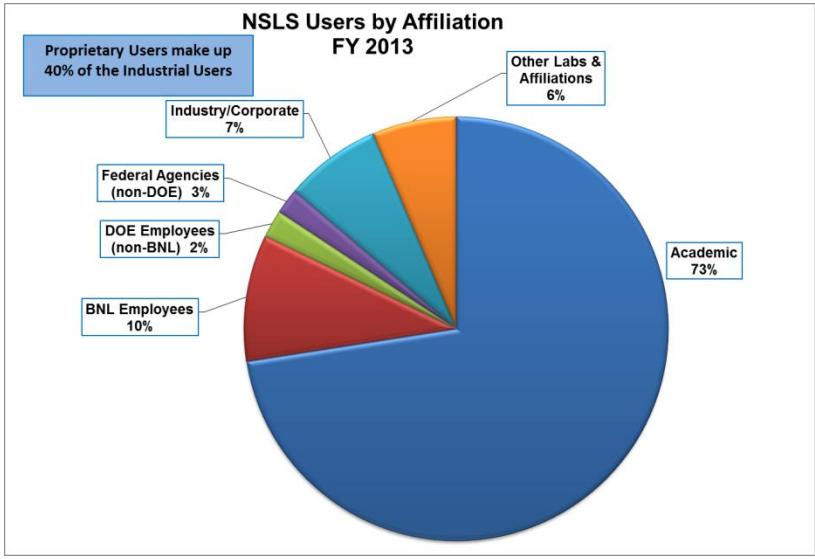
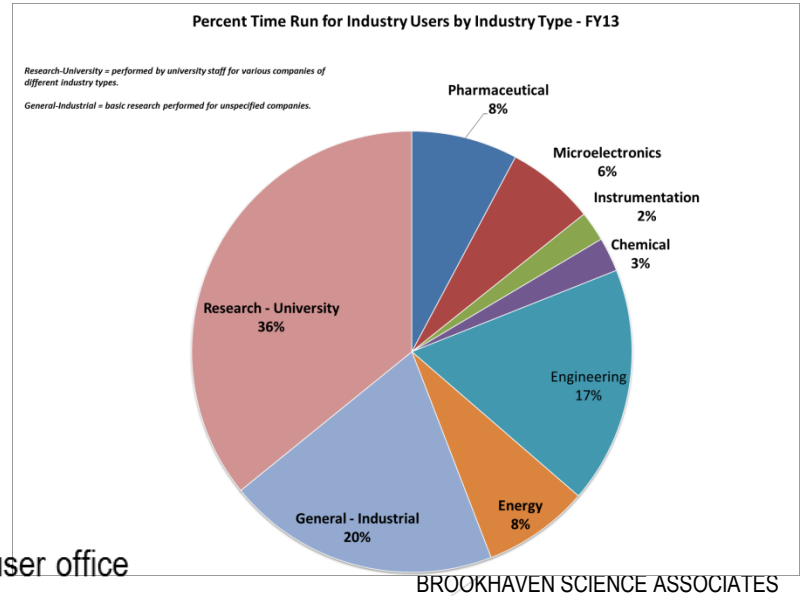
NSLS Users and Publications



NSLS Users by Affiliation



- Outstanding scientific productivity at NSLS
- Strong and diverse user community, including 7% industry users (not counting industry supported university groups)
- Photon Sciences at BNL is particularly interested in enhancing beamline access for industry user community, and in developing partnerships with industry to transition existing programs and to build new facilities at NSLS-II



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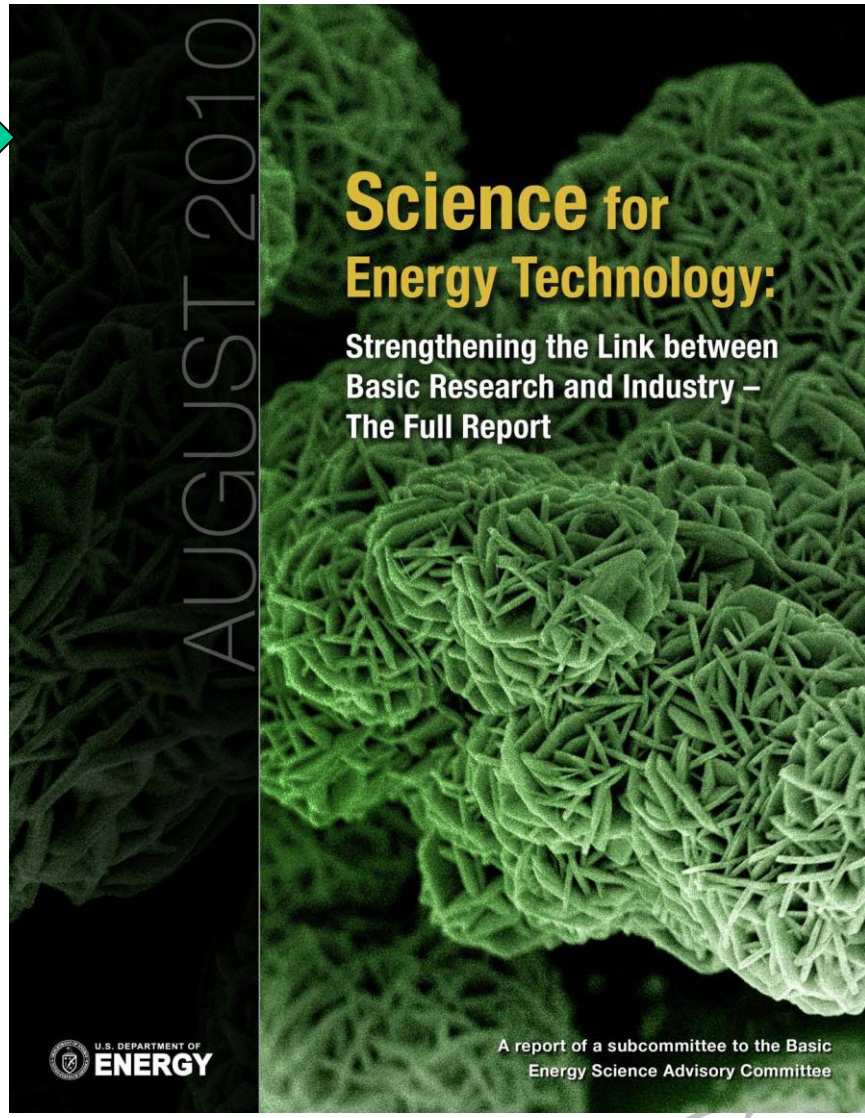
Statistics provided by Gretchen Cisco, user office

