National Synchrotron Light Source II

OVERVIEW

BROOKHAVEN NATIONAL LABORATORY

NATIONAL SYNCHROTRON LIGHT SOURCE II facility information

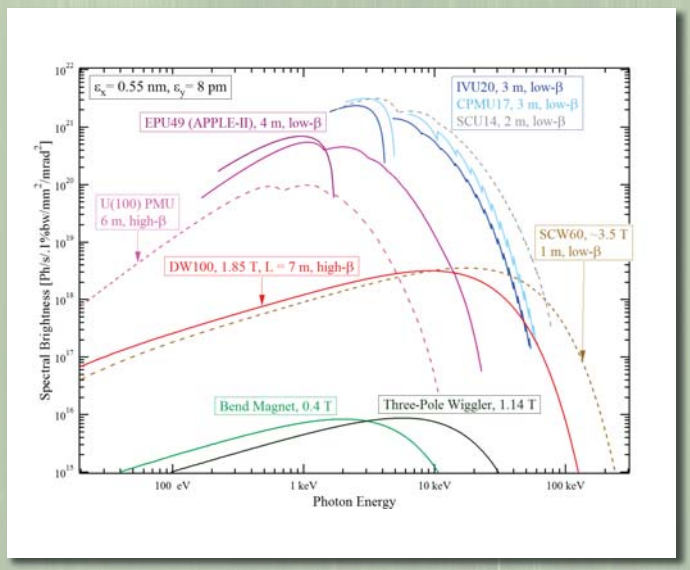
Project Scope

Accelerator Systems
- Storage Ring (~ ½ mile in circumference)
- Linac and Booster Injection System

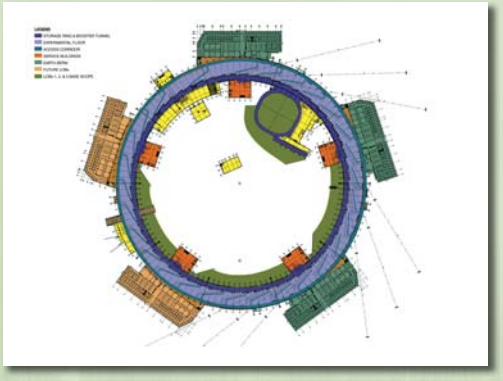
Conventional Facilities
- Ring Building and Service Buildings (~ 400,000 gsf)
- 5 Laboratory/Office Buildings (LOBs) for beamline staff & users
  - 2 full & 1 shell (114,000 gsf) in base scope, 2 more (76,000 gsf) in future scope
- Reuse of existing NSLS office/lab space for NSLS-II staff

Experimental Facilities
- Initial suite of six insertion device beamlines
- Capable of hosting at least 58 beamlines

Research & Development
- Advanced optics & accelerator components



Photon Sources and Beamlines
- World-leading high brightness based on suite of insertion devices
- 10,000 times brighter than best existing sources at NSLS
- Broad spectrum of photon sources from infrared to hard x-rays
- Photon beamlines taking advantage of these source properties



NSLS-II Design Features

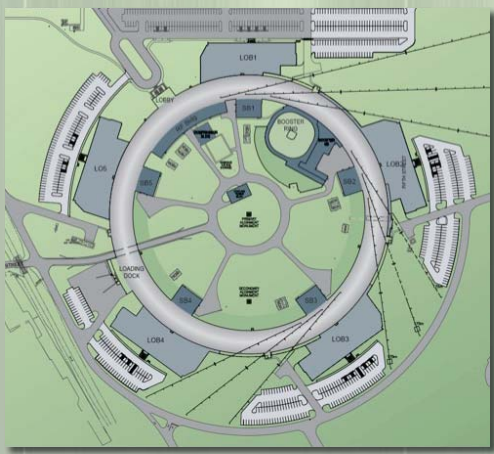
Design Parameters
- 3 GeV, 500 mA, top-off injection
- Circumference 791.5 m
- 30 cell, Double Bend Achromat
  - 15 high-β straights (9.3 m)
  - 15 low-β straights (6.6 m)

Novel Design Features
- Damping wigglers
- Soft bend magnets
- Three pole wigglers
- Large gap IR dipoles

Ultra-low Emittance
- ε_x, ε_y = 0.6, 0.008 nm-rad
- Diffraction limited in vertical at 12 keV
- Small beam size: σ_y = 2.6 μm, σ_x = 28 μm, σ'_y = 3.2 μrad, σ'_x = 19 μrad

Pulse Length (rms) ~ 15 psec

Experimental Floor Stability
- Storage ring temperature ± 0.1 °C
- Experimental floor temperature ± 1 °C
- Floor vibration (rms) < 25 μm



NSLS-II Site Plan

U.S. DEPARTMENT OF ENERGY

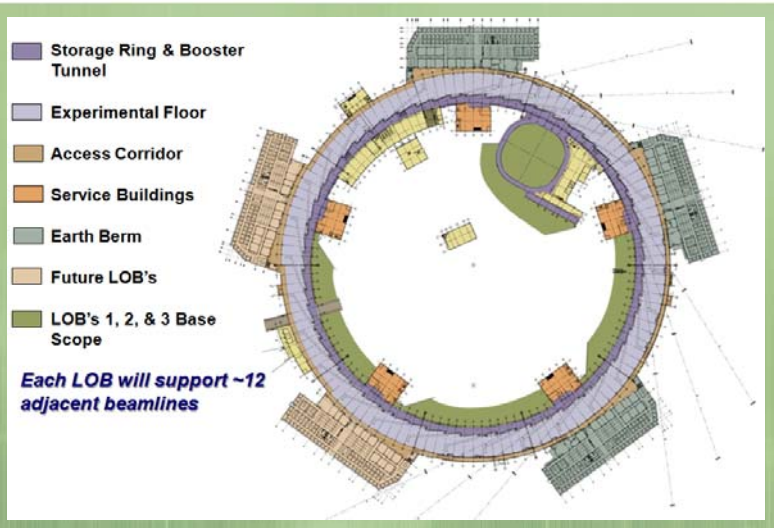
NSLS-II
LABORATORY-OFFICE BUILDINGS

BROOKHAVEN NATIONAL LABORATORY

NATIONAL SYNCHROTRON LIGHT SOURCE II facility information

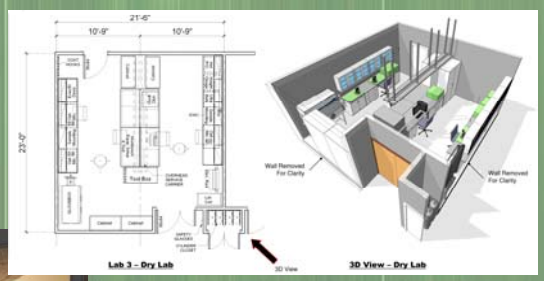
LOB DESIGN OBJECTIVES

- Promote interaction of staff & users
- Provide varied & flexible office space
- Provide flexible shared labs for range of uses
- Provide beamline support infrastructure
- User space to be flexible open-plan offices



LOB CHARACTERISTICS

- LOBs being constructed by E.W. Howell
- 11 labs and workshops:
  - ✓3 wet labs
  - ✓2 dry labs
  - ✓1 special-purpose sample characterization lab
  - ✓1 mechanical assembly lab
  - ✓1 vacuum prep lab
  - ✓2 electronic work areas
  - ✓1 machine shop
- Single- and multi-person offices for scientists, and open desk areas for users and postdocs
- 2 kitchens, 2 conference rooms, 3 interaction areas
- Loading dock for three LOBs
- Seating for approximately 124 staff and users
- Open airy building with natural lighting
- 38,000 sq. ft.; LEED-certified silver (possibly gold)
- Bathrooms with lockers and showers



Guide to NSLS-II Project, NEXT, NIH, Type 2 and Approved Beamlines

	Acronym	Title	Spokesperson	Technique
Project	CHX	Coherent Hard X-Ray Beamline	Andre Fluerasu, BNL	Coherent diffraction, X-ray Photon Correlation Spectroscopy
	CSX	Coherent Soft X-ray Scattering and Polarization	Cecilia Sánchez-Hanke, BNL	Coherent x-ray scattering / x-ray diffraction microscopy, Polarization dependent spectroscopy / scattering, Ultrafast dynamics, X-ray photon correlation spectroscopy
	HXN	Hard X-ray Nanoprobe	Yong Chu, BNL	Nano-diffraction and fluorescence, differential phase contrast (DPC) and coherent diffraction imaging (CDI)
	IXS	Inelastic X-ray Scattering	Yong Cai, BNL	High resolution (1 meV) inelastic x-ray scattering. Deep inelastic x-ray scattering at high momentum transfers
	SRX	Submicron Resolution X-ray Spectroscopy	Juergen Thieme, BNL	X-ray fluorescence trace element analysis and imaging, XANES, X-ray microdiffraction
	XPD	X-ray Powder Diffraction	Eric Dooryhee, BNL	Diffraction of polycrystalline materials; pair distribution function analysis; diffraction tomography
NEXT	ESM	Electron Spectro-microscopy for Fundamental Studies of the Physics and Chemistry of Materials	Elio Vescovo, BNL	nano-ARPES, SP-ARPES, AP-SPEM, XPEEM
	FXI	A Superconducting Wiggler Long Beamline for Full-field Imaging at NSLS-II	Jake Socha, Virginia Tech	full-field imaging; transmission x-ray microscopy (TXM)
	ISR	Integrated In-situ and Resonant Hard X-ray Studies	Karl Ludwig, Boston University	RXS; Polarized XRD; GID; CTR; TXRF; GISAXS; in-situ XPCS; in-situ CDI; in-situ XRIM
	ISS	Inner-shell Spectroscopy	Bruce Ravel, NIST	X-ray absorption spectroscopy, X-ray emission spectroscopy, X-ray energy-loss spectroscopy
	SIX	Soft Inelastic X-ray Scattering	Kevin Smith, Boston University	Resonant inelastic x-ray scattering
	SMI	Soft-Matter Interfaces	Ben Ocko, BNL	X-ray reflectivity, grazing incidence X-ray scattering, and anomalous/resonance techniques
NIH	AMX	Flexible Access Macromolecular Crystallography at an Undulator Beamline	Dieter Schneider, BNL	Macromolecular crystallography
	FMX	Frontier Macromolecular Crystallography at an Undulator Beamline	Robert Sweet, BNL	Macromolecular crystallography
	LIX	A High-brightness X-ray Scattering Instrument for Biological Applications	Lin Yang, BNL	Micro-beam, simultaneous small and wide angle x-ray, transmission and grazing incidence
Type 2	BMM	Hard X-ray Absorption Spectroscopy and Diffraction - Beamline for Materials Measurements	Daniel Fischer, NIST	x-ray absorption fine structure (XAFS) and x-ray diffraction (XRD)
	NYX*	NYSBC Microdiffraction Beamline	Wayne Hendrickson, Columbia University	Macromolecular crystallography
	SST	Soft and Tender X-ray Spectroscopy and Microscopy	Daniel Fischer, NIST	6 unique world class NEXAFS/XPS experimental stations (2 full field microscopes, 2 automated high-throughput, and 2 in-situ high pressure)
Approved	4DE	4-Dimensional Studies in Extreme Environments	Donald J. Weidner, Stony Brook University	Powder diffraction and imaging with energy dispersive/ monochromatic beam
	ABS	A Highly Automated Instrument for Static X-ray Scattering Measurements of Biological Molecules in Solution	Lin Yang, BNL	Simultaneous small and wide angle X-ray solution scattering
	AIM	Advanced Infrared Microspectroscopy	Lisa Miller, BNL	Fourier transform infrared microspectroscopy (FTIRM)
	CDI	Coherent X-ray Diffraction	Ian Robinson, University College London	Coherent Diffraction Imaging
	CMS	Complex Materials Scattering	Kevin Yager, BNL	Wide-angle, small-angle, and ultrasmallangle x-ray scattering (including reflection-mode)
	FIS	Frontier Synchrotron Infrared Spectroscopy Beamline Under Extreme Conditions	Zhenxian Liu, Carnegie Institution of Washington	Fourier transform infrared spectroscopy; diamond anvil cell techniques for static high pressure
	HIX	Hard X-ray - Inelastic X-ray Scattering	Young-June Kim, University of Toronto	Resonant and non-resonant inelastic x-ray scattering; x-ray RamansScattering
	IRI	Full-field Infrared Spectroscopic Imaging	Lisa Miller, BNL	Fourier transform infrared spectroscopic imaging with a 64x64 focal plane array detector
	IXD	Powder Diffraction Beamline for In-situ Studies of Structural and Chemical Transformations	Jianming Bai, University of Tennessee	In-situ powder diffraction studies under high temperature, high pressure, reaction gas flow and charge-discharge cycles
	MET	Infrared and THz beamline for Magnetospectroscopy, Ellipsometry and Dynamical Studies	Larry Carr, BNL	Infrared transmission, reflection and ellipsometric spectroscopies; cryo- and magnetospectroscopy to 10T, pump-probe to ~10ps resolution
	MID	Dedicated Beamline for X-ray Instrumentation Development	Jeff Keister, BNL	White and monochromatic (focused & unfocused), soft and hard x-ray optics, and multiple optical benches for flexible test arrangements
	MPP	Materials Physics and Processing	Jean Jordan-Sweet, IBM	In-situ rapid thermal annealing (RTA) and isothermals to 1000°C, X-ray scattering in a magnetic field, GIXD, HRXRD, XRR, pole figures, etc.
	MXD	Multiscale X-ray Diffraction	Kenneth Evans-Lutterodt, BNL	Primarily diffraction- centric detectors, but secondary detectors for fluorescence
	OFT*	At-wavelength Metrology and In-situ Figuring	Konstantine Kaznatcheev, BNL	X-ray interferometry (point shearing (soft xray) and Talbot-type (hard x-ray)) and wave front sensing (Hartmann detector); x-ray reflectivity and scattering; white and monochromatic x-ray topography and high resolution diffractometry
	QAS	Beamline for In-situ Studies of Chemical Transformations by Combined, Synchronous, "Quick" X-ray Absorption and Scattering Measurements	Anatoly Frenkel, Yeshiva University	X-ray absorption spectroscopy and X-ray diffraction with 10 ms time resolution
	SM3	Correlated Single-crystal Spectroscopy and Macromolecular Crystallography	Allen M. Orville, BNL	Macromolecular crystallography, spectroscopy on- and off-beamline (UV/vis, fluorescence, IR, Raman, XAS and EXAFS)
	SSS*	Soft X-Ray Spectroscopy and Scattering	Joseph Dvorak, BNL	Soft X-ray linear and magnetic scattering and spectroscopy in fields up to 10T, as well as ambient pressure fluorescence spectroscopy
	TEC	Time-resolved X-ray Diffraction and Spectroscopy Under Extreme Conditions	Alexander Goncharov, Carnegie Institute	Micro-XRD and SAXS, XAS, XFS, XES, x-ray Raman
	TES	Tender Energy X-ray Spectroscopy	Paul Northrup, Stony Brook University	X-ray fluorescence and spectroscopic imaging, high-performance and in-situ EXAFS
	XAS	X-ray Absorption Spectroscopy for Biological, Environmental and Energy Sciences	Mark Chance, Case Center for Synchrotron Biosciences	Transmission and fluorescence mode extended x-ray absorption fine structure (EXAFS) & x-ray absorption near-edge structure (XANES)
	XFM	X-ray Fluorescence Microprobe	Antonio Lanzirotti, University of Chicago	µm x-ray fluorescence (XRF), x-ray absorption fine structure (XAFS) spectroscopy, x-ray diffraction (XRD) and fluorescence computed microtomography (FCMT)
	XFP	X-ray Footprinting	Mark Chance, Case Center for Synchrotron Biosciences	Steady state and time-resolved x-ray hydroxylradical mediated protein and nucleic acid footprinting

* Not appearing in poster session

COHERENT HARD X-RAY BEAMLINE (CHX)

BROOKHAVEN NATIONAL LABORATORY

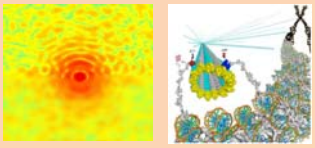
Beamline Development Group: A. Fluerasu, L. Wiegart, M. Carlucci-Dayton, L. Berman (BNL)
Beamline Advisory Team: Robert Leheny (JHU) – Chair; Karl Ludwig (BU); Laurence Lurio (NIU); Simon Mochrie (Yale); Lois Pollack (Cornell); Aymeric Robert (SLAC); Alec Sandy (APS); Oleg Shpyrko (UCSD); Mark Sutton (McGill)

NATIONAL SYNCHROTRON LIGHT SOURCE II project beamline

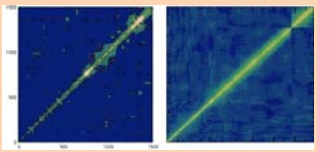
TECHNIQUES AND CAPABILITIES

- Coherent diffraction
- Studies of dynamics with X-ray Photon Correlation Spectroscopy
- Signal-to-noise (SNR) in XPCS: *SNR~brightness x (time)*$^{1/2}$
- Hence: a 10x increase in brightness at NSLS-II will allow measurement of 100x faster fluctuations with the same SNR
- Capabilities for full-field Coherent Diffraction Imaging (CDI)
- Consolidated Endstation instrument for small angle scattering (SAXS) in transmission or grazing-incidence (GI-SAXS), wide angle scattering (WAXS)
- Optical design optimized for very high mechanical stability coherent wavefront preservation
- Flexible focusing scheme using refractive optics
- Spot size: ~10 μm for SAXS, ~1-2 μm for WAXS
- Energy range: 6-15 keV,
- Monochromatic ($\Delta\lambda/\lambda$~10^{-4}) or pink ($\Delta\lambda/\lambda$~$5\cdot10^{-3}$) beam

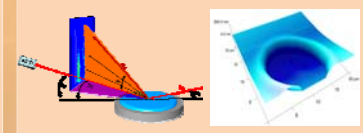
APPLICATIONS



Speckle patterns (left) simulating a "blurring" effect that motion of proteins inside a crystal will create in diffuse coherent diffraction patterns. Motion of motor proteins (right: NOD structure, J. Cochran et al.) will be measured from the speckle visibility



Two-time correlation functions showing non-equilibrium effects ("aging") and heterogeneous dynamics (left) in colloidal gels or dynamical "rare events" in a colloidal glass (right)



Left: Sketch of an experiment measuring dynamics at surfaces and interfaces by GI-XPCS (SAXS) and structural properties via GI-WAXS; Right: Dewetting of a polymer film from a polymer-polymer interface (AFM image, J. Lal, APS)

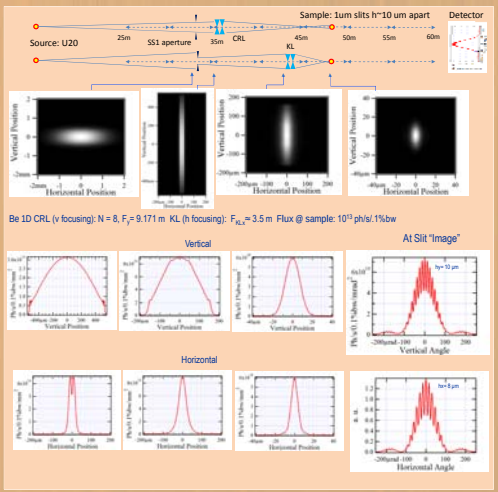
Protein dynamics: Measuring the dynamics of proteins in a crystal can provide invaluable information in understanding their biological function. Molecular motion in protein microcrystals coupled over large scales generate diffraction spots (speckles) around the main Bragg peaks. Here we propose to measure (for the first time) dynamic information about this motion by analyzing the speckle contrast in coherent diffraction patterns.

