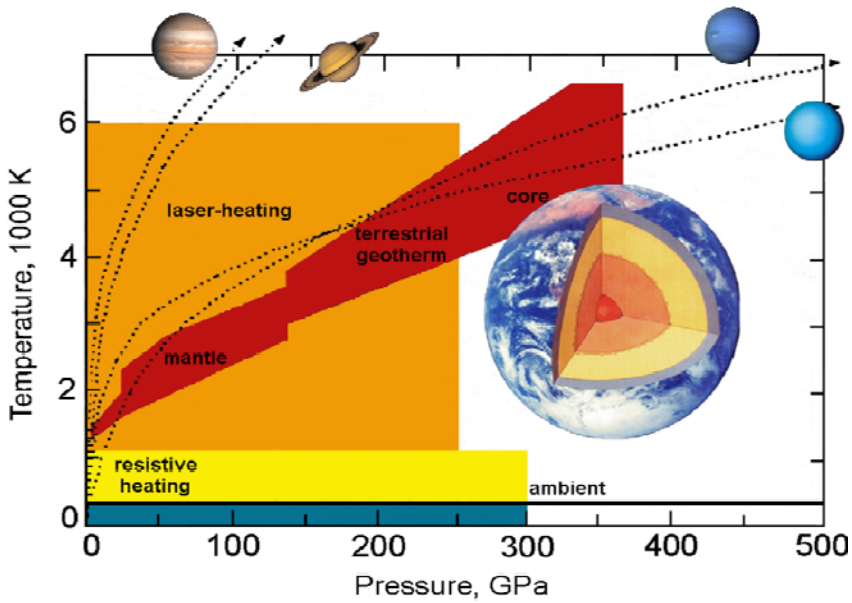
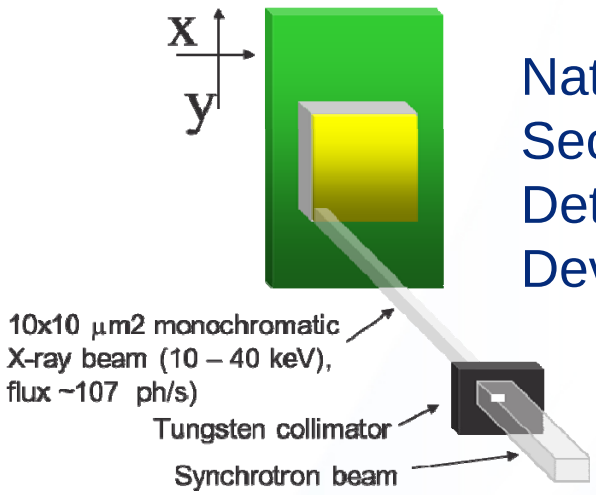
Materials Science and Engineering - Extreme Environment Materials