Motivation

Particular attention was given to identifying industry needs and the impact of basic science, of the type addressed by DOE's Office of Basic Energy Sciences (BES), in resolving the show-stoppers to industry progress.



- The desire to bridge basic and applied research
→ Synchrotron facility is ideally suited because a shared facility provides a common 'meeting ground' for industrial, academic and government researchers

- Needs expressed by industrial researchers
→ Unique instruments and advanced techniques, staff expertise, and potential collaborators from other divisions and universities.

- Enhance global competitiveness of our nation's industry and build the economy
→ Bring state-of-art research capability to our industrial community, help them improve products and processing

Challenges and Opportunities

Challenges

- Synchrotron-based industrial participation
  → has evolved
- Industrial interests
  → focus more on applied research and process engineering
- Industry-PRT (Participating Research Team) model
  → PRT to Partner User and GU

Opportunities

- New strategy is critically necessary
  → meet changes
- Open up opportunities
  → form new partnership with industries and join efforts between partners to tackle the challenges facing society today: microelectronics, energy, and human health

Industrial Research Program

- Improve the NSLS proposal review system:
  - Proposal rating review criteria modified to reflect the importance of technology development
  - Dedicated proposal review panels for industrial proposals created to equalize industrial proposals to those from academia and government laboratories
- Increase availability of beamtime to industrial researchers:
  - New capacity: e.g. TXM project, new ambient high-T furnace
  - Increased throughput : NSLS project for remote access at X14A, X18A, X20B, X27C
  - Work with Participating Research Teams (PRTs) to increase general user fraction: X14A (ORNL), X27C (SBU), X20B (IBM)
  - Partitioned up to 10% beamtime on selected beamlines for industrial users: X14A, X27C, X18A, X8C

Industrial Program Support

Support industrial users is categorized into four areas: consultation, collaborative research, information and outreach, and paid services (under development).

Consultation: help users identify the most suitable techniques and guide them to the appropriate beamlines.

Collaborative Research: develop collaborative research, joint funding opportunities and write proposals, develop new techniques, methods and instruments, perform experiments, and analyze data. (e.g. GE, IBM, Duracell GM, UOP and Henkel)

In-Situ XRD Studies of Na-Metal-Halide Batteries

J Rijssenbeek, Y Gao, Z Zhong, M Croft, N Jisrawi, A Ignatov, T Tsakalakos, *J. Power Sources*, **196**, 2332–2339 (2010).

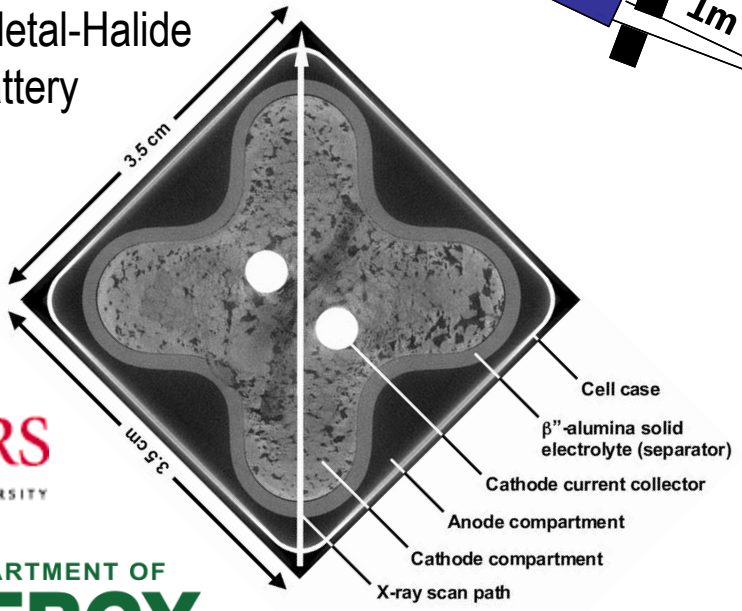
10% fuel savings and 10% emissions reduction