Non-equilibrium dynamics and self-organization in soft-matter systems: studies of "Matter far beyond equilibrium" was recently identified as one of "Five challenges for science and imagination" in a report to the DOE by BESAC. The CHX instrument will be ideally adapted to address this challenge related to the DOE scientific program. The experiments will focus on understanding complex dynamics in non-equilibrium and driven (e.g. by flow/shear) soft matter systems and the interplay between molecular scale structure, mesoscale dynamics, and macroscopic properties such as the advective response to shear.

Non-equilibrium dynamics at liquid and polymer surfaces and interfaces: with a consolidated endstation design the CHX beamline will enable studies of dynamics and structure of polymer surfaces and interfaces over a wide range of scales. Polymer interfaces are ubiquitous in applications including but not limited to photosensitive films, control of hydrophobicity, biocatalysis, etc.
A detailed in-situ study of the interplay between mesoscale surface dynamics (via GI-XPCS) and molecular scale structure (via GI-WAXS) could provide invaluable information for better understanding such systems and their possible applications.

BEAMLINE PERFORMANCE SIMULATIONS

O. Chubar, K. Kaznatcheev, L. Wiegart, M. Carlucci-Dayton, Y. Baltser, K. Evans-Lutterodt, L. Berman and A. Fluerasu (BNL)



- Beamline performance is evaluated using the Synchrotron Radiation Workbench (SRW) software.
- The SRW package provides a unique tool allowing to simulate the propagation of the "real" partially-coherent wavefront from the undulator source through imperfect optics, sample and all the way to the detector.
- SRW can be used to perform a "virtual coherent diffraction experiment". This is extremely beneficial to beamline staff in designing the instrument but has also tremendous potential applications for users preparing for an upcoming experiment.

COHERENT SOFT X-RAY: HIGH COHERENT FLUX AND FULL POLARIZATION CONTROL BEAMLINE (CSX)

BROOKHAVEN NATIONAL LABORATORY

Beamline Dev. Team: C. Sánchez-Hanke, D. Shapiro, D. Bacescu, C. Stelmach, R. Reininger
Beamline Advisory Team: S. Wilkins (BNL), S. Kevan (U. Oregon), H. Ade (NCSU), D. Arena (BNL), S. Hulbert (BNL), Y. Idzerda (Montana State U.)

NATIONAL SYNCHROTRON LIGHT SOURCE II project beamline

TECHNIQUES AND CAPABILITIES

The Coherent Soft X-Ray beamline will provide high coherent flux (HCF) and full control of the polarization (PC) with two operational independent highly optimized branches.

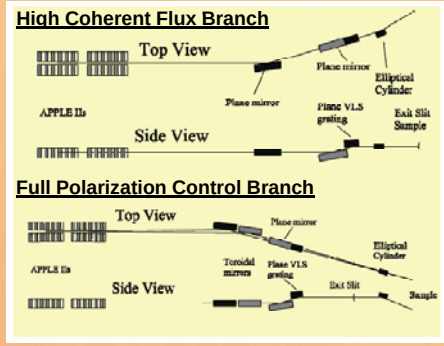
Beamline specifications

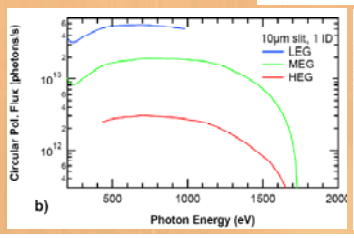
- Source: dual 49 mm period EPU, 38 periods each, canted (PC), inline-phased (HCF)
- Energy range: 200-2000* eV (*polarization dependent).
- Resolving power: $E/\Delta E=1\times10^4$ (PC), 2×10^3 (HCF).
- Photon flux: 10^{13} photons/s circularly polarized (PC), coherent (HCF).
- Spot size: v x h (μm x μm): 20 x 20 (HCF) at 500 eV, 10 x 50 (PC), and 5 x 5 (PC, refocused).
- Variable speed polarization switching up to 1000 Hz.

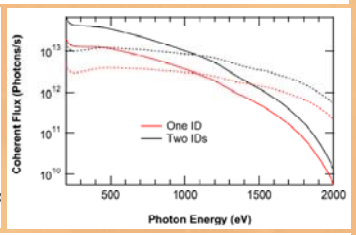
R. Reininger, K. Kriesel, S. Hulbert, C. Sánchez-Hanke, and D. Arena, Rev. Sci. Instrum. 79, 033108 (2008).

Available Techniques

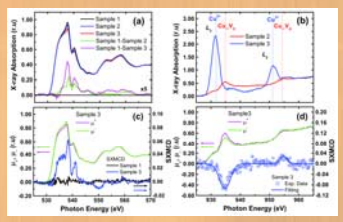
- Coherent x-ray scattering
- Polarization dependent spectroscopy and scattering
- Ultrafast dynamics
- X-ray photon correlation spectroscopy
- Coherent diffraction imaging and ptychography





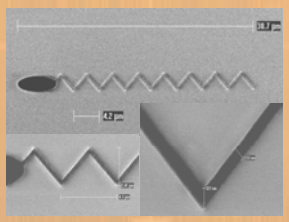


APPLICATIONS



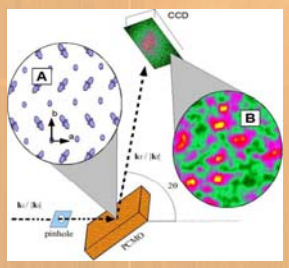
Ferromagnetism in diluted magnetic semiconductors. Such materials are interesting because of their potential to be used in spintronic devices. The figure shows Soft X-ray Magnetic circular dichroism (SXMCD) performed on Cu doped ZnO thin films demonstrating the ferromagnetic behavior of these samples. SXMCD measurements together with theoretical calculations demonstrate the intrinsic character of the effect.

T. S. Herng, *et al*, PRL **105**, 207201 (2010)



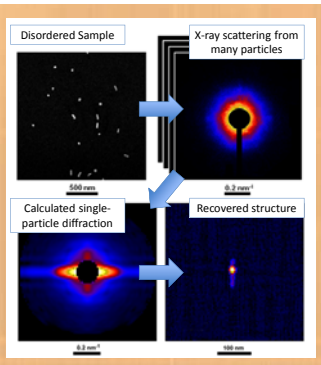
Understanding the dynamics of magnetic domain wall propagation in low dimensional ferromagnetic systems is fundamental to optimize the performance of magnetic based memories or logic devices. Timing experiments using XMCD (X-ray Circular Dichroism) as magnetic contrast allows to study with element specificity the behavior of the domains walls propagation. Because of the reduced sizes and dimensionality these experiments require high photon flux.

(Figure courtesy T. Hase, University of Warwcik)



Dynamics in strongly correlated systems studied with coherent soft x-rays. In these systems the coupling is complicated due to interplay among the charge, the spin and the lattice. Working in the time domain with coherent soft x-rays allows to study the dynamics in the systems in temperature driven phase transitions. The figure above shows the experimental set- up and data collected under Bragg conditions in a half doped $Pr_{0.5}Ca_{0.5}MnO_3$.

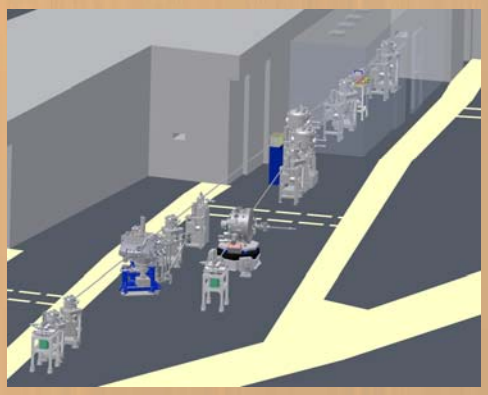
J. J. Turner, *et al*, *New Journal of Physics* 10, 053023 (2008).



Structure of biological molecules from disordered ensembles. Resolving the structure of biological molecules requires many identical copies. These structures can be resolved by means of coherent x-rays as is explained in the figure above. This will allow structural studies of proteins that are difficult to crystallize in a more natural state in aqueous solution.

D. K. Saldin, *et al*, *PRL* 106, 115501 (2011).

SPECIFIC PROJECTS / ADDITIONAL INFORMATION



- There are currently two end stations which match the capabilities of the CSX beamline
- The TARDIS endstation which is currently situated at the NSLS X1A2 beamline matches the capabilities of the HCF branch. It features a six-circle, in-vacuum diffractometer, helium cryostat (down to 5K), zone plate focusing to 30 nm and channeltron and CCD detectors
- A endstation designed for NSLS X13A beamline matches the capabilities of the PC branch. The chamber, features an in-vacuum reflectometer, a 1T in-house designed superconducting vector magnet currently under construction, sample transfer, and sample cooling capabilities down to 20 K

HARD X-RAY NANOPROBE BEAMLINE (HXN)

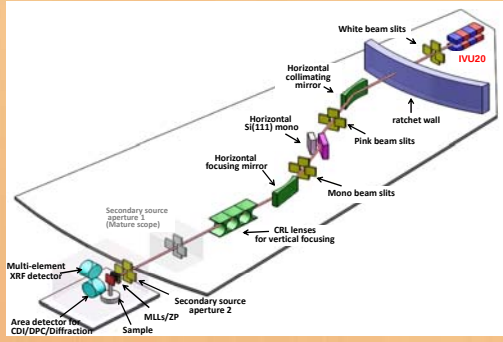
Beamline Development Team: **Y.S. Chu**, L. Margulies, H. Yan, E. Nazaretski, R. Conley, E. Lima, N. Bouet, S. O'Hara, J. Biancarosa, E. Feldman, D. Kuhne
Beamline Advisory Team: **I.C. Noyan** (Columbia), D. Bilderback (Cornell), C. Jacobsen (ANL/Northwestern U.), A. Lanzirotti (U. Chicago), T. Buonasisi (MIT), S. Vogt (ANL), M. Holt (ANL), K. Evans-Lutterodt (BNL)

BROOKHAVEN NATIONAL LABORATORY

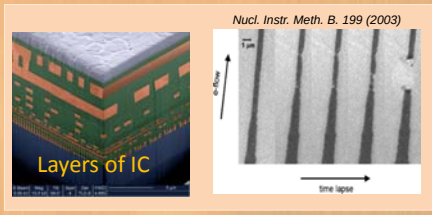
NATIONAL SYNCHROTRON LIGHT SOURCE II project beamline

HXN SCIENTIFIC CAPABILITIES

- Unprecedented fluorescence sensitivity: ~1000 cts/s for 1000 Zn atoms with 10nm focus at 10keV.
- Imaging material heterogeneity using multiple contrasts to analyze elemental composition, morphology, oxidation states, crystalline phase, orientation, and strain.
- Structural analysis of buried interfaces of a wide range of "hard" materials to investigate structure-functionality relations.
- Spectro-microscopy analysis on environmental materials will dramatically enhance current understanding of mineral interfacial reactions and bio-geochemical reactions at the nanoscale.
- Capability to image and quantify metal distributions in cells and tissues will elucidate metal-mediated biological processes, their linkage to diseases, and lead to potential treatment.

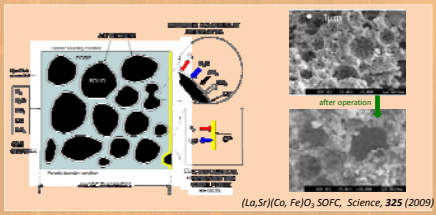


APPLICATIONS



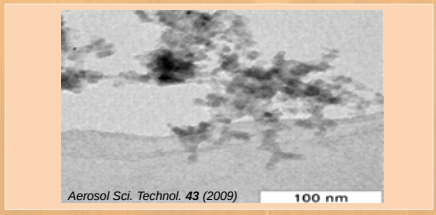
Nucl. Instr. Meth. B. 199 (2003)

In-situ investigation of electro-migration in the next-generation Integrated Circuits (IC). Parallel mapping of strain and elemental concentration will reveal evolution of strain fields before, during, and after the electro-migration and help mitigate its effects. Electron microscopy techniques require sectioning that greatly modifies the strain field.



*$(La,Sr)(Co, Fe)O_3$ SOFC, Science, **325** (2009)*

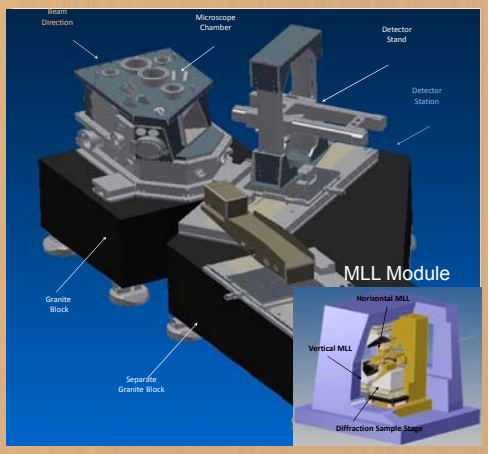
Small changes in the nanostructure of solid oxide fuel cells significantly influences macroscopic properties. Electron microscopy is inadequate for answering critical questions on 3D morphologies, composition, and oxidation states. HXN will allow measurements which correlate the nanostructure and chemical states at triple-phase boundaries with the macroscopic performance.



*Aerosol Sci. Technol. **43** (2009)*

Nano-scale heterogeneity, reactivity, bioavailability, and toxicity of environmental particulates or atmospheric nanoparticles can be investigated by imaging elemental composition and oxidation states. The results will also have atmospheric implications for climate forcing models, and provide insights into surface chemistry and mechanisms for aggregation and transport.

CAPABILITIES OF THE HXN X-RAY MICROSCOPE



Focusing optic	Spatial resolution	Energy range	Energy resolution	Fluorescence	Fluorescence tomography	XANES	Diffraction
MLL	1-10 nm	10~25 keV	~1e-4	Yes	No	Limited	Yes
ZP	≤ 30 nm	6~12 keV	~1e-4	Yes	Yes	Yes	Yes

- Highest spatial resolution with scientific flexibility
- XRF fly-scan with a minimum dwell time of 1 ms per pixel
  → 5 minutes for 5um x 5um 2D mapping with 10nm pixel size
- Coherent diffraction imaging higher spatial resolution than the probe size
- Substantial nano-diffraction capabilities
  → capable of analyzing in-plane and out-of-plane strain for crystalline samples
- Diffraction-contrast imaging with parallel XRF and DPC measurements
- Strain sensitivity of ~10^{-4}

INELASTIC X-RAY SCATTERING (IXS)

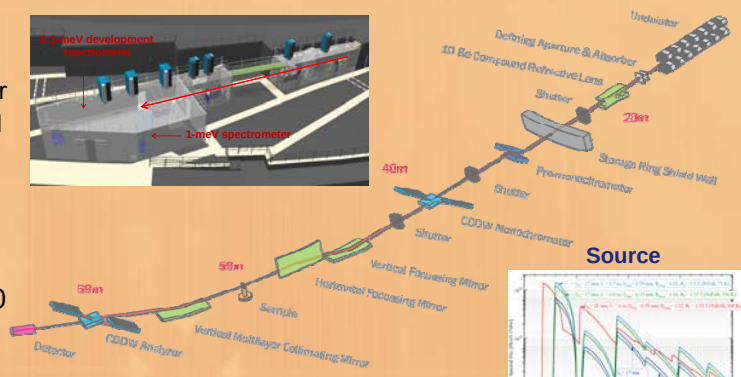
Beamline Development Team: Y. Cai[1], D.S. Coburn[1], A. Cunsolo[1], M. Hönnicke[2], X. Huang[3], J. Keister[1], C.N. Kodituwakku[1], Y. Stetsko[1], A. Suvorov[1], 1- BNL, 2- Univ. Federal de Goias Brazil, 3- Argonne National Laboratory
Beamline Advisory Team: Clement Burns (Western Michigan U), A. Baron (SPring-8/RIKEN), S.H. Chen (MIT). J. Hill (BNL), M. Krisch (ESRF), H.K. Mao (Carnegie Inst.), S. Shapiro (BNL), T. Scopigno (U of Rome), Y. Shvyd'ko (APS)

NATIONAL SYNCHROTRON LIGHT SOURCE II project beamline

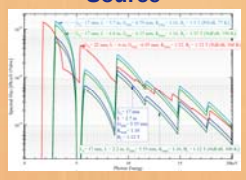
TECHNIQUES AND CAPABILITIES

- 1 meV spectrometer (5 m) with single analyzer
  - Operates at E ~ 9.13 keV with the CDW/CDDW [1] scheme or the 4-Bounce high-resolution monochromator
  - Total energy resolution ~ 1 meV with sharp resolution tail
  - Scanned energy range: ~ 1 eV
- Incident flux at sample > 10^{10} photons/sec/1meV
- Q range / resolution: 0.1 ~ 40 nm^{-1} / ~ 0.1 nm^{-1}
- Focus: ~ 5 μm (V) x 7 μm (H)
- Upgraded 1-meV spectrometer: phase plate for Q > 40 nm^{-1} (2θ > ~50 degrees)
- Ultimate goal: Ultrahigh resolution monochromator for 0.1 meV, with 0.1 meV spectrometer (10 m)

Optical Design and Beamline Layout



Source

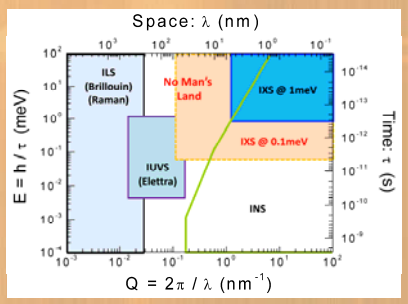


[1] Y. Shvyd'ko et al, Physical Review Letters (2006)

APPLICATIONS

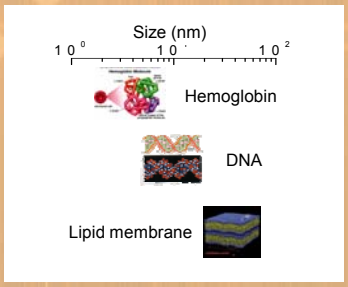
Inelastic X-ray Scattering (IXS) is a powerful technique for studying dynamics and excitations in condensed matter systems, and has been used to study excitations ranging from phonons in solids, to sound modes in liquids and polymers, to plasmons in simple metals, to complex electronic excitations in strongly correlated electron systems. The scientific objective of the NSLS-II IXS Beamline is focused on very high-resolution (1 ~ 0.1 meV) IXS experiments in order to solve some of the most important problems in dynamics. An ultra high resolution of 0.1 meV represents an order of magnitude improvement over the best currently existing instruments in the world and would allow the exploration of an important dynamical region not accessible thus far, which is expected to provide new research opportunities in studying excitations in liquid, disordered, and biological systems, as well as in phonon research.