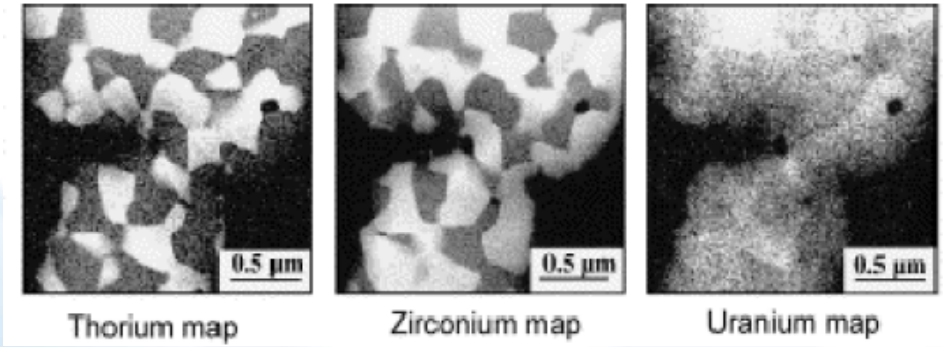
Lynne E. Ecker



High Pressure Community SBU/BNL

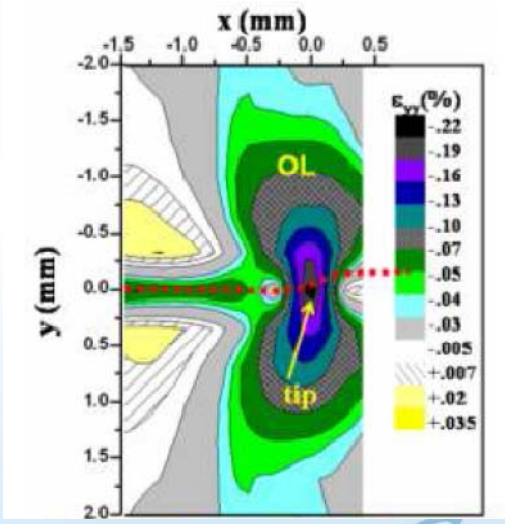


National Security/ Detector Development



Nuclear Energy

Applied Engineering SBU/BNL



Required Capabilities

High Pressure Community

- Broad user base in earth sciences (COMPRESS), materials science, physics and chemistry (X17B2, B3, C, and U2A)
- Funding needed for dedicated high-pressure beamlines

National Security/Detector Development

- Small user community, but require 75% of beamtime (X27B)
- Collaborate with another high-energy beamline

Nuclear Energy

- Large user base at other facilities (SSRL, APS, Soleil) (X17B)
- New facility for activated materials and ion beam irradiation proposed

Applied Engineering

- Large industrial user base; energy and structural engineering applications (X17A&B)
- Engineering scale experiments

Future Beamlines

Project Beamline: X-Ray Powder Diffraction (XPD)