Energy Dispersive X-Ray Diffraction

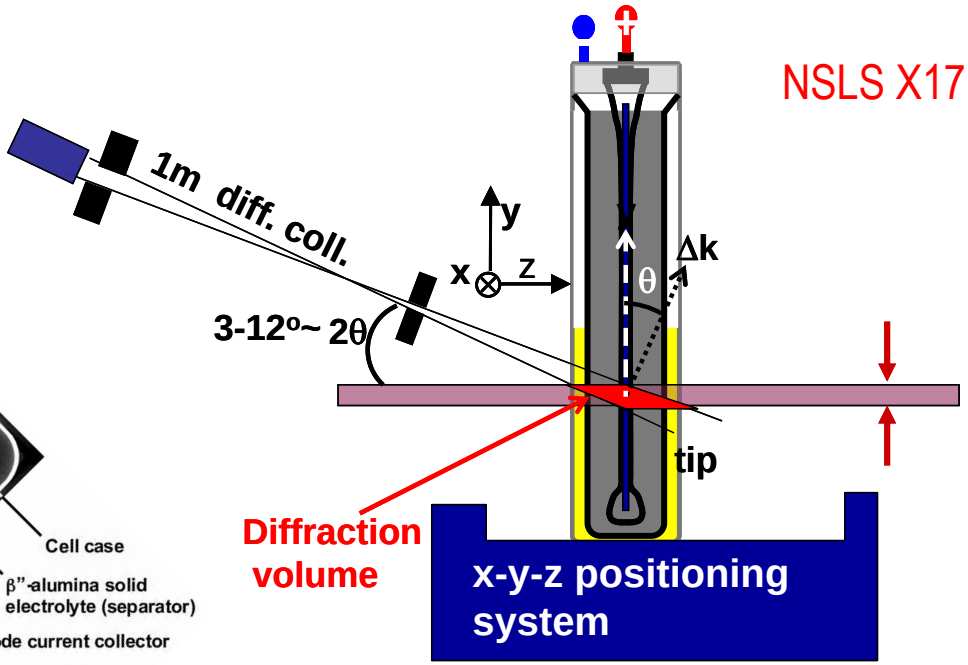
4D-Phase Mapping: 3D space + time

During charge cycle: NaCl / M → Na / MCl_2 where M=Ni-Fe alloy

GE Na-Metal-Halide Battery



NSLS X17B1



RUTGERS
THE STATE UNIVERSITY OF NEW JERSEY

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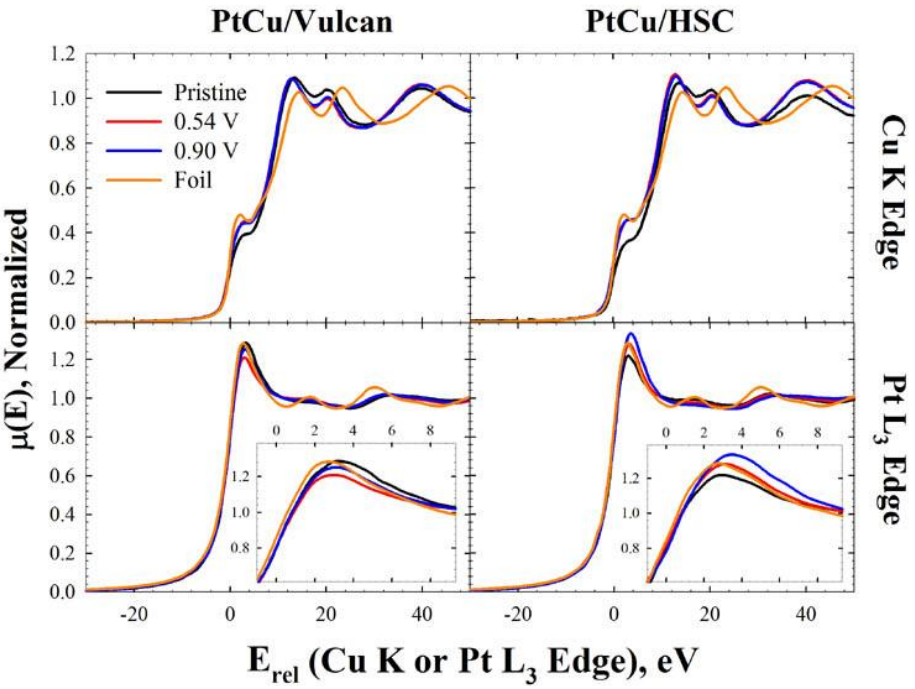
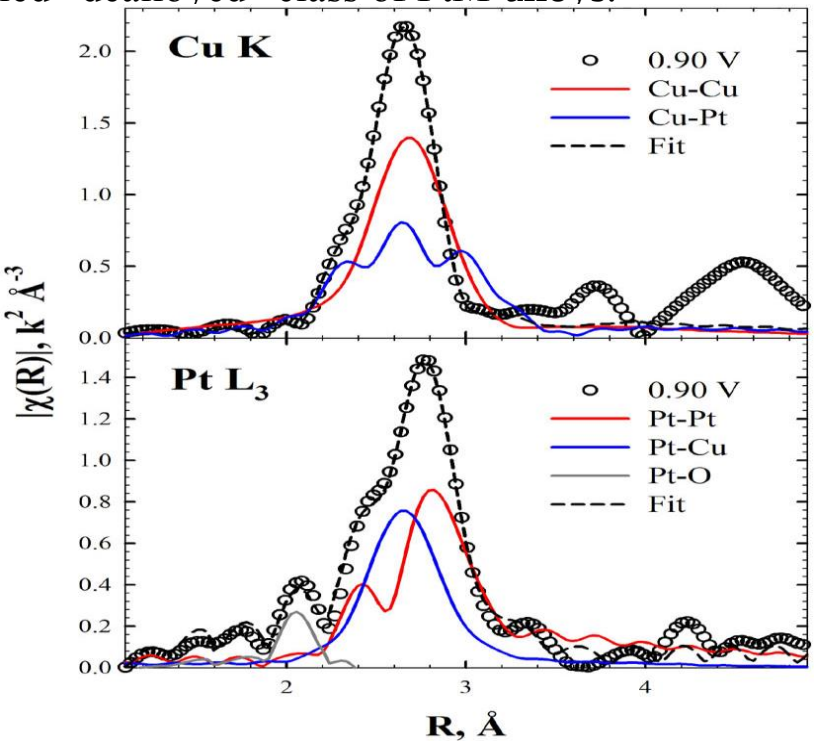
In-situ EXAFS/XANES on Dealloyed PtCu/C: possible path to 8x mass activities

J. M. Ziegelbauer, ***GM***, ECS meeting 2011 in Montreal. Beamlines X-23A2 & X-18A (NSLS), 9BM (APS)

Reduction in Pt loadings is imperative for the economic viability of PEMFCs for transportation applications. One such avenue towards Pt loading reduction is the so-called "dealloyed" class of PtM-alloys.