Dynamic domains covered by current inelastic scattering probes



Dynamics of disordered systems

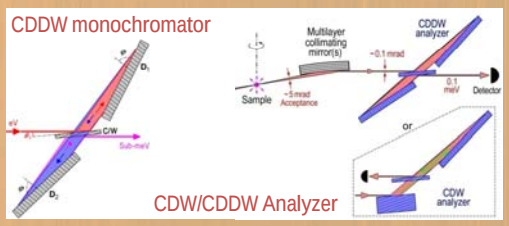
- Involves a group of atoms (e.g., bio-molecules)
- Typical length scale: 5~50 nm, part of "No Man's Land"

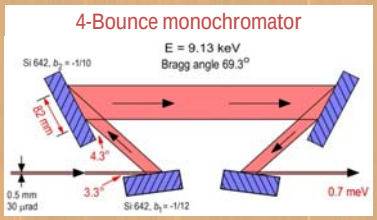


Key scientific questions to address

- Dynamics of liquids and disordered systems
  - The frequency and wavelength dependence of sound absorption in glasses still has many unsolved topics as e.g. concerning the nature of damping and the existence of non longitudinal modes etc.
- Vibration dynamics under high pressure and temperature
  - Extreme thermodynamic condition are accessible thanks to the small transversal x-ray beam size
- Phonon and phonon-damping in bio-materials
  - Experimental evidence suggest the occurrence of phonon-assisted molecule transport across lipid bilayers
- Phonon line width in high temperature superconductors
  - A challenge: measuring the strength of electron-phonon interaction

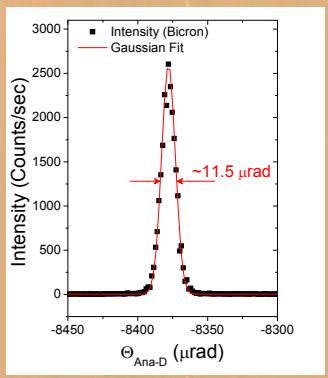
ULTRAHIGH RESOLUTION 0.1 meV OPTICS





Current CDW-CDW test results

- Performed at X16A Optics R&D beamline
- Energy resolution ~2.6 meV; efficiency > 3%
- Gaussian-like resolution function tails



Major Technical Challenges

- Lattice homogeneity of crystal:
  $\Delta d/d = \Delta E/E = 0.1meV/10keV \sim 10^{-8}$
- Temperature uniformity and stability:
  $\Delta d/d = \alpha\Delta T \rightarrow \Delta T \leq 4mK$
- Length of dispersive element (D) up to ~1.5 m for 0.1 meV
- Multilayer mirror for the analyzer system
  - Angular acceptance: 5~10mrad
  - Stringent requirements on layer thickness variation, surface roughness, and slope error

Alternative monochromator design

- A proven alternative for 1-meV monochromator with a wide scanning energy range.
- Higher efficiency than CDW/CDDW schemes for 1-meV resolution
- Smaller angular acceptance to be implemented for the analyzer

SUB-MICRON RESOLUTION X-RAY SPECTROSCOPY (SRX)

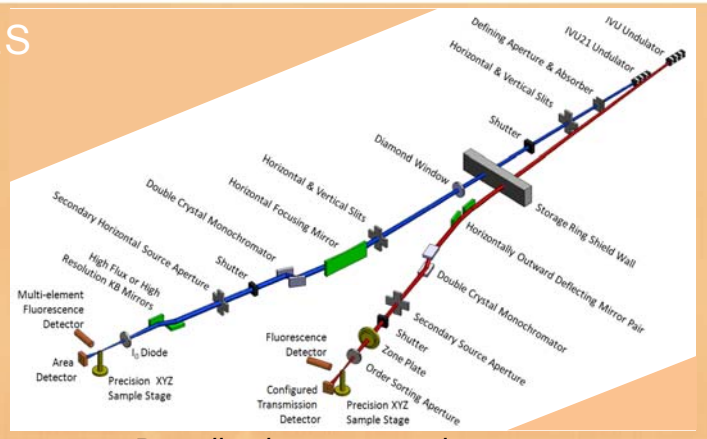
Beamline Development Team: **J. Thieme[1]**, V. De Andrade[1], Y. Yao[1]
Beamline Advisory Team: P. Eng[2], J. Fitts[1], C. Jacobsen[3], K. Jones[1], **A. Lanzirotti[2]**, L. Miller[1], M. Newville[2], P. Northrup[4], R. Reeder[4], M. Rivers[2], S. Sutton[2], S. Vogt[3], G. Woloschak[5]
[1]Brookhaven National Lab, [2]Univ. Of Chicago, [3]Argonne National Lab, [4]Stony Brook Univ., [5]Northwestern Univ.

BROOKHAVEN NATIONAL LABORATORY

NATIONAL SYNCHROTRON LIGHT SOURCE II project beamline

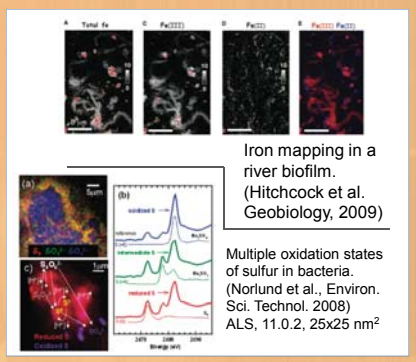
TECHNIQUES AND CAPABILITIES

- Versatile but optimized tool for studies in: Environmental, Life, Earth, Planetary, Material & Medical Sciences, Energy Research.
- X-ray spectroscopy with either sub-µm or 50 nm spatial resolution. Energy range: 2 keV ≤ E ≤ 30 keV.
- 2D fluorescence / diffraction imaging, fluorescence/diffraction-tomography
- Outstanding performance for trace elements analysis



Beamline layout, canted geometry
(Zone plate branch in red not yet funded)

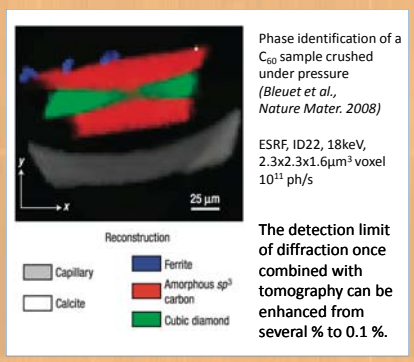
APPLICATIONS



Iron mapping in a river biofilm. (Hitchcock et al. Geobiology, 2009)

Multiple oxidation states of sulfur in bacteria. (Norlund et al., Environ. Sci. Technol. 2008) ALS, 11.0.2, 25x25 nm²

- **Microbiology of anaerobic Fe(II) oxidation as a regulator of radionuclide transport.** *Sub-100 nm resolution and high flux allow for search of trace elements within bacterial cells. Mature scope: Zone plate branch gives access to P, S, Cl, Ca-edges*

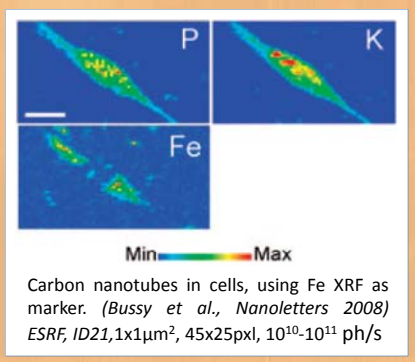


Phase identification of a C_{60} sample crushed under pressure *(Bleuet et al., Nature Mater. 2008)*

ESRF, ID22, 18keV, 2.3x2.3x1.6µm³ voxel 10^{11} ph/s

The detection limit of diffraction once combined with tomography can be enhanced from several % to 0.1 %.

- **Formation of soils by rock weathering**. *Combination of XRF & diffraction tomography (mature scope: area detector) will provide a new insight in complex weathering processes, probing complete mineral particles with a high sensitivity.*



Carbon nanotubes in cells, using Fe XRF as marker. *(Bussy et al., Nanoletters 2008) ESRF, ID21,* 1x1µm², 45x25pxl, 10^{10}-10^{11} ph/s

- **Impact of anthropogenically produced nanoparticles on health and environment**. *High resolution and stability will provide a detailed picture of these nano-objects and will allow spectroscopic approaches. A clear identification of organic matrix will be possible on the Zone Plate branch (P, S, Cl, Ca – edges).*

PERFORMANCE

Flux & spot size: (assessed by ray tracing calculations & wave front propagation simulations)

✓***High flux KB :*** *Flux:* 1.56 x 10^{13} ph/s @ 12 keV, *Spot size:* 0.8 x 0.5 µm²

✓***High Resolution KB :*** *Flux:* 10^{11} – 10^{12} ph/s @ 12 keV, *Spot size:* 50 x 50 nm²

✓ ***ZP (not yet funded):*** 7 x 10^{9} ph/s @ 12 keV, at diffraction limited resolution (≈30 nm)

Comparison of energy range, spot size and flux of similar beamlines. This compilation does not claim to be complete (*Source: facility website)*

Name	Energy range / keV	Spot size / µm²	Photon flux in spot
SRX @ NSLS-II	4.7 – 23	0.5 x 0.8 0.05 x 0.05	1.5 x 10^{13} 1 x 10^{12}
ID21 @ ESRF	2 – 9	0.35 x 0.7	10^{10}– 10^{11}
ID22 @ ESRF	6.5 – 18	3.5 x 1.5	10^{12}
NINA @ESRF	discrete 11.2/17/33.6 (NI) 5-70 (NA)	0.01 – 0.1 (NI) 0.05 - 1	2.5 x 10^{12} 1.8 x 10^{11}
2-ID-B @ APS	2 – 4	0.06 x 0.06	10^{9}
2-ID-D @ APS	5 – 30	0.2 x 0.2	4 x 10^{9}
2-ID-E @ APS	7.5 – 10	0.5 x 0.3	5 x 10^{9}
13-ID-C,D @ APS	4 – 45	2 x 2	10^{11}
20-ID-B,C @ APS	4.3 – 27	2 x 2	10^{11}
XFM @ AS	4 – 25	0.06 x 0.06	10^{10}
MicroXAS @ SLS	5 – 20	1 x 1	2 x 10^{12}
Nanoscopium@ SOLEIL	5 – 20	0.1 x 0.1	1.4 x 10^{10}

TECHNICAL CHALLENGES

To fully benefit from the high stability & ultra-low emittance of the NSLS-II source, state-of-the-art optical components are required.

- Ultra stable fixed-exit horizontally diffracting DCM
  → angular stability < 50 nrad
- Ultra stable Horizontally Focusing Mirror:
  slope errors in bent conditions < 0.3 µrad
- 2 KB systems:
  → high flux set: 35 & 30 cm long mirrors with slope errors ≤ 0.2 µrad
  → high resolution set: small ultra-stable & high surface quality (0.1 µrad) mirrors with fixed curvature to reach the diffraction limit (wave front propagation study with SRW in progress, SPIE 2011).
- Fluorescence Detection:
  Fast energy dispersive area detector (being developed by Peter Siddons' Detector Group) will provide exceptional capabilities for trace element analysis.

X-RAY POWDER DIFFRACTION (XPD)

Beamline Development Team: **E. Dooryhee**, S. Ghose, M. Rehak, X. Shi, J. Trunk, Z. Zhong

Beamline Advisory Team: ***S. Billinge*** *(Columbia U./BNL), J. Parise (SBU), P. Stephens (SBU), J. Hanson (BNL), J. Kaduk (NEOS Technologies), P. Chupas (ANL)*

BROOKHAVEN NATIONAL LABORATORY

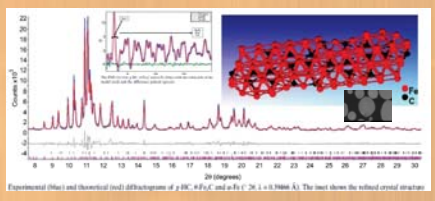
NATIONAL SYNCHROTRON LIGHT SOURCE II project beamline

TECHNIQUES AND CAPABILITIES

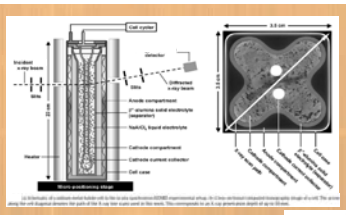
- Techniques: powder diffraction, total scattering (PDF), small/medium angle scattering (SAXS, MAXS), tomography (CT). Opportunities for *in-situ* and *in-operando* structural science (variety of sample environments, complex materials in working conditions or in operating devices).
- Source: 1.8 T Damping Wiggler, E_c = 10.8 keV.
- 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 ~10 μm
- Time Resolution: ~ms (dynamics, rapid acquisition)
- Beamline components: cryo-cooled Laue monochromator, vertically focusing mirror, channel-cut and side-bounce monochromators.

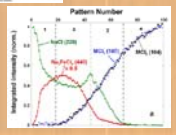
XPD features two independent branches served by Laue-crystal monochromators and three in-line endstations fitted with high-resolution, position-sensitive and large-area detectors. XPD is capable of collecting data at high energies ideal for high-*Q* data and *in situ* and time-resolved studies in environmental cells.

APPLICATIONS

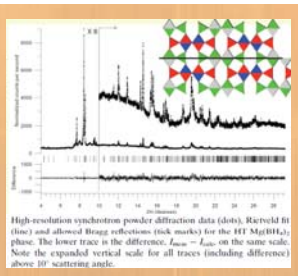


High energy total structure: XPD offers to examine materials *in situ* across μm-nm-Å length-scales by combining high *Q*-space resolution (long-range crystallographic structure) and high real-space resolution (Pair Distribution Function: local structure or nanostructure). The example above is a combined Rietveld and PDF study of Fe_5C_2 Hägg carbide from H. Esna du Plessis *et al*. J. of Synchrotron Rad. 18 (2011). The PDF analysis shows the nanocrystalline structure of active Fe_5C_2 catalyst. Comparing quantitative Rietveld and PDF shows that the bulk structure extends to the surface of the 5-40μm particles.





Space and time resolved high energy diffraction: XPD is optimized for *in operando* time and space studies of the electrochemical processes taking place during charge/discharge cycling at high temperature inside a full size battery cell. The example above is a prototype sodium metal halide cell from J. Rijssenbeek *et al*. J. of Power Sources 196 (2011). XPD will aid the advancement of battery technology by enabling the study of the fundamental mechanisms at work inside commercially relevant cells of different battery types.



In situ high energy diffraction: The structure of candidate H_2 storage material is to date determined *ex situ* on recovered structure. XPD aims for *in operando*, high resolution structure at high gradients (H_2 pressure, T) rather than on recovered samples, with time resolutions in the msecs (intermediate states in decomposition) and data at high-*Q* to image individual particles in the working device. Knowledge of the structure is a necessary step in understanding the hydrogen desorption properties and finding a practical means of regenerating the material. The example is from Jae-Hyuk Her *et al*. Acta Cryst. B63 (2007).

RESEARCH & DEVELOPMENT PROJECTS

Fig. 1

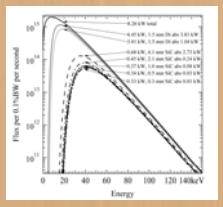


Fig. 2

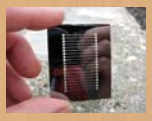


Fig. 4

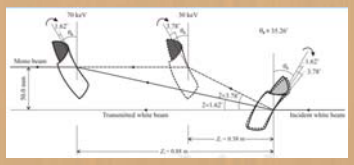


Fig. 3

- High-energy bandpass filtering: the power of the source (61 kW) is reduced by the means of 2 fixed-aperture masks and 2 fixed, redundant diamond windows followed by a set of moveable filters. **Fig. 1** shows the diamond windows at the JEEP beamline (Diamond Light Source, M. Drakopoulos). The diamond windows serve as heat filter, shield and vacuum barrier. Sequential filtering causes beam hardening. **Fig. 2** shows the power spectrum distribution.
- Double Laue Monochromator (DLM): the sagittal bending of the asymmetric Laue crystals focuses the incident horizontal fan (35 mm to 0.5 mm). Severe crystal distortions broaden the energy bandwidth and produce a significant gain in flux. Efficient crystal cooling and control of the sagittal and the anticlastic bending are challenging (X. Shi *et al*. J. Appl. Cryst. 2011). This DLM will be an unique instrument acting as the first-white beam optics at a third-generation source (**Fig. 3**).
- Laue analyzer optics: the high-resolution mode of XPD uses a post-sample array of parallel crystals. All existing powder diffractometer designs rely on the concept developed at the CNRS-ESRF by J-L. Hodeau *et al*. and operate below 40 keV with Bragg crystals. The proposed solution (P. Siddons *et al*. SPIE 1997) consists of a multi-element monolithic Laue crystal of Si (**Fig. 4**), elastically bent in a pseudo-Rowland circle geometry and combined with a multi-channel detector.
- High energy detectors: the low absorption of high-energy x-rays by silicon drastically limits the efficiency of Si-based detector technology. Pending successful engineering of higher-Z sensors in the coming years, a 10-fold increase in detector efficiency at 60 keV could be expected. A custom multi-element Germanium detector is being considered as part of an in-house R&D effort.