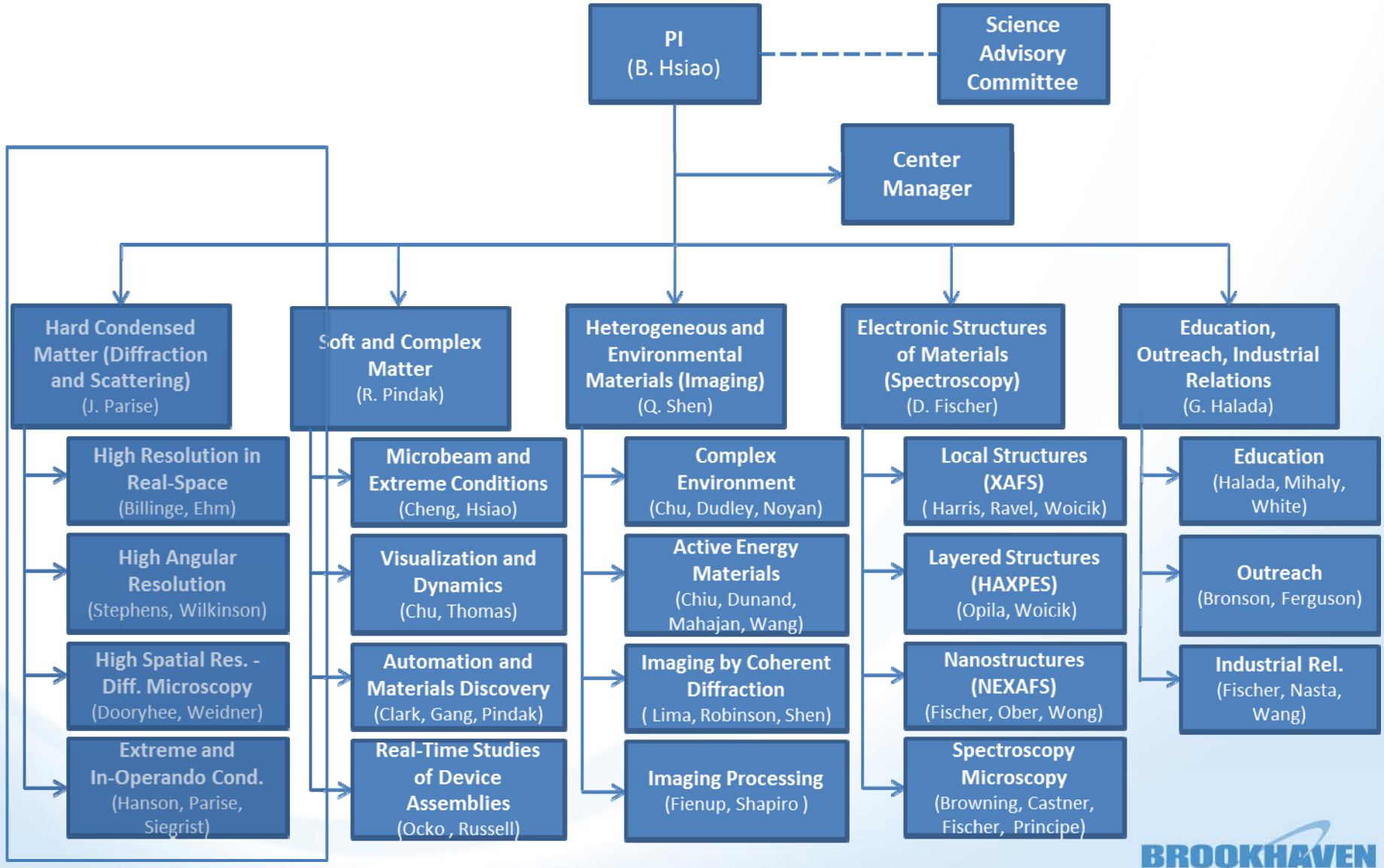
Approved Beamlines

- 4-Dimensional Studies in Extreme Environments (4DE)
- Time-resolved X-ray Diffraction and Spectroscopy Under Extreme Conditions (TEC)
- Frontier Synchrotron Infrared Spectroscopy Beamline Under Extreme Conditions (FIS)

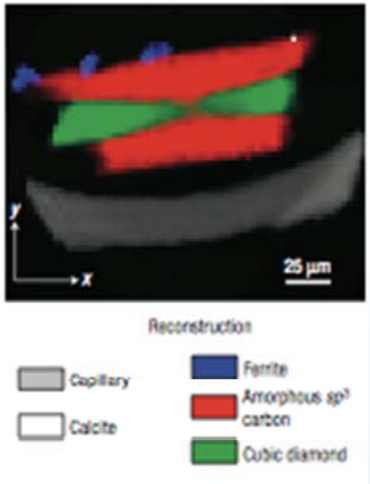
Proposed Beamlines

- Real Time and In Situ Studies of Materials in a Radiation Environment (MRE)
- High-energy X-ray Micro-mapping of Materials for Advanced Energy and Structural Engineering Applications Beamline (HEX)
- Endstation for High-Energy Beamline for High Z Radiation Detector Materials (Partner Beamline)

Center for Advanced Photon Science and Technology (CAPST)



Hard Condensed Matter at Extremes



- Rationale
  - Transformational materials/operating devices
  - Concrete oil well liners, batteries, catalysts
    - Often heterogeneous – appropriate length scale
    - Why do they work – or FAIL – under extremes?
    - Look at the whole process rather than component

Needs and way forward

- Integrated capability for
  - high Q-space (> 30 $Å^{-1}$),
  - high angular (□<0.1°),
  - high spatial (nm-µm)
- Combinations, with diffraction imaging, at extremes of PT, B, E, radiation etc
- Community driven SCIENCE initiatives through workshops to identify the most important science and NSSL-II resources to solve them
- Partner with NSLS to identify opportunities
  - Proximity of SBU-BNL-NSLS-NSLS-II possibly an unprecedented opportunity

4 Dimensional Extreme Environment ($4DE^2$)

$4DE^2$ at NSLS-II

- Contain unique world-leading high pressure devices (200 MPa- 1000GPa)
- In-situ studies of materials under extreme conditions with both static and dynamic capability
- The continuous growth of high pressure devices combined with high brightness/low noise NSLS-II will set new frontiers in high pressure research

Examples of Science Areas & Impact

- FUNCTIONAL MATERIALS: super-hard material; complex structured alloy; highly correlated electron systems; structural material
- EARTH AND PLANETS: transport properties of rocks; dynamics of grain interaction; phase transitions; kinetics, failure strength
- GAS-FLUID-SOLID INTERACTION: waste/CO_2 sequestration; gas hydrates deposition; porous materials with micron-nanopores)

High pressure devices, Left: model for 2000 ton press (up to 30 GPa, 2000K) with dynamic capability. Right: Diamond anvil cell (1000 GPa).