- Pt acts as the reaction center
- construct a core-shell model of the average catalyst particles
- Pt d-bands vacancies showed that compressive strain may not be the only property that gives rise to the enhanced Pt mass activities of these systems, but a ligand effect may also be induced through the Pt shell by the underlying Cu-rich PtCu core

Already being used to design and synthesize the next generations of these types of systems

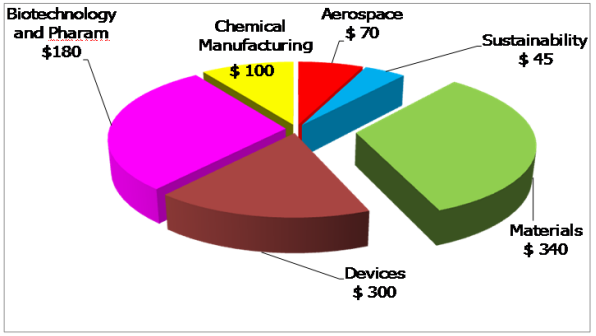
Characterization of Nanoclay Orientation in Polymer Nanocomposite Film by SAXS

B. Hsiao' group at Stony

ExxonMobil Research and Engineering

- Nanoparticles are used in making variety of nanocomposite materials, including materials that are incorporated into tires. The orientation of these organoclays dispersed in a polymer matrix is an important parameter to control the properties of polymer nanocomposites.

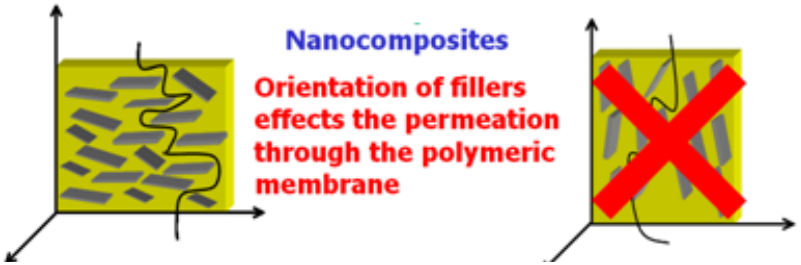
- By using SAXS analysis, we determined the degree of orientation and dispersion of these nanoclays, and also the correlation of the clay orientation and dispersion of nanoclays with the gas permeability of system.



Projected ~$ 1 tr. by 2015 and nanomaterials has prominent role (all values above are in $ billion) Source: National Science Foundation

Nanomaterials incorporated in the inner-liner of tires to enhance properties of tires.

Impact on Society



Clay Orientation and Gas Permeability

In conclusion, the orientation of organoclay is responsible for the improvement of gas permeation properties in polymer nanocomposites.

Lower gas permeability → Better tire performance → Significant savings on gasoline (up to 30%)!

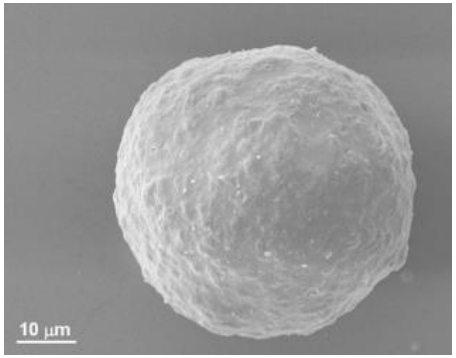
Polymer (2010), doi:10.1016/j.polymer.2010.08.048. Work performed on X27C beamline