ELECTRON SPECTRO-MICROSCOPY FACILITY FOR FUNDAMENTAL STUDIES OF THE PHYSICS AND CHEMISTRY OF MATERIALS (ESM)

BROOKHAVEN NATIONAL LABORATORY

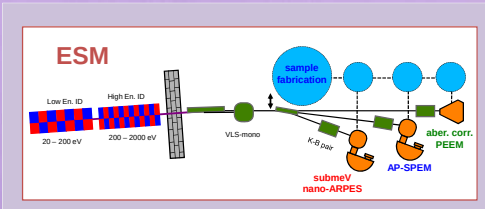
Proposal Team: D. Arena[1], J. Chen [2], T. C. Chiang [3], S. Hulbert[1], P.D. Johnson [1], A. Kaminski[4], M. Kiskinova[5], R. Osgood, Jr.[6], R. Reininger [1], J. Rodriguez[1], J. T. Sadowski[1], B. Sinkovic[7], K. Smith[8], D. Starr[1], R. Tromp[9], T. Valla[1], **E. Vescovo[1]**

[1]Brookhaven National Lab, [2]Univ. Of Delaware, [3]Univ. Of Illinois, [4]AMES Lab, [5]ELETTRA, [6]Columbia Univ., [7]Univ. of Connecticut, [8]Boston Univ., [9]IBM

NATIONAL SYNCHROTRON LIGHT SOURCE II approved: NEXT

TECHNIQUES AND CAPABILITIES

- Source: 2 ID – EPU 56 (4.5 m, EM , 20-200 eV)
  EPU 180 (3.5 m PM, 200-2000 eV)
- High-Resolution Angular Resolved Photoemission Scanning Microscopy (μ-ARPES): 20-1500 eV, 1 μm, < 1 meV, <0.1°, 5-2000 K
- Ambient Pressure Scanning Photoelectron Microscopy (AP-SPEM): 200-1800 eV, < 300 nm, 10^{+3} Torr
- Low-Energy Electron Microscopy & X-ray Photoemission Electron Microscopy (LEEM/XPEEM): 20 – 1800 eV, < 10 nm, high-transmission aberration correction



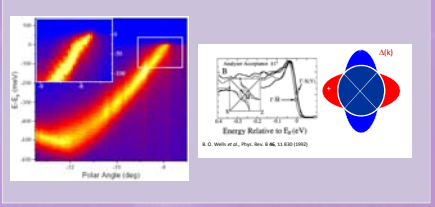
E-range 20 – 2000 eV

E-resol. < 1 meV to 70 eV (0.2 meV @ 20 eV)
< 10 meV to 1000 eV
< 100 meV to 2000 eV

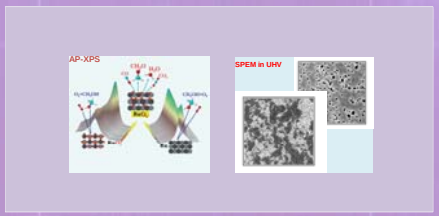
Flux >10^{12} ph/sec @ 10000 resolving power

Spot-size 200 x 300 nm (K-B), ~ 50 nm (ZP)

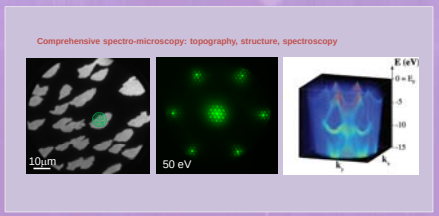
APPLICATIONS



μ-SP-ARPES:
momentum resolved electronic structure of solids; spin-polarized electronic bands of magnetic materials and non-magnetic materials with large spin-orbit interaction; a 1 μm spot from NSLS-II will allow for scanning spectro-microscopy.

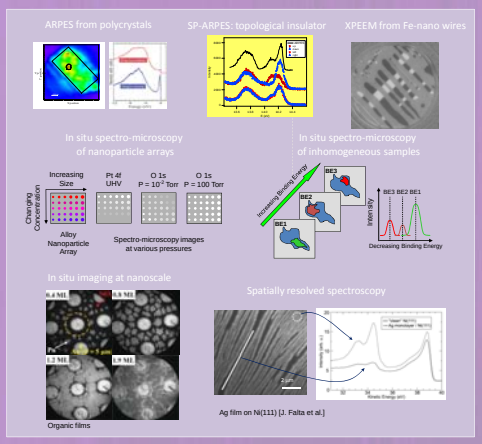


APSPEM:
unites two experimental techniques (AP-XPS and SPEM) provide < 300 nm spatially resolved, quantitative chemical analysis of surfaces at pressures up to atmospheric pressure. This is a direct result of the superb brightness of NSLS-II facility in the soft-x-ray region.



XPEEM:
Energy filtered-XPEEM with aberration-corrected, high transmission imaging spectrometer is an intrinsically non-destructive, fast-imaging technique allowing simultaneous surface sensitive microscopy and spectroscopy.

SPECIFIC PROJECTS / ADDITIONAL INFORMATION



- ARPES from spatially inhomogeneous samples, as well as artificially formed hetero-structures (nano-dots, wires, field-effect transistors, graphene ribbons, etc.)
- ARPES from polycrystalline or micro-crystal materials
- SP-ARPES from inside a magnetic domain
- AP-SPEM: chemical mapping of inhomogeneous and nanofabricated samples with spatial resolution of 300 nm or less
- XPEEM: fast, full-field probe for spatially resolved topographic, structural and spectroscopic measurements

FULL FIELD X-RAY IMAGING (FXI)

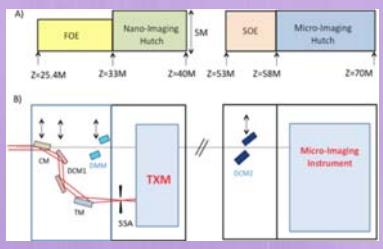
Proposal Team: G. Amatucci[1], L. Berman[2], L. Butler[3], W. Chiu[4], Y. Chu[2], P. Crane[5], D. Dunand[6], C. Erdonmez[2], K. Evans-Lutterodt[2], P. Herendeen[7], Y. Hwu[8], K. Jones[2], J. Jordan-Sweet[9], K. Kornev[10], A. Lanzirotti[11], W.-K. Lee[12], H.-K. Mao[13], L. Makowski[12], C. Ortiz[14], **J. Socha[15]**, S. Stock[16], J. Thieme[2], P. Vlachos[15], U. Wegst[17], C. Willson[3], W. Yang[12], **J. Wang[2]**
[1]Rutgers Univ., [2]Brookhaven National Lab, [3]Louisiana State Univ., [4]Univ. of Connecticut, [5]Yale Univ., [6]Northwestern Univ., [7]Chicago Botanic Garden, [8]Inst. of Physics Taiwan, [9]IBM Research Center, [10]Clemson Univ., [11]Univ. of Chicago, [12]Argonne National Lab, [13]Carnegie Inst. of Washington [14]Massachusetts Inst. of Technology, [15]Virginia Tech, [16]Northwestern Univ. Medical School, [17]Drexel Univ.

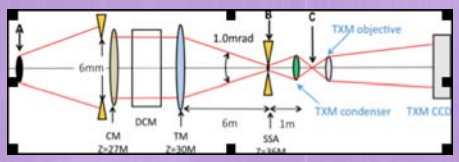
BROOKHAVEN NATIONAL LABORATORY

NATIONAL SYNCHROTRON LIGHT SOURCE II NEXT

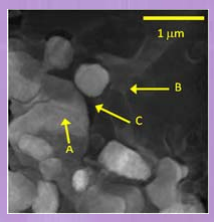
TECHNIQUES AND CAPABILITIES

- Long beamline utilizing a SCW source
- Full-field configuration will enable 2D and 3D dynamic imaging of centimeter-scale samples with micron resolution
- Transmission x-ray microscope (TXM) will achieve high-speed imaging with 30 nm resolution
- Energy range: 5-60 keV (TXM: 5-11 keV)
- Spatial resolution: ~1–5 μm (full-field); 30 nm (TXM)
- Large user community serving national needs and addressing fundamental issues across diverse fields of science and engineering

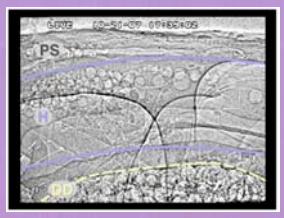




APPLICATIONS



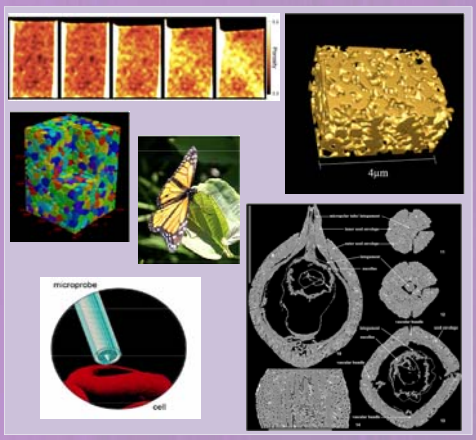
Tomographically reconstructed 3D volumetric data of a Ni-YSZ cermet sample. Enhanced contrast between the Ni and YSZ phases are achieved by tuning the incident x-ray energy just above the Ni K-edge. A: Ni phase (anode), B: YSZ phase (electrolyte), C: porous TPB between the anode and electrolyte. Wilson et. al. J. Electrochemical Society, 157 (6) B783-B792 (2010). The NSLS-II will enable a larger field of view and faster imaging.



Particle tracking in a section of the tubular heart of a grasshopper, demonstrating the feasibility of synchrotron PIV of biological flows. FXI at NSLS-II would enable visualization of the entire heart at once, elucidating how exactly flows are created for possible biomimetic microfluidic applications. W.-K. Lee & J.J. Socha., BMC Physiology 9:2 (2009).

3D imaging of a solid oxide fuel cell anode showing the distinct Ni and YSZ phases: orange-Ni; red-YSZ, black-pore/empty space. Direct observations of energy systems under real operating conditions can provide fundamental insight into the physical and chemical phenomena that underlie the operation of energy devices, which will catalyze the development of new materials and processes required for future energy systems.

SPECIFIC PROJECTS / ADDITIONAL INFORMATION



- X-ray imaging for biofuels to evaluate biomass breakdown.
- *In situ* and 3D imaging of functional nano-materials under realistic environmental conditions.
- *In situ* studies on voiding in semiconductor devices to image and characterize buried 3D structures in a non-destructive manner, including studies of study of crack formation, interfacial delamination, void and pore formation, material decomposition and bulk reactions.
- Pore-scale investigation of soils and sediments using tomography to understand natural processes such as diagenesis, chemical weathering, and hydrate dissolution/formation.
- Advances in bio-inspired micro- and nano- fluidics, structural biological materials, and paleontology.

INTEGRATED *IN-SITU* AND RESONANT HARD X-RAY STUDIES (ISR)

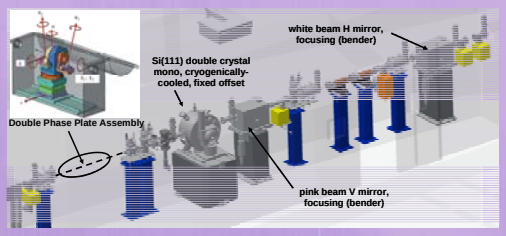
Proposal Team: R. Clarke[1], A. Fluerasu[2], R. Headrick[3], J. Hill[2], J. Jordan-Sweet[4], V. Kiryukhin[5], **K. Ludwig[6]**, P. Lyman[7], C. Nelson[2], R. Pindak[2]

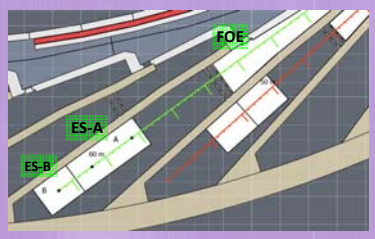
[1]Univ. of Michigan, [2]Brookhaven National Laboratory, [3]Univ. of Vermont, [4]IBM, [5]Rutgers Univ., [6]Boston Univ., [7]Univ. of Wisconsin

NATIONAL SYNCHROTRON LIGHT SOURCE II NEXT

TECHNIQUES AND CAPABILITIES

- Two Stations using 3m undulator in high-β straight section:
  - ES-A with psi 6-circle diffractometer and heavy-load diffractometer with 10+ T magnetic field
  - ES-B with base diffractometer accommodating custom large environment chambers
- Energy range of 2.4-23 keV for resonant studies of a very wide range of important elements
- Full polarization control and analysis
- KB mirrors giving 1 μm vertical focusing
- Coherence-preserving optics for studies of nanoscale dynamics and complementary imaging studies in complex environments
- Capable of combining a wide range of techniques for integrated materials investigations including: *Polarized XRD, RXS, CTR, XRR, GISAXS, GID, XPCS, XRIM*

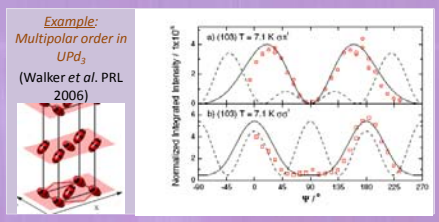




APPLICATIONS

Example: Multipolar order in UPd_3

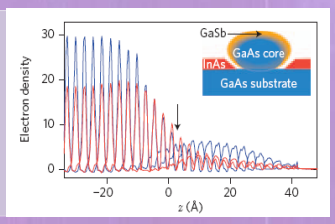
(Walker *et al.* PRL 2006)



ISR will provide a unique tool for studies of new materials with novel electronic and magnetic properties. The combination of such an extended energy range with full polarization control/analysis would allow definitive characterization of electronic states, including their multipolar character and the quantum-mechanical nature of the magnetic moment.

***Example:* Atomic-scale mapping of quantum dots formed by droplet epitaxy**

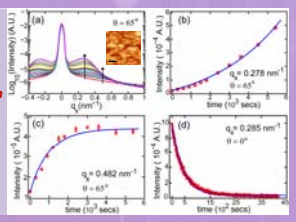
(Kumah *et al.* Nature Nano. 2009)



ISR will bring unprecedented brilliance to probe the structure of chemistry of functional surfaces and interfaces through CTR studies. Its wide energy tunability will also enable resonant scattering studies that can dramatically improve diffraction phasing. Moreover, the polarization control of the beamline will facilitate the study magnetic order at interfaces and surfaces.

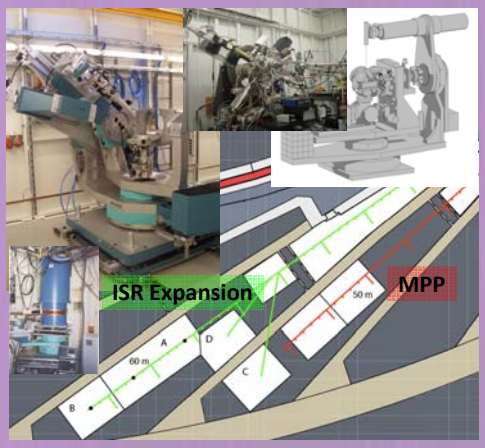
***Example:* Real-Time GISAXS measuring kinetics of self-organized nanoscale structure formation on Si during Ar^+ bombardment**

(Madi *et al.* PRL 2011)



ISR will accommodate a wide range of chambers for studies of surface and growth processes in complex environments. In addition to enabling higher flux/higher time resolution "incoherent" surface-sensitive studies it will also give an enabling impetus to the development of surface-sensitive coherent x-ray studies of surface dynamics.