Beamline Capabilities

TECHNIQUE(S): powder diffraction and imaging with energy dispersive/monochromatic beam

SOURCE: Superconducting wiggler

ENERGY RANGE/RESOLUTION: 20–100 keV / 100 eV

SPATIAL RESOLUTION: ~1 – 5 μm

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Spokesperson: Donald J. Weidner, Stony Brook University

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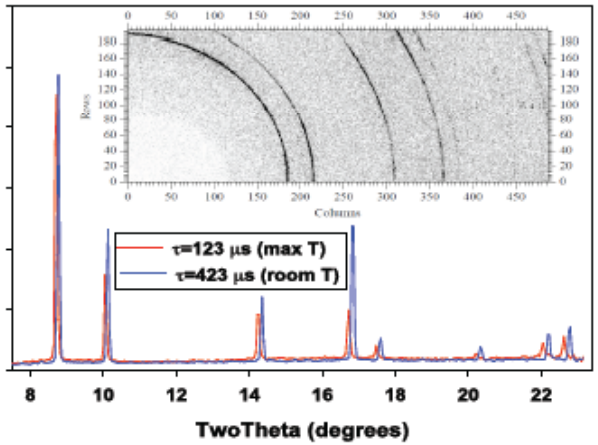
Time-resolved X-ray diffraction and spectroscopy under extreme conditions (TEC)

TEC at NSLS-II

- Will enable time-domain capabilities (femto to milli seconds) for combined x-ray and pulsed laser diagnostics under extreme conditions of pressure (static & dynamic), temperature (0.001 eV to 1 eV), and magnetic field (dc fields to 35 T and pulsed to 50 T) to study ultrafast physical, chemical, and transport phenomena
- Will enable high spatial resolution to study *in-situ* dynamic processes and transport properties at submicron scale
- Tightly focused, brilliant beams will make TEC the world's leading beamline for extreme conditions research

Examples of Science Areas & Impact

- MATERIALS SCIENCE: Material response to extremes, synthesis of new materials with unique properties (nano, superconducting, superhard) in nonequilibrium conditions
- CHEMISTRY: Energetic materials under detonation conditions, bonding character changes under extreme conditions
- GEO- & PLANETARY SCIENCES: Origin, evolution, and interior structure of planets through *in-situ* direct study of transport and melting phenomena, equations of state (including fluids), fluid-fluid transitions, electronic and spin transitions, change in redox and valence states, chemical reactivity, mineral interfaces
- ENVIRONMENTAL SCIENCE: Carbon Sequestration



Time-domain laser heating x-ray diffraction experiments in platinum at 40 GPa (APS, GSECARS). Blue line – the pattern measured (with 1 μs time resolution) at the delay time well outside the laser pulse duration. Red solid line- the pattern collected at the delay time corresponding to the largest thermal expansion and hence the largest bulk sample temperature (3000 K). The inset shows the diffraction image corresponding to the pattern measured from the hot sample (red curve). A. Goncharov et al., *Rev. Sci. Instrum.*, in press (2010).

Beamline Capabilities:

TECHNIQUES: micro-XRD and SAXS, XAS, XFS, XES, x-ray Raman

SOURCE: high beta undulator

ENERGY RANGE / TIME RESOLUTION: 5-20 keV (up to 40 keV) / down to10 ps, 1 μs typical

SPATIAL RESOLUTION: 0.2 – 5 μm

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Spokesperson: Alexander Goncharov, Carnegie Institute

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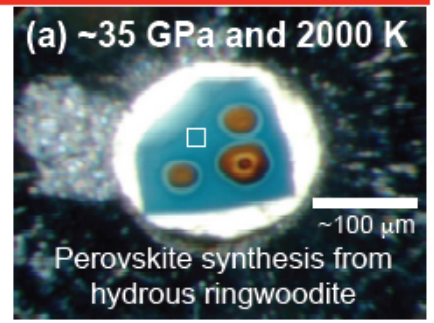
Frontier Synchrotron Infrared Spectroscopy Beamline under Extreme Conditions (FIS)

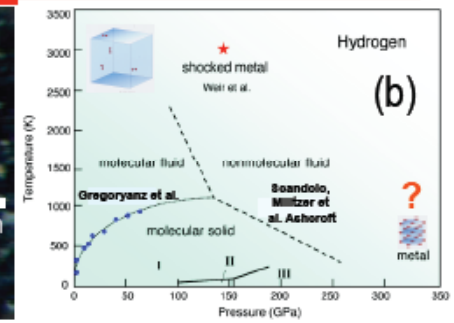
FIS at NSLS-II

- Will enable in-situ optical studies of a wide variety of materials by spectroscopic techniques at extreme P-T conditions (to several hundred GPa and 4~6000 K)
- Measurements of far-infrared to visible spectra with diffraction-limited spatial resolution
- The combination of the high brightness and low noise of NSLS-II with dedicated high-pressure facilities will be unique and world leading