Characterization of a Fluidized Catalytic Cracking Catalyst

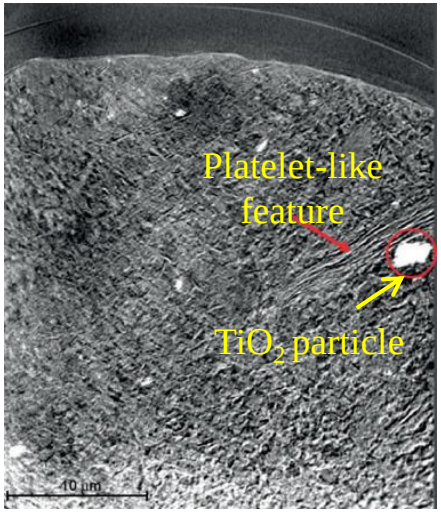
X8C

UOP
A Honeywell Company

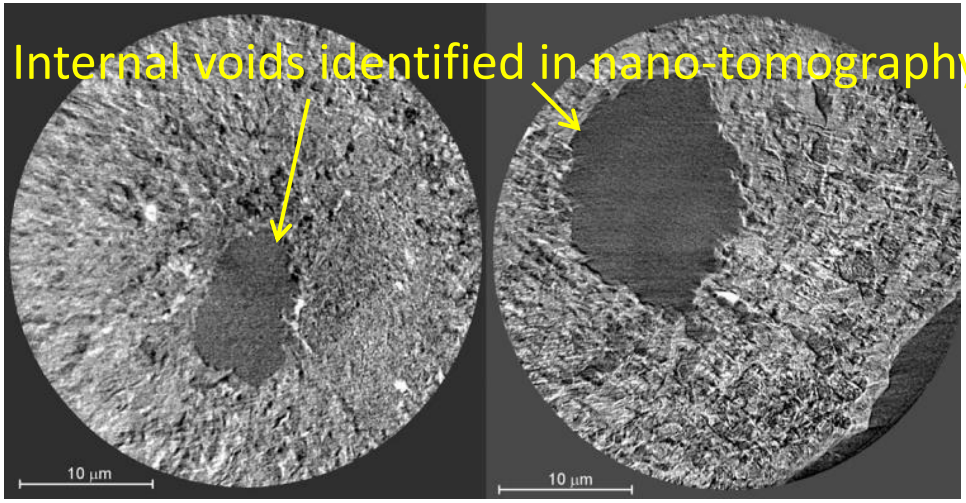
with Simon Bare

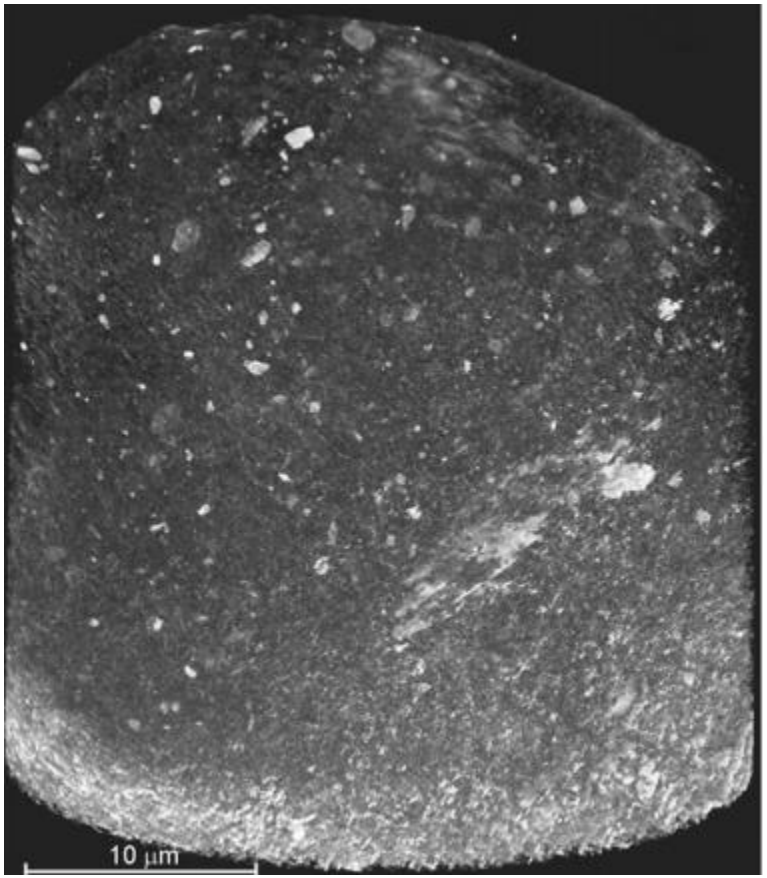


SEM: surface of equilibrium fluid catalytic cracking catalyst (ECAT)



TXM Cross-section

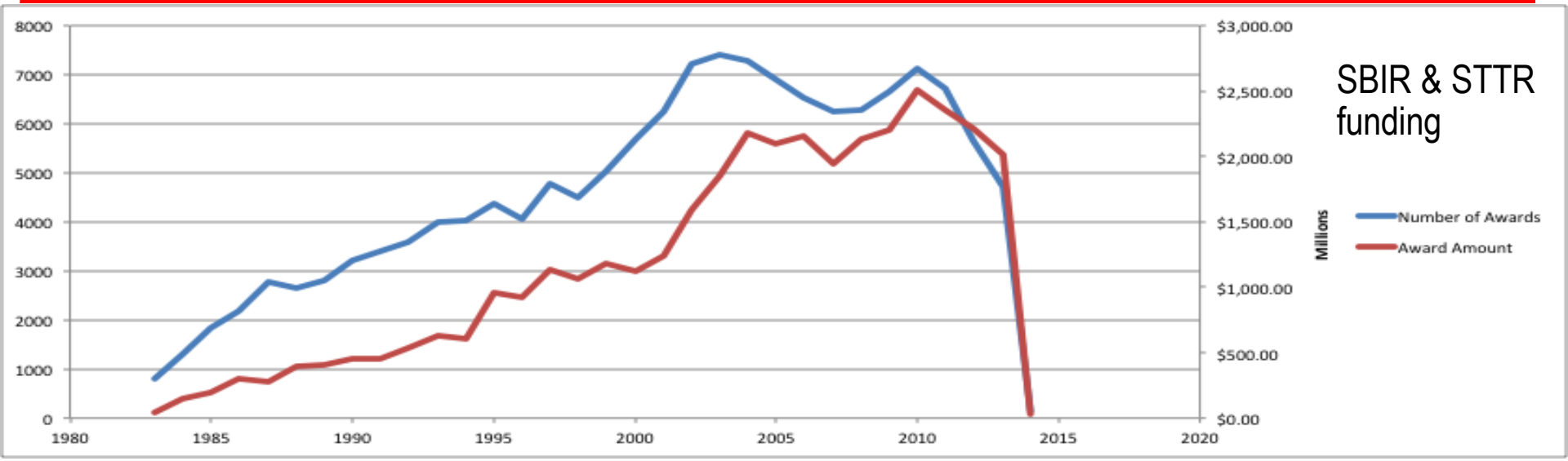




TXM 3D volume rendering

Result published in ChemCatChem, 2014 by Simon et al.