SPECIFIC PROJECTS / ADDITIONAL INFORMATION



- Psi 6-circle diffractometer in ES-A will enable use of small environmental chambers for *in-situ* experiments
- ES-B will enable use of flexible range of sophisticated environment large chambers for *in-situ* study of materials during growth/processing
- A 1 μm vertical focus of the full beam intensity (nearly 10^{14} photon/s with Si(111) resolution) with 1.5 m working distance from the focusing optic
- Synergy with proposed NEXTGen Materials Physics and Processing (MPP) Beamline
- Future build-out possibility adding canted undulator feeding one or two stations with side bounce fixed energy monochromators

INNER-SHELL SPECTROSCOPY (ISS)

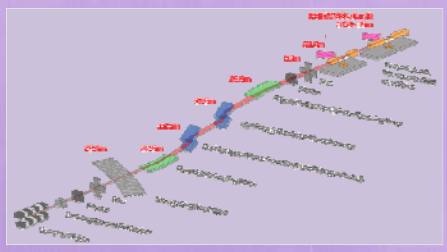
Proposal Team: S. Bare[1], L. Berman[2], J. Bohon[3], B. Bunker[4], M. Chance[3], S. DeBeer[5], A. Frenkel[6], C. Glover[7], S. Kelly[1], P. Northrup[8], W. Mao[9], B. Mishra[10], S. Myneni[11], **B. Ravel[12]**, G. Seidler[13], T. Tyson[14], J. Woicik[12]
[1]UOP, [2]Brookhaven National Laboratory, [3]Case Western Reserve University, [4]Notre Dame University, [5]Cornell University, [6]Yeshiva University, [7]Australian Synchrotron, [8]Stony Brook, [9]Stanford University, [10]Argonne National Laboratory, [11]Princeton University, [12]NIST, [13]University of Washington, [14]New Jersey Institute of Technology

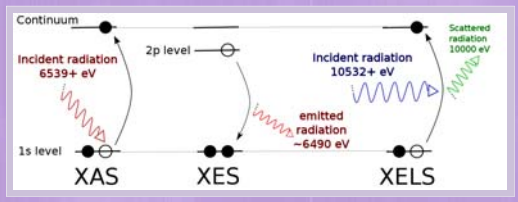
BROOKHAVEN NATIONAL LABORATORY

NATIONAL SYNCHROTRON LIGHT SOURCE II NEXT

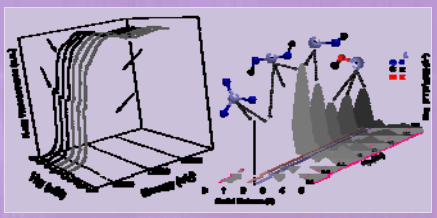
TECHNIQUES AND CAPABILITIES

- Very high flux up to 10^{14} ph/sec from a wiggler source
- The very best X-ray Absorption Spectroscopy (XAS) on dilute or low-volume samples of environmental, biological, technological, and other scientific relevance
- High-resolution X-ray Absorption Near Edge Structure (XANES) using crystal spectrometers
- Novel spectroscopies: X-ray Emission Spectroscopy (XES) and X-ray Energy Loss Spectroscopy (XELS) using novel spectrometers well suited for the beam characteristics of a wiggler source
- Combined experiments: XAS combined with XES and XELS on the same sample and in one visit to the beamline
- Real measurements on real samples under real conditions via tight integration of optics and end-station instrumentation
- Click and play beamline operations



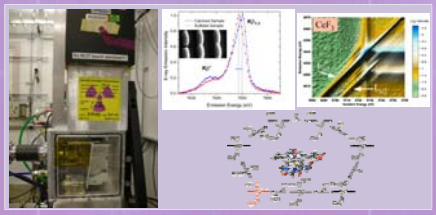


Low concentration XAS



The adsorption of Hg to *B. subtiliis* and *S. oneidensis* MR-1 biomass was investigated to understand the interaction of Hg with bacterial cell surfaces. A wide range of Hg^{2+} concentration (120 nM to 350 μM) was measured at a fixed bacterial cell density and pH. The Hg L_{III} edge XAS analysis showed that Hg complexes entirely with sulfhydryl groups at the nanomolar and low micromolar concentrations, and with carboxyl sites at high micromolar concentrations. The sulfhydryl interaction would not be observed at Hg concentrations measurable at a dipole XAS beamline.

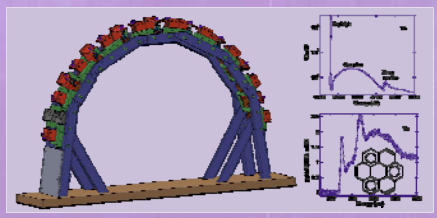
XES



The miniXS spectrometer allows for a fast, efficient measurement of XES in a flexible, dispersive geometry that can be incorporated into many *in situ* measurements.

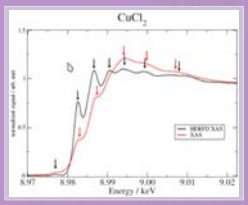
Example: Methane monooxygenase (MMO) is an ideal systems for time-resolved, dispersive XES studies. MMO is a common methane hydroxylation enzyme with an enzymatic cycle composed of structurally distinct, short lived states. With miniXS the transformations can be monitored on ms time scale in a stop-flow arrangement, as a function of spin state at the iron (Kβ main line), and changes in ligand/substrate binding (valence core region).

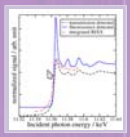
XELS



XELS offers an enticing alternative to conventional soft X-ray spectroscopy. Using an inelastic scattering spectrometer, low energy edges are probed via an energy loss channel. Since a hard X-ray probe is used, XELS is compatible with many *in situ* sample environments, opening soft X-ray spectroscopy to operating batteries, controlled atmosphere environments, and more. In the example shown, C K edge spectra from graphite are shown from a study of polyaromatic hydrocarbons for which the size of an individual molecule exceeds the Auger escape depth. This molecule can *only* be measured by XELS.

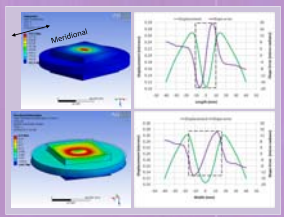
High resolution XANES





- Use a crystal analyzer to measure XANES spectra below the core-hole lifetime.
- Enhanced resolution of XANES features and more precise determination of edge position and absorber valence.
- This is a standard tool at ISS – ***all*** XANES measurements will be measured at high-resolution

High heat-load optics



- Management of the high heat load of the wiggler source is essential to meet the 10^{14} ph./sec. flux target. This represents the central technical challenge of the beamline.
- Preliminary finite element analysis show that a directly cooled first mirror can support >6 kW total power while suffering tolerable slope errors.
- Preliminary FEA shows that a directly-cooled, “hockey puck” style mono crystal can support >2 kW with slope errors small compared to the Si(111) width.

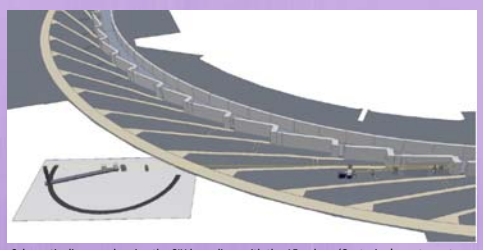
SOFT X-RAY INELASTIC SCATTERING (SIX)

Proposal Team: D. Arena[1], A. Baron[2], Y. Cai[1], Y.-D. Chuang[3], F. de Groot[4], J. Guo[3], J.P. Hill[1], S. Hulbert[1], C. McGuinness[5], R. Reininger[1], J.E. Rubenson[6], C. Sanchez-Hanke[1], T. Schmitt[7], **K. Smith[8]**
[1]Brookhaven National Laboratory, [2]SPring-8, [3]Lawrence Berkeley Laboratory, [4]Utrecht University, [5]Trinity College Dublin, [6]Uppsala University, [7]Paul Scherrer Institute, [8]Boston University

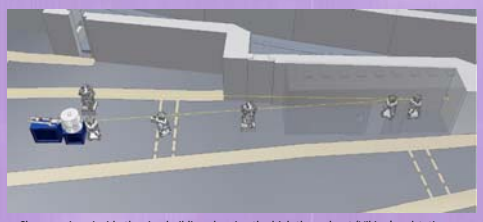
NATIONAL SYNCHROTRON LIGHT SOURCE II NEXT

TECHNIQUES AND CAPABILITIES

- Resonant inelastic scattering at unprecedented resolution (10 meV @ 1000 eV) to revolutionize study of low energy excitations in many important materials.
- Continuously tunable momentum transfer (q) to study the dispersion of excitations in condensed matter.
- Soft X-ray energy range (~260-2000 eV) to access transition metal L edges, rare earth M edges, and the K edges of C through Si.
- Focused beam (3x10 µm) to study small crystals / micron scale patterned device structures.
- Two endstations using an EPU source: an ultrahigh resolution 'Centurion' endstation (R=100,000) and a high throughput 'Viking' endstation (R=5000).

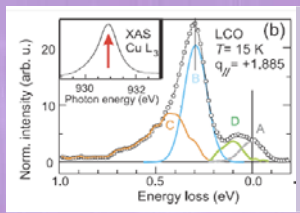


Schematic diagram showing the SIX beamline, with the 15 m long 'Centurion' spectrometer located in a satellite building.



Close up view inside the ring building showing the high throughput 'Viking' endstation.

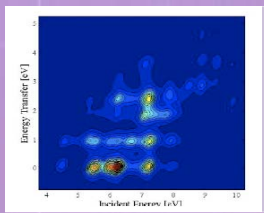
APPLICATIONS



RIXS spectra taken at the Cu L_3 edge, in La2CuO4, showing the elastic scattering (A), phonons (D), magnons (B), and multi-magnon processes (C). These data were taken at 100 meV resolution. The SIX beamline will have 10 meV resolution and the ability to continuously scan q, allowing non-cuprate and 3D systems to be studied.

Complex Materials for Advanced Technologies

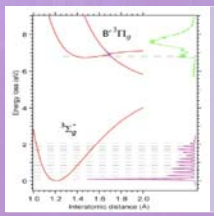
Enables the study of magnetic, orbital, phonon, and Kondo excitations in correlated electron materials and heterostructures-> potential applications in high Tc superconductivity, multiferroics, and CMR materials.



Charge transfer multiplet calculation of the RIXS plane for Co nanoparticles. At SIX, such planes will be able to be measured with the highest resolutions, *in-situ*. RIXS reveals important properties such as electronic structure, symmetry, and excitations.

Chemical and Energy Sciences

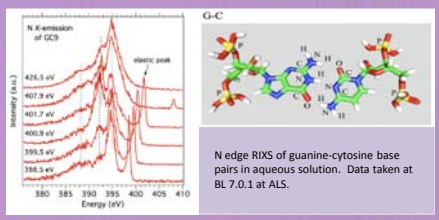
The size dependency of the electronic structure (which is a key factor determining reactivity) of nanoparticles holds the potential to create novel, tailored materials. RIXS allows both the occupied and unoccupied density of states to be measured *in-situ*.



Vibrational fine structure in the electronic ground atate and the first allowed state measured at the 1s-1π* resonance in O_2. These data were taken at the ADRESS beamline at the Swiss Light Source.

Atomic and Molecular Science

The nature of the RIXS process will serve as an experimental test-bench for advanced quantum chemical theory, including the behavior of highly excited states, ultrafast wave packet dynamics, and role of localization and symmetry.



N edge RIXS of guanine-cytosine base pairs in aqueous solution. Data taken at BL 7.0.1 at ALS.

Life and Environmental Science

The ability to study liquid systems in-situ will allow studies of active centers of large biomolecules in an aqueous environment, homogenous catalysis, and complex processes at mineral surfaces, for example.

ADDITIONAL INFORMATION

- Achieving the ambitious goal of 10^5 resolving power at 1 keV requires several key components and technical accomplishments, including:
  a. State-of-the-art gratings - VLS plane gratings with 0.05 µrad figure error.
  b. Micrometer level stability of several key optical components over distances of ~ 50 m.
  c. A 15 m large spectrometer located in a satellite building.
  d. High sensitivity soft x-ray detector capable of operating at near grazing incidence.

SOFT MATTER INTERFACES (SMI)

Proposal Team: E. DiMasi[1], A. Fluerasu[1], M. Fukuto[1], O. Gang[1], B. Hsiao[2], J. Kline[3], T. Koga[2], I. Kuzmenko[4], **B. Ocko[1]**, R. Pindak[1], M. Schlossman[4], H. Strey[2], D. Vaknin[5], L. Wiegart[1], T. Xu[6], K. Yager[1], L. Yang[1], J. Zhang[4]

[1]Brookhaven National Lab, [2]Stony Brook Univ., [3]NIST, [4]Argonne National Lab, Univ. of Illinois, [5]Ames Lab, [6]Univ. of California Berkeley

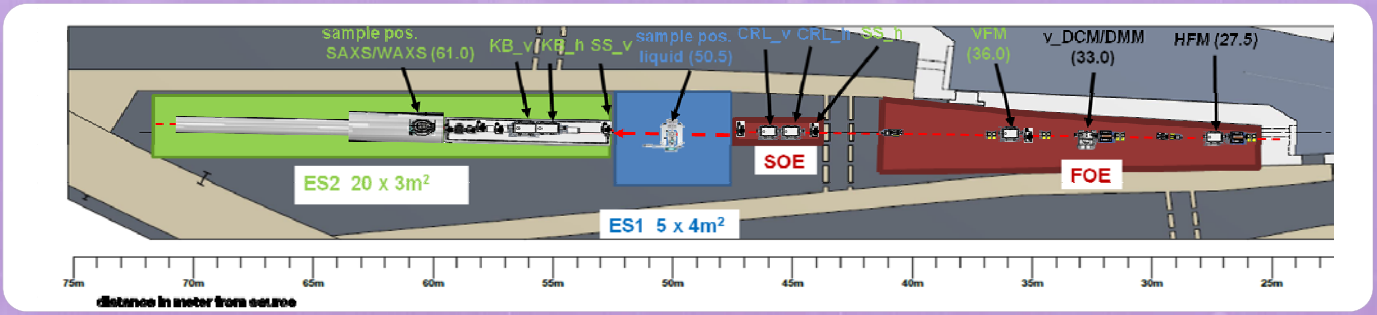
BROOKHAVEN NATIONAL LABORATORY

NATIONAL SYNCHROTRON LIGHT SOURCE II NEXT

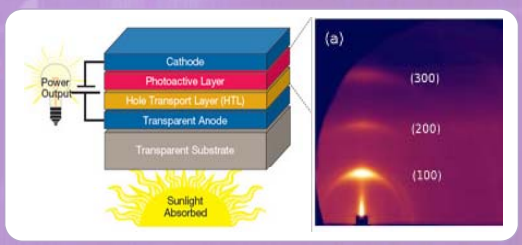
TECHNIQUES AND CAPABILITIES

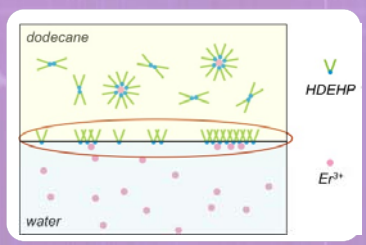
- Will enable in-situ studies of the structure of soft-matter interfaces using a variety of x-ray scattering methods
- Two different dedicated endstations will allow studies to be carried out at the solid/vapor, solid/liquid, liquid/vapor, liquid/solid and liquid/liquid interfaces
- The high brightness of NSLS-II will allow both very small x-ray beams and new opportunities for resonant studies in the tender x-ray regime

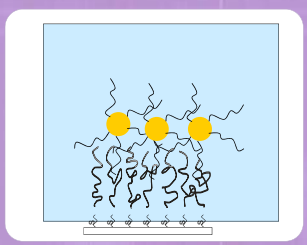
- GISAXS / GIWAXS / GIXD
- diffuse scattering, Bragg Rods
- fast x-ray reflectivity
- undulator beamline
- Energy Range: 2.1 – 27keV (P-Cd)
- Energy Tunability for Resonant Studies
- Beam Size: 5-500μm x 2-100μm (hxv)
- independent horizontal &vertical focusing



APPLICATIONS







ENERGY: To understand the microphase behavior and orientation of materials in thin film organic photovoltaic devices, a promising new class of energy materials.

To understand gas hydrate formation at the gas-water interface.

ENVIRONMENT: To improve our understanding of the extractant-mediated transfer of metal ions from an aqueous to an immiscible organic phase.

BIOMOLECULAR MATERIALS: To develop a fundamental understanding of self-assembly in order to create new materials.

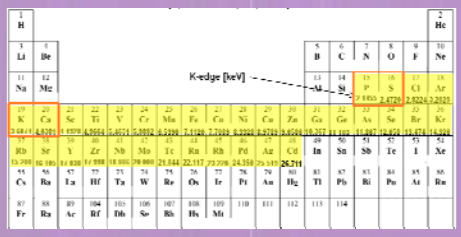
SPECIFIC PROJECTS / ADDITIONAL INFORMATION

NSLS-II Opportunities for "Tender"-Energy (2-5 keV)

- Sensitivity to key elements in soft/biomolecular materials, biological processes:
- P (2.15 keV), S (2.47 keV), Cl (2.82 keV), K (3.61 keV), Ca (4.04 keV)
- Liquid-Vapor Interface and from thin organic films

Grazing Angle Fluorescence (Depth dependence)

- fast changes in grazing angle via 2 crystal tilt stage



HIGHLY AUTOMATED MACROMOLECULAR CRYSTALLOGRAPHY BEAMLINE (AMX)

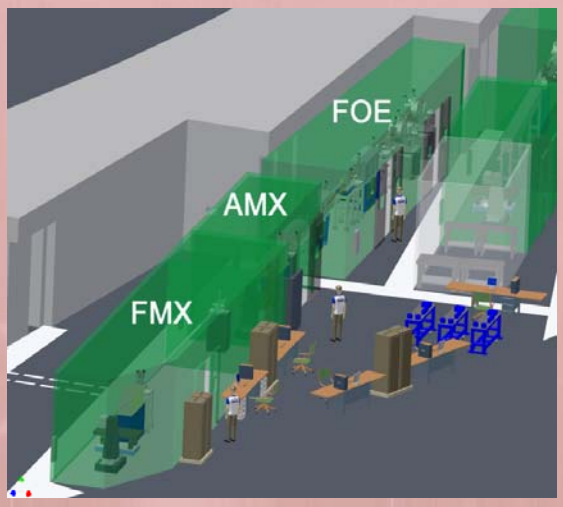
BROOKHAVEN NATIONAL LABORATORY

Proposal Team: M. Allaire[1], L. Berman[1], M. Chance[2], W. Hendrickson[3], A. Héroux[1], J. Jakoncic[1], A. Orville[1], H. Robinson[1], **D. Schneider[1]**, W. Shi[2], A. Soares[1], V. Stojanoff[1], R. Sweet[1]
[1]Brookhaven National Laboratory, [2]Case Western Reserve University, [3]Columbia University

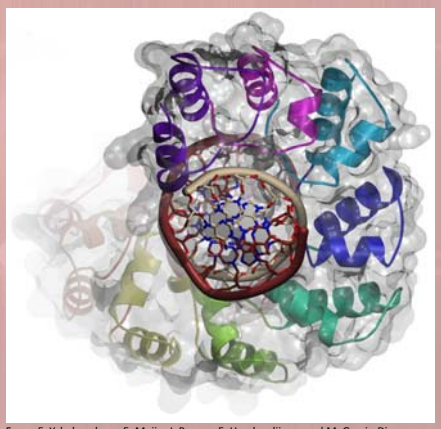
NATIONAL SYNCHROTRON LIGHT SOURCE II National Institutes of Health

MISSION

- The AMX beamline at NSLS-II will provide structural biologists with ready access to an advanced facility for the elucidation of structure and function of macromolecular complexes.
- Its high flux, tunable energy, and natively small focal spot will make it a crystallographer's preferred beamline.
- Its high degree of automation will provide a high throughput capability and invite remote participation.
- AMX will be part of a suite of specialized MX beamlines and complement the micro-focusing capabilities of FMX, the high energy resolution of NYX, and the optical spectroscopy capability of SM3.