Examples of Science Areas & Impact

- EARTH AND PLANETARY SCIENCES: mimic the mantle extreme conditions and study the Earth's deep water cycle
- MATERIAL SCIENCES: study metallization of hydrogen and hydrogen-rich materials under extreme conditions
- DEEP CARBON CYCLE RESEARCH: study behavior of carbon-bearing materials in Earth's deep interior conditions by vibrational spectroscopy
- DYNAMIC COMPRESSION: probe material behavior on short time scales combined with the pulsed synchrotron radiation





(a) Synthesized perovskite (yellow spots) from hydrous ringwoodite in the sample chamber of a laser heated diamond anvil cell (DAC) (Reid at al., AGU fall meeting, 2006) and (b) hydrogen phase diagram. FIS will enable *in situ* high P-T optical studies of various hydrous minerals as well as hydrogen metallization at extreme high P-T conditions due to the brighter and exceptionally stable infrared source at NSLS-II.

Beamline Capabilities

TECHNIQUE(S): Fourier transform infrared spectroscopy; diamond anvil cell techniques for static high pressure; gas-gun launchers for dynamic compression; cryogenic techniques combined with DACs; laser heating techniques combined with DACs

SOURCE: large-gap (90 mm) dipole magnets

ENERGY RANGE / RESOLUTION: 50 – 10000 cm^{-1} / 1 cm^{-1}

SPATIAL RESOLUTION: diffraction limited resolution in the entire energy range

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Spokesperson: Zhenxian Liu, Carnegie Institution of Washington

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BROOKHAVEN SCIENCE ASSOCIATES

High energy Engineering X-ray Characterization (HEX)

HEX at NSLS-II

- Core technologies: energy, structural…
- Deeply penetrating 30-200 keV x-rays
- Real materials, real conditions, in real time
- Phase & strain mapping in space & time

Examples of Science Areas & Impact

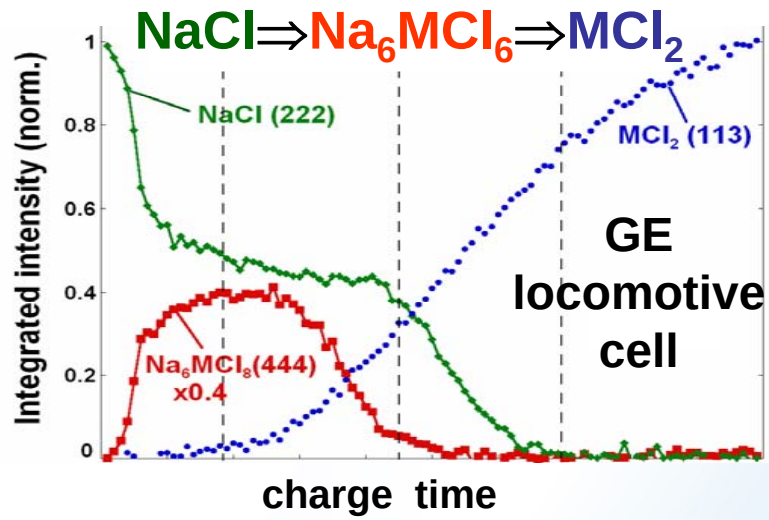
- Real battery in situ electrochemistry
  time &space phase mapping
- Fatigue & fracture mechanics
  in situ loading strain mapping
- Processing & composite materials
  laser/shot peening, ceramic on metal coatings…
- Nuclear energy materials

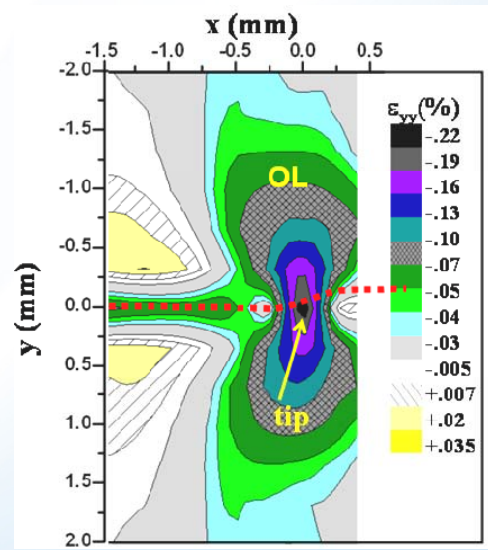
Beamline Capabilities

TECHNIQUE(S): Diffraction, energy dispersive,
& monochromatic area detection;
small angle x-ray scattering; PDF; high-energy tomography