COLLABORATIONS BETWEEN BNL AND SMALL BUSINESSES



2 Examples for SBIR and STTR proposals (Phase I and Phase II awarded):

- Advanced Compact X-Ray Spectrometer with High Resolution and Efficiency
  collaboration: Incom Inc & PS
  Award Year: 2013 (phase I), 2014 (phase II)
- Theory-Based High-QE Photocathode Development
  collaboration: RMD & PS
  Award Year: 2013 (phase I), 2014 (phase II)

Industrial Program Support

•Information and Outreach

Targeted communication - new webpage dedicated to industrial users created http://www.bnl.gov/ps/nsls/industry

- Workshops
- Meetings (industry-focused)
- Visit industries
- Host industrial visitors
- Funding proposals
- New beamline proposals related to industrial research for NSLS-II (FXI, IXD, HEX)

- synchrotron techniques with detailed descriptions of capabilities of each beamline
- highlights of applications of the research performed in different industrial areas
- policies pertaining to performing experiments at the NSLS.

National Innovation Summit
Speakers from NSLS industrial users

Washington, DC | May 12-16, 2013
NATIONAL INNOVATION
SUMMIT AND SHOWCASE

- A White House and U.S. Congress initiative to showcase the emerging technologies in industry and academia that are ready for commercialization.
- Not only regular scientific talks and poster sessions, but also keynotes, panel discussions and roundtables that include industrial innovation executives and government representatives.
- Opportunities for both individual and corporate inventors to network with government agencies and large corporations that are looking for the “next breakthrough”.

In parallel with the Summit, there is also a Conference on Small Business Innovation Research (SBIR) and Small Business Technology Transfer programs (SBTT), a source of early stage / high risk R&D funding for small business. Exhibition by equipment vendors and various governmental agencies also accompany the meeting

Parallel Session: How National User Research Facilities Can Give Your Company an Advantage - Special Session, 40 attendees

Dr. Ben Brown (Senior Science and Technology Advisor, U.S. Department of Energy)

Dr. Alex Norman, (Principal Scientist, United Technologies Research Center), NSLS industrial user

Dr. Stan Petrash (Research Scientist, Henkel Corporation), NSLS industrial user

Dr. Altaf Carim, Director for Nanotechnology at the White House Office of Science and Technology Policy.

Transition Period Support

Working with other facilities (SSRL, APS, ALS)

- Spectroscopy
- Imaging
- High energy x-ray diffraction
- Protein crystallography

THE DARK PERIOD FOR SPECTROSCOPY

Klaus Attenkofer

Current spectroscopy (hard) situation at NSLS

- NSLS shuts down end of September 2014
- NSLS has about 5 fully working and dedicated EXAFS beamlines covering various focus areas
- No dedicated ID-lines and no emission spectroscopy available

Future at NSLS-II:

- No “project beamline” is dedicated to hard x-ray spectroscopy
- There will be at one point 4 beamlines: ISS (ID-line), QAS, TES, BMM
- There are also 2 micro (nano) probes with spectroscopy capabilities (project beamlines, HXN, SRX)

Timeline:

- Most likely first general user experiments end of FY 16
- Dark period for hard x-ray spectroscopy at BNL

Agreement with SSRL to help our users during this time

SSRL AGREEMENT

Klaus Attenkofer

Beamline

- Bending magnet 2.2
- Energy range 4.9-20keV
- Typical EXAFS capabilities (with Ge-detector)
- QEXAFS capability
- Gas-handling-system will integrated to serve catalysis community

Timeline:

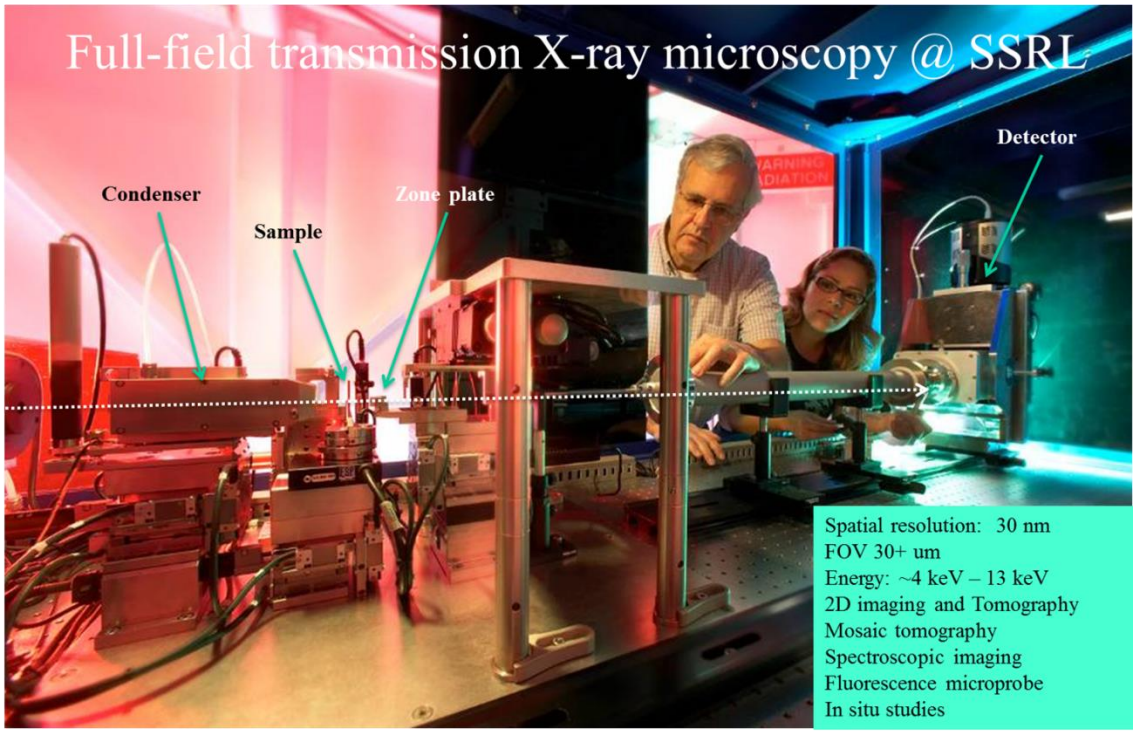
- Will start in September 2014
- Will be effective at least 2 years (third year will be an option)