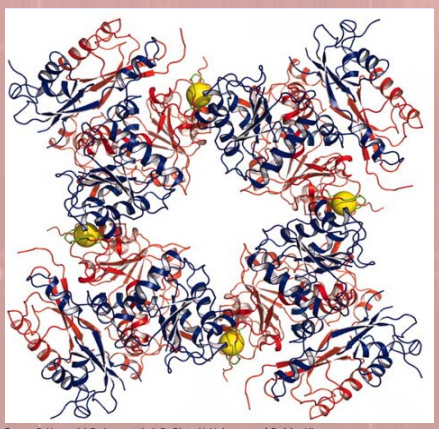


CAPABILITIES



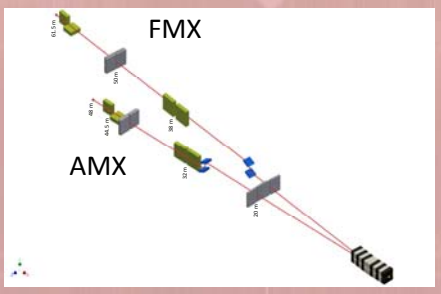
From: E. Yakubovskaya, E. Mejia, J. Byrnes, E. Hambardjieva, and M. Garcia-Diaz, Helix Unwinding and Base Flipping Enable Human MTERF1 to Terminate Mitochondrial Transcription, Cell 141, 982-993 (2010).

Optimized for structure analysis of large molecular complexes at unprecedented rates.



From: P. Yuan, M.D. Leonetti, A.R. Pico, Y. Hsiung, and R. MacKinnon, Structure of the Human BK Channel Ca^{2+}-Activation Apparatus at 3.0 Å Resolution, Science 329, 182-186 (2010).

Optimized to support efficient exploration of vast numbers of specimen and conditions.



Source: Canted U20 undulator
Optics: - Double crystal mono
- Deflecting mirrors
- K-B focusing mirrors
E-range: 4.8 – 21 keV
Flux in focal spot: ~10^{14} ph/s
Focal spot min: 5 x 6 μm^2
Focal spot range: 5 – 300 µm

CHALLENGES

This ALS-style crystal automounter at NSLS beamline X29 is fast in mounting crystals, but holds only 64 specimen in its dewar. By contrast, robotic systems based on six-axis robots such as a CATS system from IRELEC, may hold hundreds of specimen, but are slow in mounting and retrieving them.

The remarkable flux from a U20 undulator source will make it possible to complete data collections in seconds or less, thus leading to new crystallographic methods and practices.

To exploit this unique capacity we envision:

- A time-shared method of scheduling multiple projects for interleaved asynchronous data collections.
- A multi-stage robotic specimen change system including a fast crystal mounting robot and one to replenish specimen pucks through the hutch wall.
- Software-assisted crystallographic decision making.
- Development by the crystallographic community of new sample preparation methods and specimen carrier formats.

FRONTIER MACROMOLECULAR CRYSTALLOGRAPHY AT AN UNDULATOR BEAMLINE (FMX)

Project Team: M. Allaire[1], L. Berman[1], M. Chance[2], W.Hendrickson[3], A. Héroux[1], J. Jakoncic[1], Q. Liu[4], A. Orville[1], H. Robinson[1], D. Schneider[1], W. Shi[1], A. Soares[1], V. Stojanoff[1], D. Stoner-Ma[1], **R. Sweet[1]**

[1]Brookhaven National Laboratory, [2]Case Western Reserve Univ., [3]Columbia University, [4]New York Structural Biology Center

BROOKHAVEN NATIONAL LABORATORY

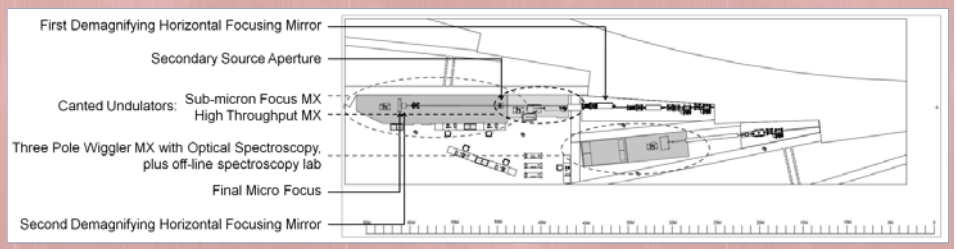
NATIONAL SYNCHROTRON LIGHT SOURCE II National Institutes of Health

THE MISSION FOR THIS PROJECT:

Macromolecular crystallography (MX) is in a Golden Age. Biologists can create crystals of huge, multi-compo-nent molecular assemblies. They deserve instruments to reveal structures of these assemblies. We are working now to create an MX beamline that will push the state of the art in x-ray optics and sample delivery, exploiting the finest properties of NSLS-II.

A tunable beam, down to one micron in dimension, with variable divergence, will address the most difficult problems in MX: small, weakly diffracting crystals, and very large unit cells. We will preserve beam coherence to make possible experiments we may not yet have conceived. Cryogenic automation will be at the state of the art. We will accommodate frontier experiments, for example serial crystallography.

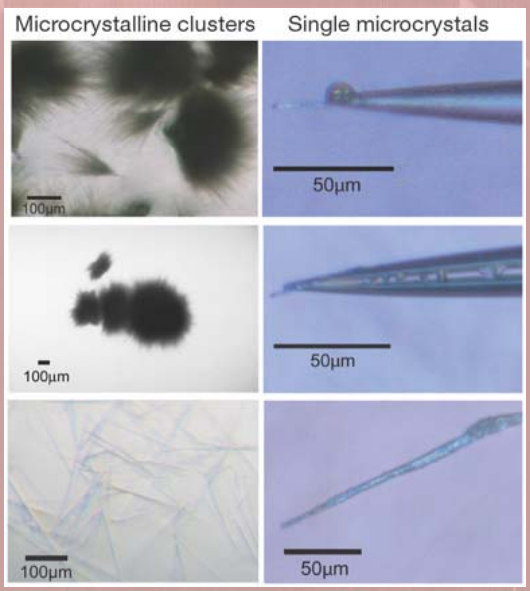
TECHNIQUE: Macromolecular Crystallography
SOURCE: Canted U20 In-vacuum Undulator
ENERGY: 5-20 keV; $\Delta E/E$ ~5×10^{-4}
BEAM SIZE: from 1x1 to 100x100 μm^2.
DIFFRACTION RESOLUTION: to 1 Å



The figure shows the basic design, with two-stage focusing. Horizontal beam properties are shown in the accom-panying table. We estimate that this beamline, viewing a U20 undulator source installed in an NSLS-II low-beta straight section, could deliver as much as 5×10^{11} – 1×10^{12} ph/sec at 12 keV into a 1 µm beam size with a horizontal angular divergence of 1 mrad. This flux is an order of magnitude higher than has been achieved in a 1 µm beam size.

Optical Component	*Location [m]*	*H-Size [µm]*	*H-Divergence [µrad]*
Source	0	66	45
Front End Slit	20	234	15
Horizontal Focusing Mirror	38	504	15 incident
Secondary Source Aperture	50	27	47
Second Horz Focusing Mirror	61	490	47 incident
Final Focal Point	61.5	≤ 1.5	≤ 1045

AN APPLICATION: TINY CRYSTALS



Prion diseases, such as Alzheimer's or scrapie, result when proteins such as β amyloid form un-natural structures in the brain. Because these structures are often strained, crystals may be long and very thin. Work like this will benefit from the 1µm beam.

Sawaya, … Eisenberg *et al.* Nature 447, 453-7 (2007).

OPPORTUNITIES & PROBLEMS

- ***Wide Focus Range***: We say that we'll be able to give a range of beam sizes in the range 1 – 100 µm.
  - How, exactly, will we make the change quickly?
- ***High-Energy Limit***: Damage of delicate macromol-ecules in the x-ray beam is a serious impediment to the quality of data.
  - Studies suggest that use of x-rays in the range 20-50keV ameliorates that damage.
  - The damage reduction may be even more effective when the beam size is in the 1 µm range.
  - How far should we try to go? The undulators aren't effective, and our detectors become much less efficient above 15keV.
- ***A Coherent Beam***: We would like to retain the coher-ence of this fine NSLS-II undulator beam for studies of morphology and strain in crystals in the 1µm size range.
  - How do we do this while still accomplishing other goals?
  - Here are things that come to mind: super-smooth mirrors; minimizing numbers of reflective surfaces; only one, very thin, vacuum window (diamond? Si_3N_4?)

HIGH BRILLIANCE X-RAY SCATTERING FOR LIFE SCIENCES (LIX)

Proposal Team: O. Bilsel[1], B. Hsiao[2], H. Huang[3], T. Irving[4], A. Menzel[5], L. Pollack[6], C. Riekel[7], J. Rubert[8], H. Tsuruta[9], **L. Yang[10]**

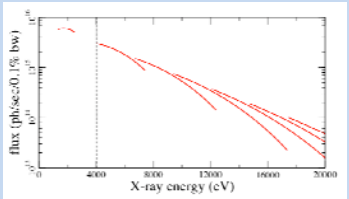
[1]University of Massachusetts, [2]Stony Brook University, [3]Rice University, [4]IIT, 5SLS, [6]Cornell University, [7]European Synchrotron Radiation Facility, [8]NEU, [9]Stanford Synchrotron Radiation Lightsource, [10]Brookhaven National Lab

BROOKHAVEN NATIONAL LABORATORY

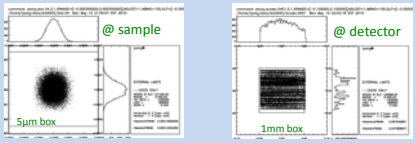
NATIONAL SYNCHROTRON LIGHT SOURCE II National Institutes of Health

TECHNIQUES AND CAPABILITIES

- Energy range 2-20keV using undulator source. Simultaneous SAXS/WAXS to cover 0.003-3Å^{-1} at 12keV with 1 micron spot size
- Time-resolved solution scattering with resolution of (1) microseconds to milliseconds using continuous-flow mixing (5μm x 10μm spot size) and (2) milliseconds using stopped-flow mixing or micro-drop mixing (50μm x 20μm spot size)
- Grazing incidence scattering, including anomalous scattering near P K-edge, from membrane structures in multiple bilayers as well as single lipid bilayers that contain membrane proteins (1μm vertical spot size)
- Micro-beam diffraction from biological tissue for imaging and tomography (1μm spot size)



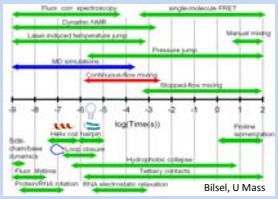
The X-ray flux captured by the primary focusing mirrors. The source is assumed to be a U23 located in a high-b straight section. A secondary focusing pair then further focuses the beam onto the sample as necessary.



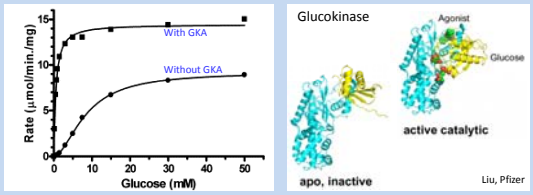
In the most challenging configuration (1 micron beam, low beam divergence to give q_{min} ~0.003A^{-1} with a safety factor of ~2.5), ~3% of the flux, or ~10^{13} photons/sec will be available at the sample.

APPLICATIONS

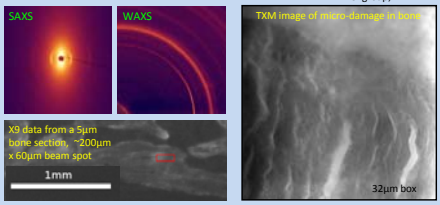
Protein and RNA folding



Structural change in enzymatic reaction



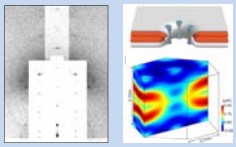
Micro-structures in bones



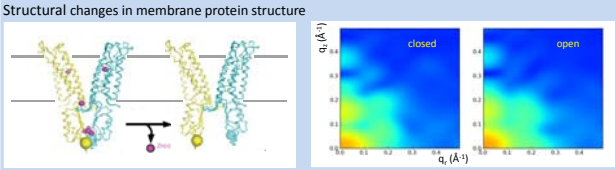
Time-resolved solution scattering with microseconds time resolution is useful for studying protein and RNA folding. With optimized flow cell design, the LiX will achieve time resolution of ~10 μs, a factor of 10 improvement compared to the current state of the art. Time resolution of milliseconds will be useful for studying the structural changes in enzymatic reactions. Time resolved measurements combined with equilibrium measurements provide a more complete picture of how enzymes carry out their functions.

X-ray diffraction using small beams is useful for mapping out structural features in heterogeneous samples. The 1-micron beam size provided by the LiX beamline will enable studies on small structures like micro damage in bones and engineered biomaterials such as collagen-like fibers. Scattering-based tomography also makes it possible to map out the 3-dimensional internal structure of the sample.

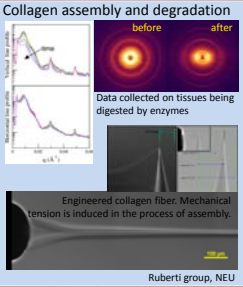
membrane pores induced by peptides



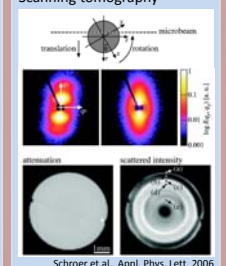
Structural changes in membrane protein structure



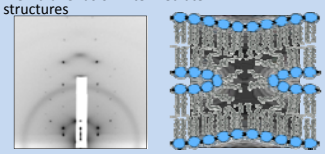
Collagen assembly and degradation



Scanning tomography



membrane fusion intermediate structures



Grazing incident diffraction has already been utilized to study the structures in multi-bilayers. In addition to extending the energy range for these measurements, the LiX beamline will also provide capabilities to study membrane proteins embedded within a single bilayer, in a solution-like state and under physiological conditions.

ADDITIONAL INFORMATION

The LiX beamline will be funded by the NIH. It will be highly complementary to the approved ABS beamline, which is optimized for high throughput X-ray scattering measurements of biomolecules in solution.

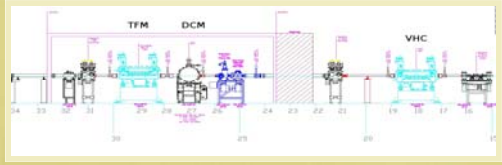
Beamline for Materials Measurement (BMM)

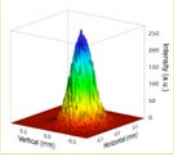
Proposal Team: **D. Fischer**, J. Woicik, B. Ravel
National Institute of Standards and Technology

NATIONAL SYNCHROTRON LIGHT SOURCE II approved: type 2

TECHNIQUES AND CAPABILITIES

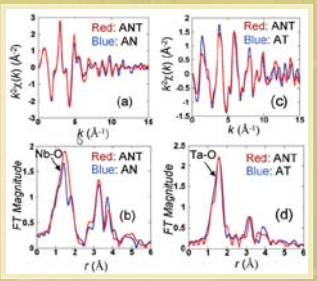
- The mission of BMM is to apply X-ray Absorption Spectroscopy (XAS) and X-ray Diffraction (XRD) to the materials science of important societal challenges in energy, health, environment, and national security; to establish structure/function relationships in advanced materials, often at the nanoscale; to accelerate the development of new materials into devices and systems with advanced functionality; and to promote innovation which enhances US industrial competitiveness.
- BMM uses a three-pole wiggler source along with a paraboloid collimating mirror, a Si (311) monochromator, and a toroidal focusing mirror to provide light to a highly usable end-station.
- The end-station is equipped to provide high-quality, high-throughput XAS coordinated with an eight-circle goniometer for XRD and constant-**q** spectroscopy measurements such as *refflexafs* or DAFS.

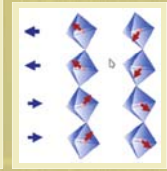




	Si(111)	Si(311)
flux at 10 keV	2.0×10^{12} ph./sec.	4.1×10^{11} ph./sec.
flux at 20 keV	5.9×10^{10} ph./sec.	1.2×10^{10} ph./sec.
spot size	0.18 mm x 0.18 mm	
energy range	4.5 keV to 25 keV	

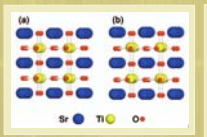
APPLICATIONS

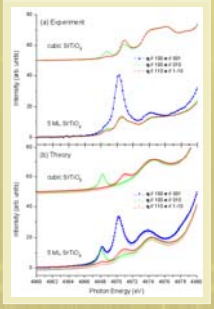




Structure/Function Relationships in Electronic Ceramics

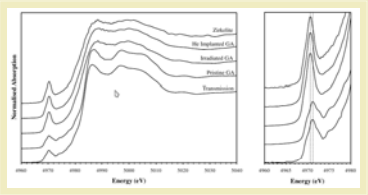
Device applications require concurrent optimization of many functional charac-teristics of materials via modifications of their crystal structures using complex chemistry. We found anomalous XAS results on the temperature dependence of the coordination geometry of Ag(Nb,Ta)O3, the only known system that exhibits large, temperature-stable dielectric constants and modest dielectric loss at microwave frequencies.





Strain Engineered Electronic Thin Films

Strain engineering via epitaxial thin-film growth effectively tailors the electronic and mechanical properties of a material. Industrial applications range from enhanced electron mobility devices to the realization of ferroelectric memory conjoined with silicon. Strain also alters both the nature and temperature of ferromagnetic, ferroelectric, and superconducting phase transitions. Here we show data and theory for cubic SrTiO3 and a 2 nm ferroelectric SrTiO3 thin film grown coherently on Si(001).