SOURCE: Superconducting wiggler 30-200 keV

SPATIAL RESOLUTION: laterally to below 1μm





Fatigue crack tip strain field

Brookhaven Science Associates

BROOKHAVEN NATIONAL LABORATORY

Spokesperson: Mark Croft, Rutgers University

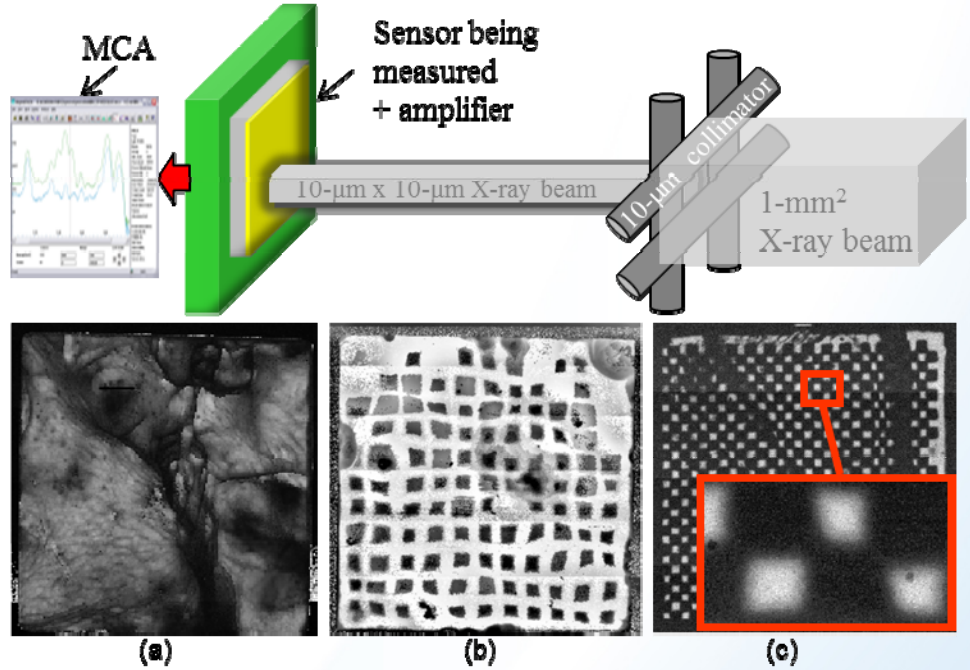
Micron-scale Detector Mapping for X-ray Sensors (MDM)

MDM at NSLSII

- Will provide spatially-resolved characterization of the performance of an X-ray sensor, which is crucial to understand the effects of dislocations and other extended defects on the device performance
- Will pin down the driving forces for the non-uniformities of the E-field observed for solid-state X-ray detectors
- Study of polarization effect for high flux detectors used in medical applications
- Establish room-temperature detectors with energy resolution comparable to cryogenically cooled HPGe detectors
- Answer the key questions to obtain large area/volume X-ray detectors

Examples of Science Areas & Impact

- Better energy resolution (< 0.5% FWHM at 662 keV) detectors for synchrotron, space, environmental, energy, and homeland security applications
- Better spatial resolution for imaging detectors for numerous medical and synchrotron uses
- Motivate the new design of low power readout electronics for X-ray detectors
- Benefit the crystal growers to achieve large volume ingots for X-ray and gamma applications



Schematic diagram of the micron-scale detector mapping for X-ray sensors. A monochromatic X-ray micro-beam is used to map the whole area of the sample, and for each micro-area interrogated the energy spectrum is acquired, stored, and subsequently used to plot an X-ray response map. Three X-ray response maps are shown for three different CdZnTe (CZT) detectors of area 20 x 20 x 2 mm³; (a) hole collection map of a planar CZT detector from Redlen Technologies, which shows subgrain boundaries, tellurium inclusions and clusters of dislocations; (b) electron collection map of a 121-pixel anode CZT detector from eV Products, the dislocations cause non-uniformities of the E-field; (c) electron collection map of a CZT detector from Caltech for the NuSTAR astrophysics mission: subgrain boundaries and E-field non-uniformities cause a distortion and incomplete charge collection.