How to access:

- Beamtime proposal through NSLS proposal system
- PS will provide with partner users supporting stuff at 2.2
- 80% of beamtime at 2.2 is available through PS proposal system
- Experts in catalysis, battery, and bio research will be available

Transmission X-ray Microscopy

- BL6-2, Multipole Wiggler beamline
- Xradia TXM
- Current operation shared with RIXS/Raman program
- New beam line for other RIXS/Raman under construction
- Full time TXM operation once RIXS/Raman moves

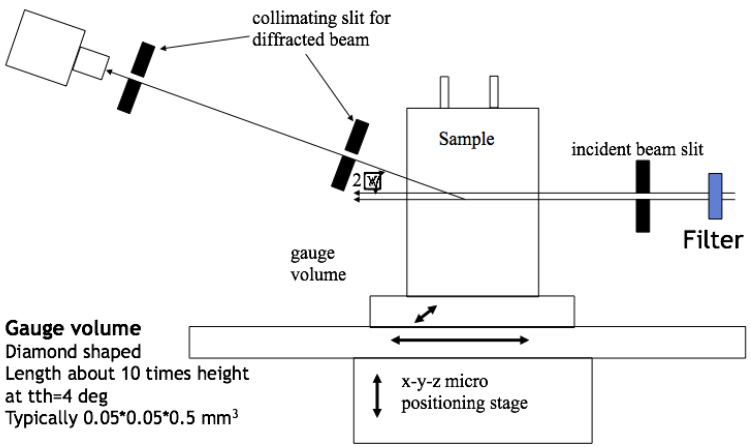


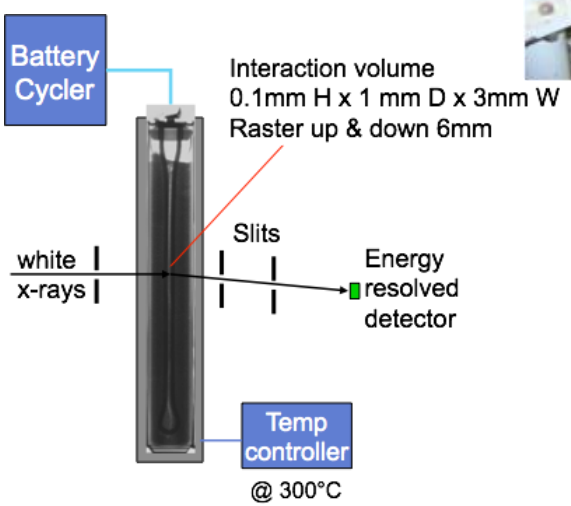
Working with SSRL on a MOU to accommodate NSLS TXM GU
APS has a new microscope ready for GU, a MOU is under discussion

Enhanced capabilities at X17B1 for battery research

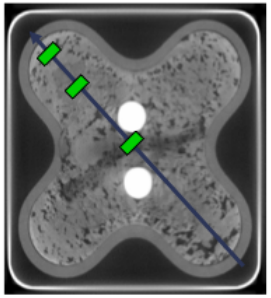
Ron Pindak, John Parise

NSLS X17B1 Experimental Setup



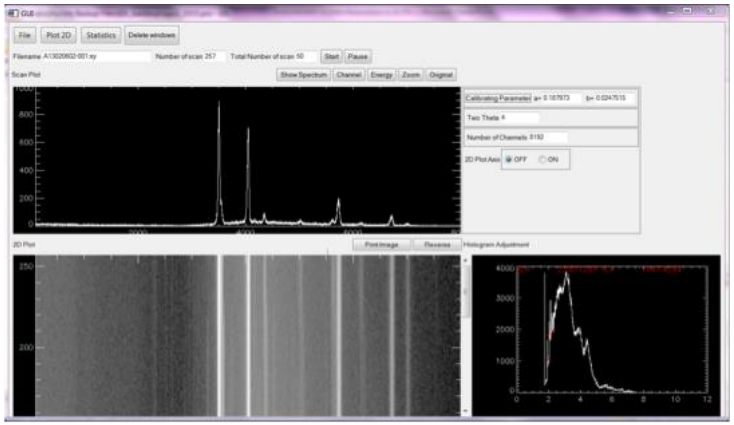


1) Diagonal scans



2) Single point

- Using DOE funding allocated to reduce the impact of the NSLS shut-down, beamline 6 BM at the APS was recently refurbished for user operations (6 BM includes 2 white beam experimental enclosures).
- The X17B1 Energy Dispersive X-Ray Diffraction (EDXRD) instrumentation will be transferred to 6 BM-A, following the NSLS shut-down and be commissioned during the remainder of 2014.
- In Jan. 2015 - General User operations start at 6 BM.
- Current plan: 50% of the 6 BM beam time will be available to general users and 10% set aside for the SBU-BNL-GE NY-BEST Battery Consortium .