Radiation Damage in Nuclear Materials

A study using Grazing Angle XAS on beamline X23A2 was the first to quantify the effect of radiation damage on the coordination environment of key elements in zirconolite ($CaZrTi_2O_7$), a model system for actinide immobilization. Analysis of the XANES and EXAFS confirms a transformation to ~100% 5-fold coordination in the damaged surface layer. Grazing angle XAS studies like this will benefit from the higher flux of BMM as well as the superior optics and end-station instrumentation.

NIST INVESTMENT IN BMM

- The BMM monochromator has been procured and tested at NSLS.
- Four- and one-element silicon drift detectors with high-speed, pulse counting electronics are in use at X23A2.
- A low vibration, liquid helium cryostat suitable for use in conventional XAS, glancing angle XAS and diffraction is being commissioned at X23A2.
- Extensive, high-quality XAS hutch instrumentation is in use at X23A2.
- Collimating and focusing mirrors with multiple coatings for extended energy range have been designed and will be procured in FY12.

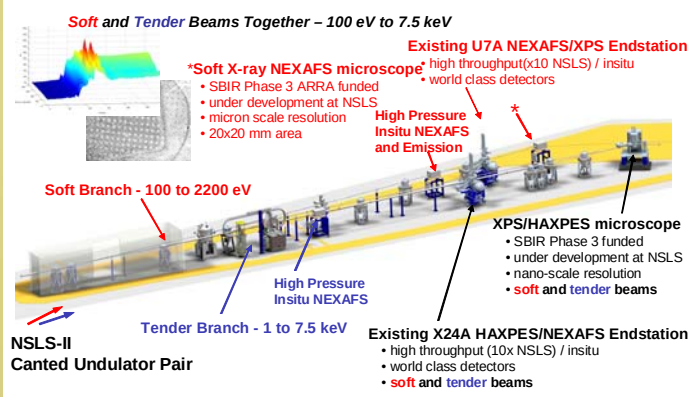
SPECTROSCOPY SOFT AND TENDER (SST)

Proposal Team: **Daniel Fischer**, Joseph Woicik, Bruce Ravel
National Institute of Standards and Technology

NATIONAL SYNCHROTRON LIGHT SOURCE II approved: type 2

TECHNIQUES AND CAPABILITIES

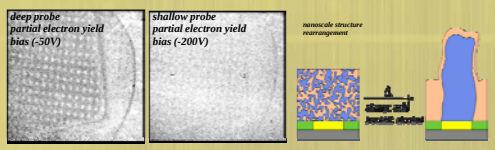
- The mission of SST is to apply X-ray Photoelectron Spectroscopy (XPS) and Near Edge X-ray Absorption Fine Structure (NEXAFS) spectroscopy to the materials science of important societal challenges in energy, health, environment, and national security; to establish structure/function relationships in advanced materials, often at the nanoscale; to accelerate the development of new materials into devices and systems with advanced functionality; and to promote innovation which enhances US industrial competitiveness.



- SST will have 6 unique world class NEXAFS/XPS experimental stations:
  2 full field microscopes, 2 automated high-throughput stations, and 2 in-situ high pressure stations.

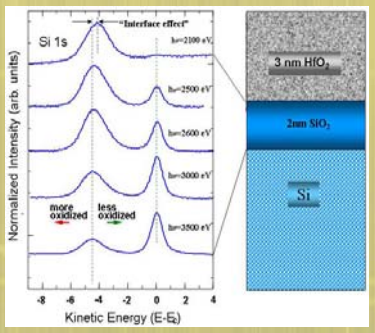
- Soft and tender beams enabling a continuous selection of X-rays from 100 eV to 7.5 keV at a common focal point in a single experiment (unique capability, enhancing depth selectivity in XPS or HAXPES).

APPLICATIONS



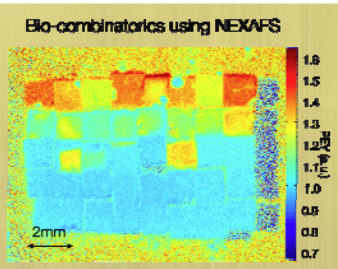
Accelerating Organic Photovoltaics with Soft and Tender Spectroscopy and Microscopy

The commercialization of OPV technology has been slowed by two key technical challenges: enhancing the power conversion efficiency, and preventing aging-related performance loss. To address these challenges requires precise measurements of materials composition and molecular orientation with sub-100 nm spatial resolution. These needs will be met by NIST's new soft and tender X-ray microscopes and beamlines for NSLS-II, which promises to greatly accelerate the development of high-efficiency, long-lasting solar modules with the potential to build a new domestic solar cell manufacturing industry in the U.S. and transform our energy production landscape in the coming decades.



Nanoscale Spectroscopy for Next Generation Semiconductor Microelectronics: CMOS and Beyond

Imaging the structure and chemistry of buried layers and interfaces of real device architectures is essential towards ultimate Complementary Metal Oxide Semiconductor (CMOS) scaling and beyond. Above shows Si 1s spectra from 3nm HfO_2/2nm SiO_2 sample recorded with variable kinetic energy high-energy XPS (VKE-XPS) illustrating depth profiling and an interface effect near the HfO_2/SiO_2 interface.



Designing Biomaterials with High Throughput Screening: Using the NIST NEXAFS Microscope

Interactions between solid surfaces and the biological environment play an important role in many areas of medicine. Examples include biocompatibility of implants, biomolecule separations, bacterial-induced corrosion, and biosensors. The NEXAFS Microscope allows for unprecedented high throughput screening of samples for biomedical applications. We rapidly evaluated different self-assembled monolayer (SAM) preparation methods by imaging arrays of 45 samples with a surface area of 1 mm^2 each (above, azobenzene surface density image of 1 hour was equivalent to the data typically collected during five days of serial data acquisition).

NIST INVESTMENT IN SST *(at NSLS for NSLS-II)*

XPS Full Field Magnetic Projection Microscope

Goal: (3D) chemical mapping of the structure of nanomaterials and nanodevices at all points within their volume.

✓SBIR Phase-III collaboration between NIST and R. Browning Consultants, development at NSLS(U4A).
- Super conducting magnetic projection lens
- 3 Patents granted, 4 Patent Applications.
- 100nm spatial resolution.
- Major improvement: Laboratory based Imaging XPS systems throughout the USA and world.(Lab spatial resolution 5000 nm, fixed X-ray energy)
- No high voltages; samples can be rough or insulating, large depth of field.

At NSLS-II: Nanoscale spatial and spectral XPS imaging over the full kinetic energy (100 eV to 7.5 keV).

✓ Receive soft and/or tender beams via two KB mirror pairs that have a common focal spot of 13 µm.

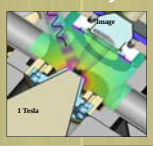
✓ Tuning the sensitivity from surface to bulk length scales ideally suited for photovoltaics and microelectronics.

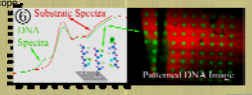
Large Area Imaging NEXAFS Microscope

Goal: NEXAFS full field microscope producing highly efficient, spectroscopic chemical and orientation maps of gradient samples, combinatorial arrays.
- **e.g. 1000s of compositional catalyst samples at once, and device arrays up to 4 cm^2 with micron scale resolution.**

✓SBIR working prototype at NIST's U7A beamline: 1 Tesla magnetic projection partial electron yield full field microscope.
- Image: 13x18 mm at 50 µm spatial resolution.
- Large depth of field (rotatable sample at right).

✓ Science case examples: Microscope images of combinatorial arrays of **ss-DNA** (right), batteries, OPV marine anti fouling designer surfaces and more.





NEXAFS microscope for NSLS-II ARRA funded (FY09 NIST SBIR Phase III, Synchrotron Research, Inc.

✓ Superconducting magnet (8T)

✓Under construction, commissioning at NSLS U8B beamline.

✓ At NSLS-II, receive soft x-rays (100-2200 eV) a pair of dithered mirrors, 4x4 cm high flux sample illumination.

Soft x-ray Emission and NEXAFS : Micro-calorimeter

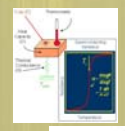
Micro-calorimeter High-resolution Soft X-ray Detector: Soft x-ray Emission and NEXAFS Spectroscopy at beamline U7A for NSLS I&II

Major Accomplishments:
- NIST-Boulder Quantum Sensors Project collaborating with NIST-Synchrotron Methods Group at NSLS
- Novel soft x-ray-fluorescence spectrometer, no wavelength-dispersive element, based on a array of 256 superconducting transition-edge microcalorimeters.
- **256 pixels, 200 um, and each capable of receiving up to 100 x-ray counts per second with energy resolution of 1 eV (FWHM); the total instrument will have collecting area of 10 mm^2 and count rate of 25 kHz**
- Fully-populated detector snout, has received first-light photons from a tube source at NIST-Boulder in July, 2010

Key Capabilities:

✓ High-resolution x-ray fluorescence (XRF) spectroscopy, like x-ray photoelectron spectroscopy (XPS), sensitive to occupied electronic states **(measurement of HUMO).** XRF probes deeply buried layers (100nm) from which photoelectrons cannot escape.

✓ Also for reducing background in FY NEXAFS **(measurement of LUMO).** Crucial for studying layer-interface chemistry that determines device properties. (e.g. gatestacks, organic PV, catalysts)



***Technical NIST Investment** – (1) Ruben Reininger, Scientific Answers and Solutions - Optical Design, (2) FMB Oxford Initial conceptual CAD Model, (3) Tender DCM and VLSPGM, purchased*

4 DIMENSIONAL EXTREME ENVIRONMENT (4DE^2)

Project Team: J. Bass[1], P. Burnley[2], A. Campbell[3], J. Chen[4], P. Dera[3], P. Duffy[5], W. Durham[6], L. Ecker[7], L. Ehm[8], S. Karato[9], L. Li[8], W. Lindquist[8], L. Margulies[7], M. Rivers[3], S. Shim[6], O. Tschauner[2], M. Vaughan[8], **D. Weidner[8]**, T. Wong[8], Y. Zhao[2]

[1]Univ. Illinois, [2]Univ. Nevada Las Vegas, [3]Univ. of Chicago, [4]Florida International Univ., [5]Princeton Univ., [6]MIT, [7]Brookhaven National Lab, [8]Stony Brook Univ., [9]Yale Univ.

BROOKHAVEN NATIONAL LABORATORY

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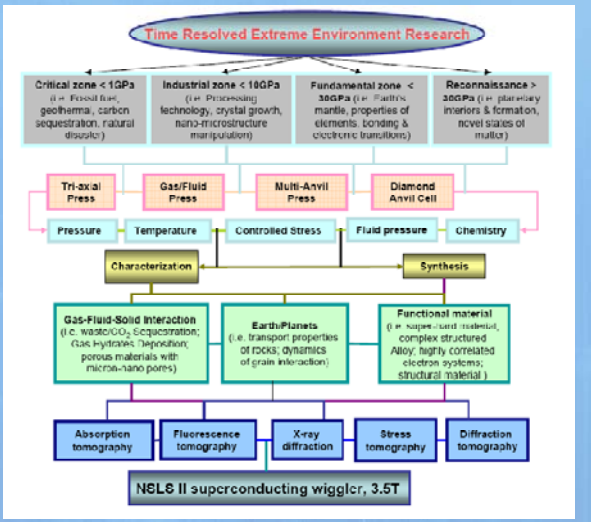
TECHNIQUES AND CAPABILITIES

4DE^2 at NSLS-II:

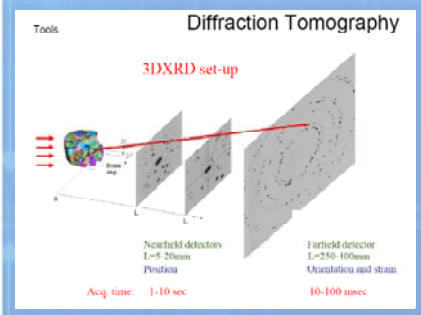
- Contain unique world-leading high pressure devices (200 MPa- 1000GPa)
- In-situ studies of materials under extreme conditions with both static and dynamic capability
- The continuous growth of high pressure devices combined with high brightness/low noise NSLS-II will set new frontiers in high pressure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

Examples of Science Areas & Impact:

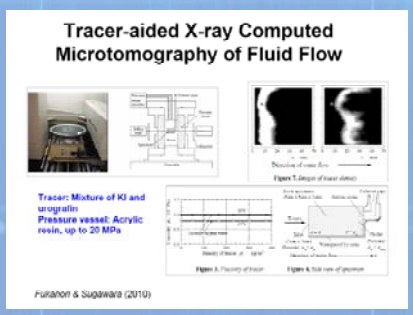
- **Functional Materials**: super-hard material; complex structured alloy; highly correlated electron systems; structural material
- **Earth and Planets**: transport properties of rocks; dynamics of grain interaction; phase transitions; kinetics, failure strength
- **Gas-Fluid-Solid Interaction**: waste/CO_2 sequestration; gas hydrates deposition; porous materials with micron-nanopores)



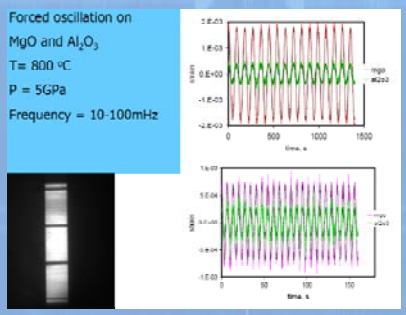
APPLICATIONS



The Three Dimensional X-ray Diffraction Microscopy (3DXRD) technique can provide complete microstructural information within a gauge volume containing up to 1,000's of individual grains. Monitoring this information as a function of time for samples under extreme conditions of pressure, temperature and stress provides insights into polycrystalline sample evolution.



Capturing and sequestering anthropogenic waste associated with weapon and energy production in geologic formations remains an outstanding planning, implementation, and remediation challenge. Imaging tools can be used to identify evolution of the sample porosity as a function of time for samples in natural environments.



Stress and strain can be monitored as a function of time yielding elastic, anelastic, plastic, and brittle properties of materials under extreme environments. This enables a wide range of studies relevant to planetary interiors and to material mechanical properties.

Material properties on scales from microns to thousands of kilometers and time scales from microseconds to billions of years are governed by the interaction of atoms, grains, and interfaces. Environmental conditions of pressure, temperature, and stress have played a critical role in probing these interactions in order to understand and create novel properties of materials demanded by today's science and technology development. We are proposing 4DE2, 4-Dimensional studies in Extreme Environments, as a beamline for the NSLS II that is to be dedicated to the study of materials at the extreme conditions of pressure, temperature, and deviatoric stress. The facility will be designed to help define the equilibrium state and to inform us about the evolution of systems towards this state. This beamline will enable monitoring of the three dimensional distribution of chemistry, stress, and structure as a function of time in order to model real world processes. The science that will be possible at this beamline will have applications to such diverse issues as: geologic sequestration of CO_2; material strength and performance; synthesis of new materials suitable for the next generation of energy systems; enhancing production of fossil fuels; understanding and prediction of natural disasters; and the origin and state of planets.

HIGHLY AUTOMATED BIOMOLECULAR SOLUTION SCATTERING (ABS)

Project Team: M. Allaire[1], D. Barrick[2], Z. Bu[3], J. Flanagan[4], S. Liu[5], L. Makowski[6], W. Peti[7], H. Tsuruta[8], **L. Yang[1]**, S. Yang[9]

[1]Brookhaven National Lab, [2]John Hopkins Univ., [3]City Univ. of New York, [4]Pennsylvania State Univ., [5]Pfizer Inc., [6]Argonne National Lab, [7]Brown Univ., [8]SLAC National Accelerator Lab, [9]Case Western Reserve Univ.

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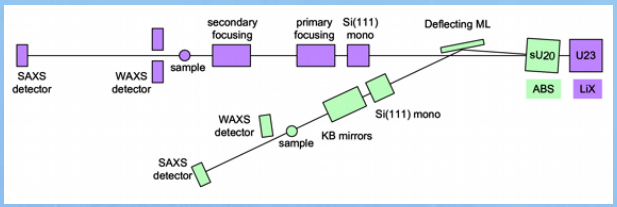
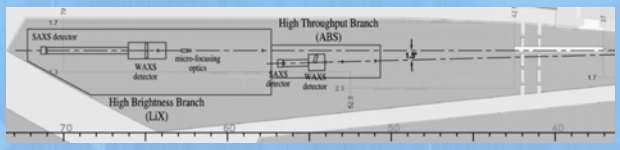
TECHNIQUES AND CAPABILITIES

- X-ray scattering from biological molecules in solution
- Simultaneous SAXS/WAXS to cover the continuous *q*-range of 0.005-3.0 Å^{-1} at 12keV
- Automated sample handling, data collection and processing at the rate of 1 sample per minute or faster (including measurement of buffer scattering and necessary cleaning)
- Optional in-line purification by gel filtration
- A 3PW source has been reserved for the ABS beamline. (undulator source preferred, see note in Additional Information)

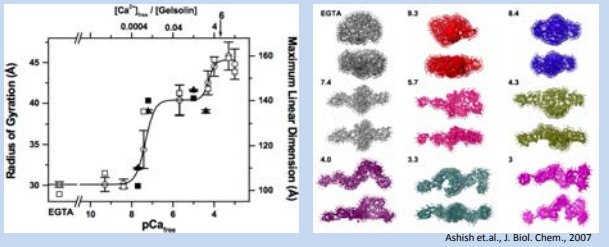


Schematic layout of LiX and ABS beamlines sharing the same straight section.



APPLICATIONS

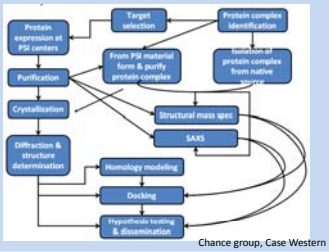
Structural biology



Ashish et.al., J. Biol. Chem., 2007

Solution scattering is becoming a standard tool for characterizing the structure of proteins under real-life conditions. The ABS beamline's high throughput capability will greatly increase the capacity for the structural biology community to carry out these measurements.