Beamline Capabilities

TECHNIQUE: µm-beam pulse height spectra measurements
SOURCE: damping wiggler
ENERGY RANGE: 10 keV – 90 keV
SPATIAL RESOLUTION: 1 µm – 10 µm variable

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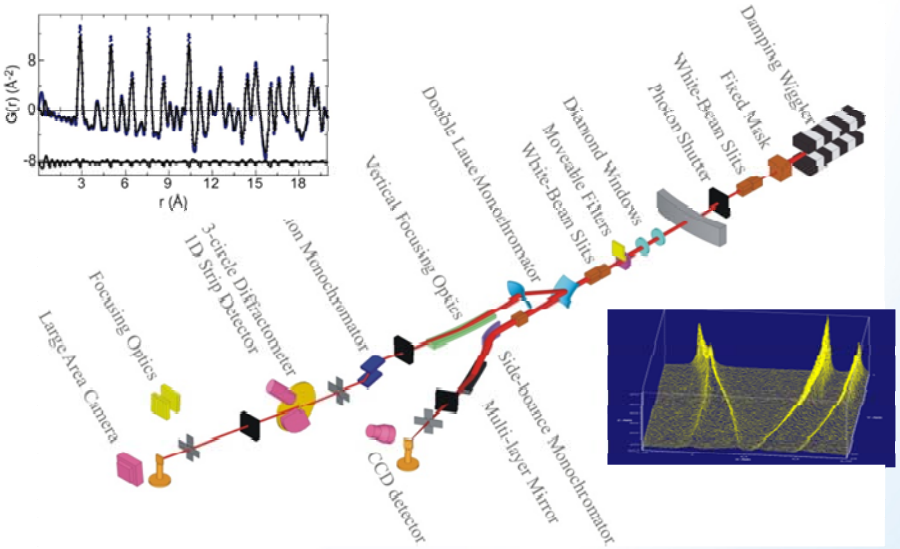
X-Ray Powder Diffraction (XPD)

XPD at NSLS-II:

- powder diffraction of complex materials using high energy x-rays either in high photon flux mode or high angle resolution mode.
- *in situ* and *in operando* structural science (variety of sample environments, materials in working conditions or in operating devices).
- dynamics, rapid acquisition, time-resolved (msec to sec scale).
- total structure diffraction studies: high Q-space resolution (average, long-range structure) combined with high real-space resolution (pair distribution function: local structure or nanostructure).

Examples of Science Areas & Impact:

- STRUCTURAL AND IN SITU MATERIALS SCIENCE: high T synthesis, structural ceramic composites, thermal barrier coatings…
- MATERIALS CHEMISTRY AND ENERGY SCIENCE: hydrogen storage, fuel cells, catalysis, cements, batteries, porous materials…
- EARTH AND PLANETARY SCIENCES: ices and clathrates, new phases or structurally modified earth materials…
- ENVIRONMENTAL SCIENCE: sequestration of pollutants, sorption of contaminants, CO_2 sequestration, speciation in solution…
- CONDENSED MATTER PHYSICS: magneto-resistance, charge ordering, magnetic frustration, superconductors, Negative Thermal Expansion materials, multiferroic materials…
- MATERIALS ENGINEERING: materials under irradiation, stress-strain,…
- ORGANIC AND BIO-MATERIALS SCIENCE: pharmaceuticals, protein cores and capsids…



The layout of XPD features two independent branches served by Laue-crystal monochromators and three in-line endstations fitted with 0, 1 and 2D detectors. XPD is capable of collecting data at high energies ideal for high-Q data and in situ and time resolved studies in environmental cells.

Beamline Capabilities:

TECHNIQUE(S): powder diffraction, total scattering, Medium angle scattering, tomography