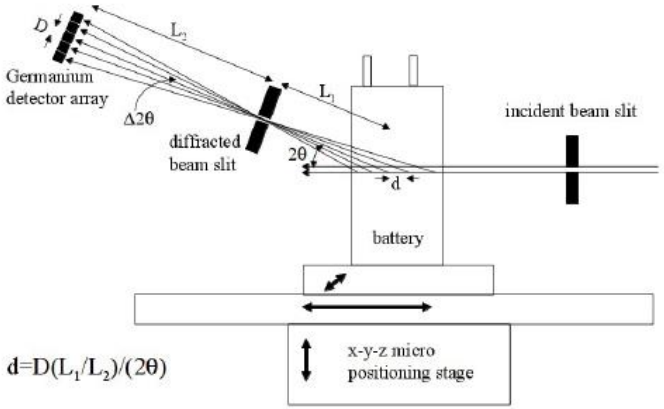


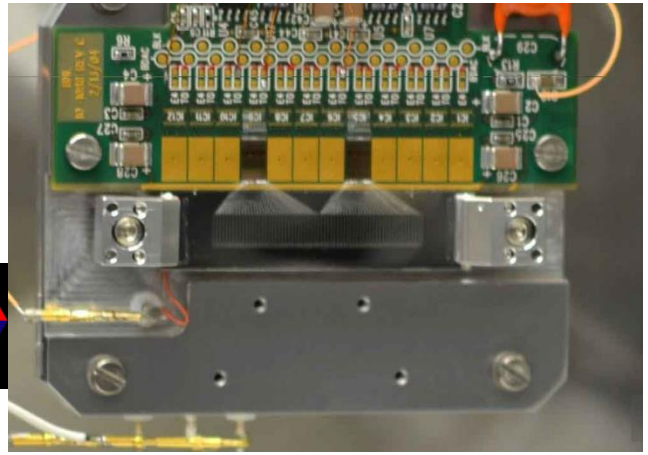
NY BES+ GE Global Research Stony Brook University nsls BROOKHAVEN NATIONAL LABORATORY

Plans for PS EDXRD in the "dark period'

Ron Pindak, John Parise

- Since 6 BM is a bend magnet source, there will be a factor of 5 reduction in flux at 6 BM compared to the flux obtained using the NSLS X17 wiggler source.
- This reduction can be more than offset through parallel detection of different gauge volumes using a pixelated germanium detector as shown to the right .
- A proto-type of this detector was constructed by the PS Detector Group with 64 (0.5mmx5mm) germanium sensor strips controlled by two 32-channel ASICs with pulse-height discriminators and pulse counters.





Proto-type germanium pixelated detector. New ASICs are underdevelopment to pulse height (energy) analyze the output from each sensor strip.

Complex Materials Scattering (CMS)

Masafumi Fukuto

- 3PW-based beamline for SAXS/WAXS and GISAXS/GIWAXS studies of bulk and interfacial structure of complex materials
- Next to two undulator-based beamlines serving “soft-matter” community:
  - Coherent Hard X-ray Scattering (CHX)
  - Soft Matter Interfaces (SMI)
- Goal: facilitate rational design of new functional materials, by unraveling the interrelations between constituents, processing, structure, and properties
- A variety of materials: liquids, small molecules, liquid crystals, polymers, biomolecules, nanoparticles, colloids, composites, porous materials, nanopatterned materials, … etc.
- Structural characterization at multiple lengthscales (sub-um to Å): particle sizes, size distributions, lattice structure, lattice spacings, correlation lengths, domain sizes
- Non-equilibrium, strongly dependent on assembly/processing pathway
- Part of NxtGen project; scheduled to begin commissioning in fall 2015.

Work with interested groups to write proposal together to other facilities

Protein Crystallography

Vivian Stojanoff

Continued support to MX industrial research.

- NSLS will cease operations September 30th, 2014

- Before normal operation can start at NSLS-II
  - members of our staff will assist MX users at other synchrotron facilities – depends on funding level
  - Proprietary research needs to have proper agreements in place
  - agreements with other synchrotron are being finalized – user will be prompted if former NSLS users
    - ✧ MOU with SSRL is ready to be sign by Facility Directors
      - ✓ will provide 50% of beam time on a bending magnet
      - ✓ insertion device time as needed on a fast access basis
    - ✧ MOU with ALS is still under discussion
      - ✓ will provide beam time at any of their beam lines
      - ✓ fractions are being under discussion

Protein Crystallography (cont'd)

Vivian Stojanoff

Continued support to MX industrial research.

- Before normal operation can start at NSLS-II (*cont'd*)
  - agreements with other synchrotrons are being finalized (*cont'd*)
    - ✧ MOU with APS discussions are being finalized
      - ✓ under consideration is sector 23ID
      - ✓ 50% of beam time on the bending magnet
      - ✓ insertion device time as needed on a fast access basis, following the sector policies
    - ✧ No special agreements with MacCHESS
- Collaborative research
  - will continue with the development of crystallization plates, robotics, automation
  - new opportunities
  - training

Issues

- Access policy
- Workhorse instrument – high throughput instruments, better data analysis support
- Engagement improvement

"Engaging its beamline scientists and support staff to provide greater assistance to industrial users solving critical challenges in development and deployment of clean energy technologies" ----recommendation from the DOE report

- Pre-experiment discussions/experimental planning,
- proposal writing assistance,
- Post-experiment data analysis,
- Experimental summary

Changing mode of access from PRT to GU, Need more support

Users want: not only get beamtime to perform experiments, more importantly, they bring in problems and get help and solutions

Summary

- Industrial research program is important at NSLS/NSLS-II
- Industrial research enhancement program has been successful by increasing:
  - visibility of industrial research
  - beamtime for industrial research
  - new capabilities
  - collaborations
  - outreach to industrial user community
- Industrial application is one of strategic directions at BNL
  - considerable coordination exists at lab
  - strong regional interest and participation
- One of strategic directions at DOE Office of Basic Energy Sciece
  Bridging gap between practical problems and advanced tools and discoveries

Open discussions tomorrow is critical to collect comments and thoughts

Thank you