Source: 1.8T Damping Wiggler

ENERGY RANGE : 30 -80 keV

ENERGY RESOLUTION: 10^{-3} to 2×10^{-4} $\Delta E/E$

SPATIAL RESOLUTION: ~mm horizontal beam (powder averaging) down to ~ 50 μm

TIME RESOLUTION: ~ms

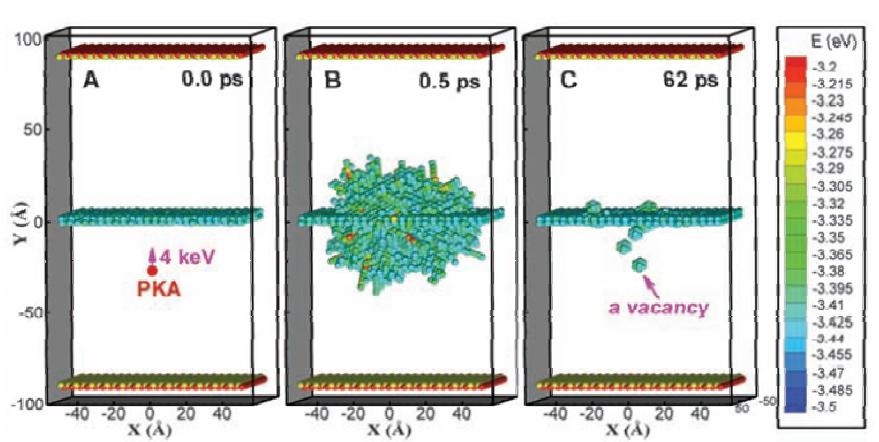
Real time and in-situ studies of Materials in a Radiation Environment (MRE)

MRE at NSLS II

- Ion accelerators for in situ experiments of radiation effects and mesoscale microstructural changes due t radiation
- Separate endstation for radioactive materials
- Will enable studies of new materials for reactors, nuclear fuels and structural materials in high radiatio environments
- Data for verification of computer simulations, materia performance during off-nominal conditions, and licensing

Examples of Science Areas & Impact

- NUCLEAR STRUCTURAL MATERIALS: Role of interfaces in radiation resistant materials
- NUCLEAR FUELS: Characterization of spent fuel, metal fuels, and new synthesis routes for oxide fuels
- NATIONAL SECURITY: material characterization for nuclear forensics
- SEMICONDUCTOR FABRICATION: Understand the effect of doping induced defects on semiconductor performance



Displacement Cascade Near a Grain Boundary
Bal, et al. Science, 2010

Beamline Capabilities
Techniques: Diffraction, Imaging, Spectroscopy
Source: Damping or superconducting wiggler
Energy range: 10-90 keV
Time resolution: msec-μsec
Beam Size: > 1 μm

Discussion Session

Lynne Ecker, lecker@bnl.gov, x2538

Goal: Identify strategies to develop and enhance research programs at BNL and SBU that will benefit from NSLS-II's capabilities

Identify high impact, field changing experiments that can be preformed early at NSLS II and will brand the facility

- Interactions of defects with interfaces (key to developing new radiation resistant structural materials)
- Plutonium alloy phase diagram (key to fast reactor fuels)
- Actinide speciation during fuel fabrication (key to new fuels)

What is the expected usage of the currently planned beamlines by the existing research programs and personnel at SBU and BNL?

- High-pressure group may work with XPD to put high pressure equipment into hutch-D, but it will be underserved until a dedicated high-pressure beamline is operational

Are there opportunities to enhance BNL/SBU representation in the areas of interest among the existing six NSLS-II beam-line advisory teams?

Is there a need to propose additional beam-lines (beyond those already approved) to provide additional capabilities needed to meet the current and emerging challenges identified by the BNL and SBU community?

What other infrastructure is needed at either BNL or SBU (besides NSLS-II) to attract and support the S&T communities that will utilize NSLS-II.

- Satellite building for easier transportation and handling of active samples
- Radioactive Materials Storage Area in another building to avoid exceeding activity limits at NSLS II
- Sample preparation area in another building for loading low level samples into containment (glove box)
- Ability to accommodate different sample environments at the beamline

- Satellite building for easier transportation and accommodation of engineering scale experiments and components

- Common indexing capability between beamlines

What are the areas of strength for personnel in the existing research communities at BNL/SBU and what are the most pressing needs for additional expertise or personnel in order to mount leading research initiatives utilizing NSLS-II?

- Material scientist with extensive experience working in a facility that handles active samples

What are the most effective strategies for strengthening the BNL/SBU research community (e.g., strategic hires, partnerships, etc.), including leveraging industrial collaborations, especially in NYS?

- The Ion Beam Laboratory, SUNY Albany

What are the funding opportunities to foster and support innovative, high-risk-high-reward R&D that benefits from utilization of NSLS-II at BNL/SBU?

- DOE-Nuclear Energy (NE)
- Nuclear Regulatory Commission (NRC)
- Commercial Nuclear Industry

How do we nurture new communities and/or new investments by these communities in research at NSLS-II?

- Nuclear user community requires support with safety procedures, containment options and paperwork
- Formal relationship with facilities at other national laboratories that produce test specimens such as ATR at INL and MaRIE at LANL
- May require longer beamtime if containment procedures are time consuming

Comments on new aspects of the User Access Policy