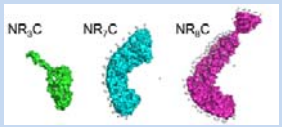
Structural Genomics



Chance group, Case Western

Information obtained from solution scattering can be integrated into the structural genomic pipeline to help understand the structure and functions of genomic products.

Protein engineering

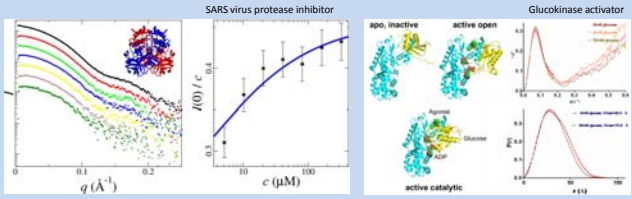


Stable protein scaffolding is envisioned to recruit enzymes into well-defined spatial sequence to provide synergetic activities.

Barrick group, Johns Hopkins

Solution scattering can also be used as a quality control tool to validate the structure of engineered proteins and protein complexes, such as the artificial cellulosome.

Drug discovery

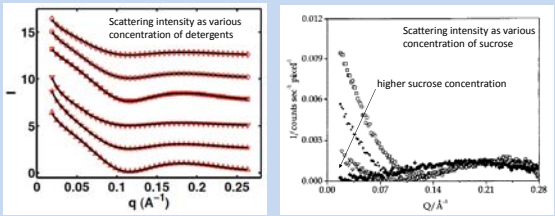


Graziano et.al., Biochem., 2004

Liu et.al., Pfizer

High throughput solution scattering can supplement chemical assay–based screens to facilitate drug discovery, providing structural information as direct evidence of whether the drug candidate is performing the expected function.

Membrane proteins



Lipfert et.al., J. Appl. Cryst., 2007

Bu et.al., Biophys. J., 1999

Measurements of membrane proteins in aqueous solution is complicated by the presence of detergents. Automated measurements are well suited for the necessary measurements at various concentrations of detergents or contrast-matching agents.

ADDITIONAL INFORMATION

The ABS beamline offers the opportunity to transition existing programs from the NSLS. R&D efforts are currently under way to improve the throughput of solution scattering measurements (beamline X9). As approved, ABS will utilize a 3PW source. However, the preferred source of this beamline is a short undulator, which could share the same straight section with the LiX undulator. The undulator source will provide higher flux and therefore higher throughput of measurement and the possibility of anomalous scattering measurements and in-line sample purification. An undulator source is also a better match to the existing beamline optical components that could be transferred from NSLS.

ADVANCED INFRARED MICROSPECTROSCOPY (AIM)

Proposal Team: L. Carr,[1] O. Chubar,[1] G. Ellis,[2] G. Flynn,[3] H.-Y. Holman,[4] S. Judex,[5] **L. Miller**,[1] J. Reffner,[6] G. Smith,[7] R. Smith,[1] E. Stavitski,[1] B. Weckhuysen,[8] P. Yu[9]

[1]Brookhaven National Lab, [2]Inst. Polymer Science & Technology Madrid, [3]SUNY Plattsburgh, [4]Lawrence Berkeley National Lab, [5]Stony Brook Univ., [6]John Jay College, [7]Indianapolis Museum of Art, [8]Utrecht Univ., [9]Univ. Saskatchewan

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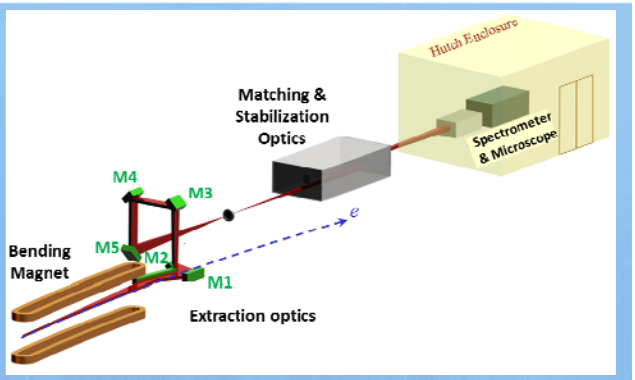
TECHNIQUES AND CAPABILITIES

- TECHNIQUE: Fourier transform infrared micro-spectroscopy (FTIRM)
- SOURCE: Dipole magnet
- ENERGY RANGE / RESOLUTION: 500 – 4000 cm^{-1} / 1 cm^{-1}
- SPATIAL RESOLUTION: ~1 – 5 μm (diffraction-limited)

AIM at NSLS-II:

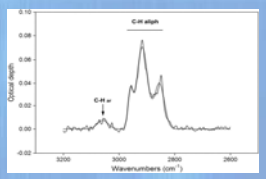
- Will enable high resolution studies of organic composition of materials by vibrational spectroscopy
- Measurements with sub-micromolar detection sensitivity and sub-micron spatial resolution
- The combination of the high brightness and low noise of NSLS-II with a confocal imaging system will be world leading

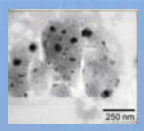


- The IR radiation will be collected (16 mrad vert x 50 mrad horiz) using a horizontal geometry, by positioning the extraction mirror (M1) inside the dipole vessel.
- This mirror will have a central slot to avoid overheating by the X-ray beam.
- The beam will be directed to a second flat mirror (M2) that will direct the beam vertically to a 90 degree toroidal mirror (M3) that will produce a 1:1 image of the source at the diamond window.
- The wedged CVD diamond window will separate the UHV conditions of the storage ring and the rough vacuum of the IR beamline.
- The beam pipe will be terminated with an IR-transparent window (KBr) to isolate the beamline vacuum from the ambient pressure of the microscope.

APPLICATIONS

Composition of Comet 81P/Wild 2 Samples Returned by Stardust

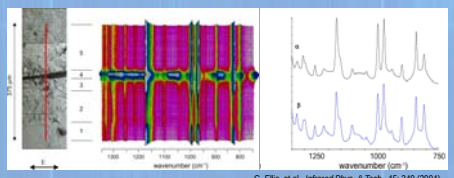




L.P. Keller, et al., *Science*, 314, 1728 (2006).

Particles from comet 81P/Wild 2 collected by the NASA Stardust spacecraft were investigated with SIRMS and X-Ray Fluorescence Microscopy. Particle sizes were 1 – 5 microns in size, requiring synchrotron-based FTIRM. Results showed indigenous aliphatic hydrocarbons similar to those in interplanetary dust particles thought to be derived from comets, but with longer chain lengths than those observed in the diffuse interstellar medium. Particles also contained amorphous silicates in addition to crystalline silicates such as olivine and pyroxene. The presence of crystalline silicates in Wild 2 is consistent with mixing of solar system and interstellar matter. With NSLS-II, improved S/N for even smaller particles and minor substituents.

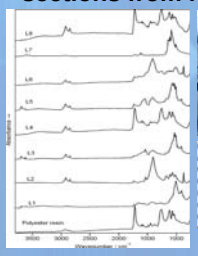
Nature of the crystalline interface in polymer-fiber composites

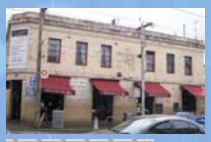


G. Ellis, et al., *Infrared Phys. & Tech.*, 45: 349 (2004).

Fiber-reinforced semi-crystalline thermoplastic polymers are of great technological importance and the interfacial morphology can considerably modify the ultimate properties of the composite material. A synchrotron IR source is necessary because the crystalline domains are between 5 - 50 μm, and sometimes smaller. With polarized SIRMS, both α and β iPP polymorphs in the interface region of sheared LCP-fibre / iPP model composites can be distinguished. When the fiber has been sheared at the isothermal crystallization temperature of 140°C, distinct regions are visible along the line indicated in the figure. With NSLS-II, finer interface regions and smaller spectral differences can be examined, including in situ analysis as a function of time, temperature, etc.

Microanalysis of paint cross-sections from historical landmarks

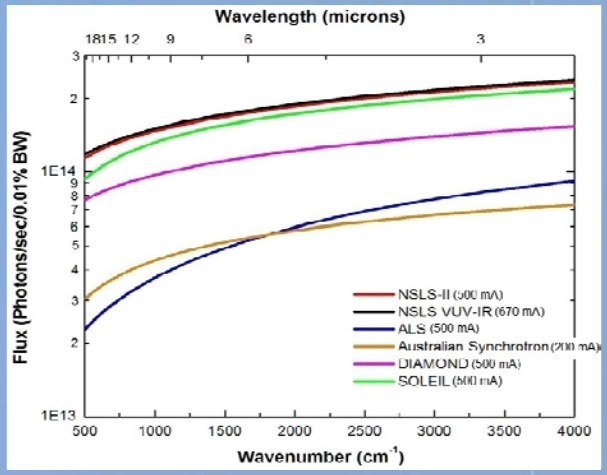




R. Sloggett, et al., *Vibr. Spectr.*, 53: 77-82 (2010).

The Provincial Hotel in Melbourne, Australia is a landmark building that represents the changing socio-economic role of Melbourne inner city pubs. The building supports painted signage from the 1880s through the 1950s. The pigment and binder used in each layer remain unknown and reflect the history of paint technology over the last century. Some paint layers and binders are <10 μm thick, requiring a synchrotron source. Representative Ge-ATR infrared spectra obtained from the polyester support and the regions L1–L8 show distinct layers. The Ge-ATR objective is a low-throughput technique that benefits from the synchrotron brightness and improves the spatial resolution by 4-fold. With NSLS-II, thinner layers and minor components can be studied.

BEAMLINE PERFORMANCE: WHY NSLS-II?



Improved signal:

- IR signal depends upon ring current and the ability to extract IR photons from the ring (i.e. extraction aperture).
- The 500 mA current of NSLS-II provides a high IR flux.
- The NSLS-II dipole magnet design can extract 16 mrad (vert) x 50 mrad (horiz), which permits 90% transmission of mid-IR photons (to 500 cm^{-1}).
- Thus, IR flux at the sample will be world-leading at NSLS-II.

Reduced noise:

- Synchrotron IR light at NSLS is noisier than the conventional globar (thermal) source. This is typical of older facilities; at the NSLS, the electron beam motion is ~50 μm.
- The electron beam motion at NSLS-II will be <10% of its size, which will be < 1 μm. This stability is expected to provide at least a 10-fold improvement in signal-to-noise (S/N) ratio over the NSLS VUV-IR ring.

COHERENT DIFFRACTION IMAGING (CDI)

Proposal Team: P. Evans[1], P. Fuoss[2], C. Jacobsen[2], I. McNulty[2], C. Rau[3], **I. Robinson[4]**, O. Shpyrko[5], J. Wall[6]

[1]University of Wisconsin, [2]Argonne National Laboratory, [3]Diamond Light Source Ltd, [4]University College London, [5]University of California at San Diego, [6]Brookhaven National Laboratory

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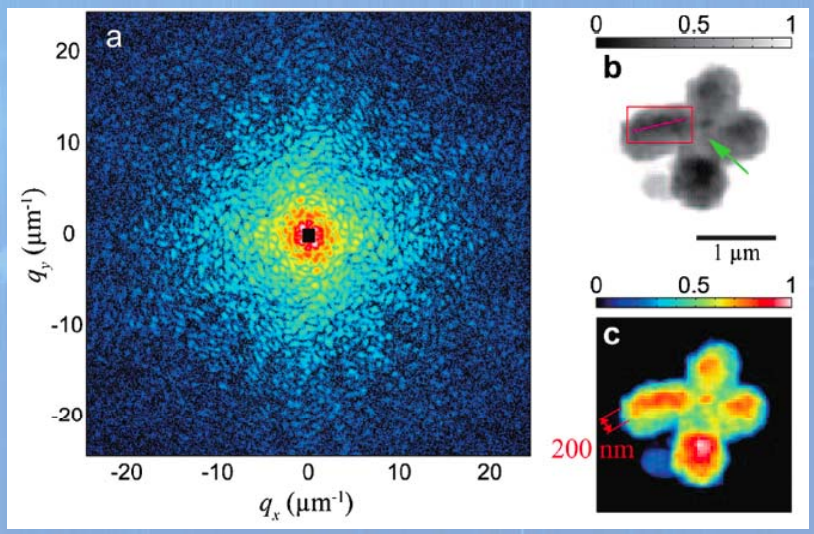
TECHNIQUES AND CAPABILITIES

- Exploitation of coherence to obtain real-space sample information
- Canted IUV20 undulators (two branches) low-β
- Long beamlines (200m) for maximum demagnification
- Long hutches, stable floors, isolated from ring building
- Monochromatic beam 2.5-10keV in **Branch A** (in-line CDI: **Biology**)
- Monochromatic beam 8-20keV in **Branch B** (Bragg CDI: **Materials**)
- Cryo sample manipulation in vacuum on branch A
- KB optics and ultra precise goniometer on branch B
- Diffraction imaging of cryo-frozen cells, organelles and tissues
- Diffraction imaging of crystal shapes in 3D on nm scale

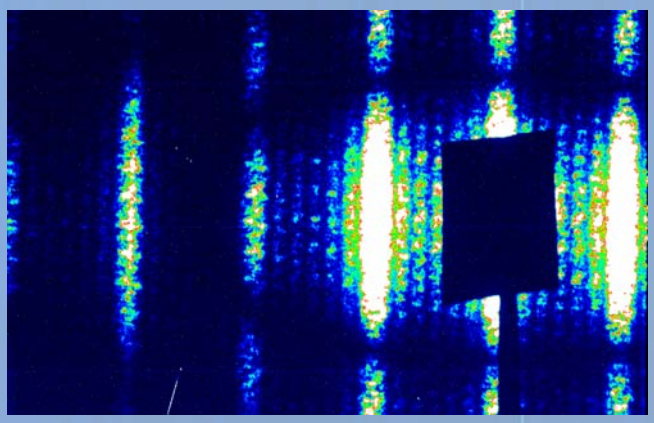
APPLICATIONS



CDI imaging of a human chromosome, Y. Nishino et al PRL 102, 018101 (2009)

- Imaging of strain fields inside crystals as 3D phase maps
- Time evolution of shape/strain under working conditions
- Manipulation/deformation/ indentation on the nm scale
- Ptychographic imaging for domains in ferroelectric materials
- Ptychographic imaging of biological samples using phase contrast, dark-field and phase encoding methods
- Applications in nanoscale semiconductor devices, strain engineering, catalysis and energy materials

SPECIFIC PROJECTS / ADDITIONAL INFORMATION



- Plans to develop novel imaging capabilities, notably using phase modulation for phase contrast
- Customised lithographic gratings will be inserted to modulate the beam (structured illumination) and scanned
- Example shown is diffraction from a "Collagen Phase-plate" by Felisa. Berenguer and Richard Bean (UCL)

COMPLEX MATERIALS SCATTERING (CMS)

Proposal Team: C. Burger[1], K. Cavicchi[2], E. DiMasi[3], A. Fluerasu[3], S. Fraden[4], M. Fukuto[3], O. Gang[3], B.S. Hsiao[1], R.J. Kline[5], S. Kumar[6], O. Lavrentovich[6], B. Ocko[3], R. Pindak[3], M. Rafailovich[1], R.A. Register[7], S. Sprunt[6], H.H. Strey[1], B.D. Vogt[8], W.-L. Wu[5], L. Wiegart[3], **K.G. Yager[3]**

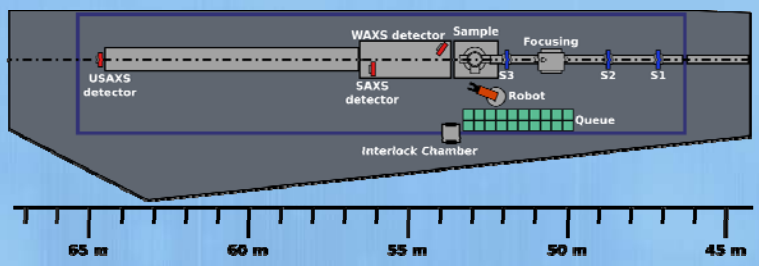
[1]Stony Brook University, [2]University of Akron, [3]Brookhaven National Lab, [4]Brandeis University, [5]NIST,[6]Kent State University, [7]Princeton University, [8]Arizona State University

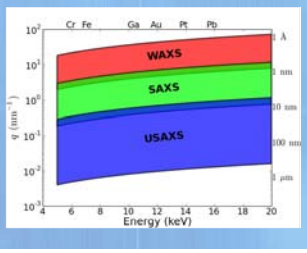
BROOKHAVEN NATIONAL LABORATORY

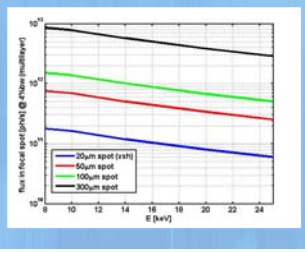
NATIONAL SYNCHROTRON LIGHT SOURCE II approved beamline

TECHNIQUES AND CAPABILITIES

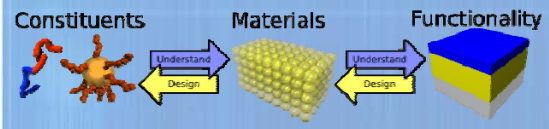
- Small- and wide-angle x-ray scattering on 3PW source, in transmission and reflection mode: **USAXS, SAXS, WAXS, GISAXS, GIXRD**
- **High-throughput** x-ray scattering for intelligent exploration of vast parameter spaces
- **Versatile** sample environment for stimuli-responsive and *in-situ* experiments
- **Broad *q*-range** (4×10^{-4} to 7.0 Å^{-1}) to study complex, hierarchical materials, including next-generation nanomaterials
- Microbeams and energy tuning (5 to 20 keV) for heterogeneous **sample mapping**

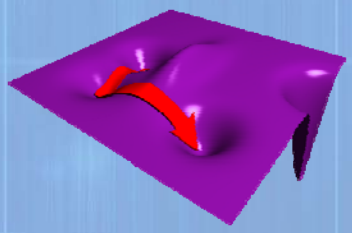


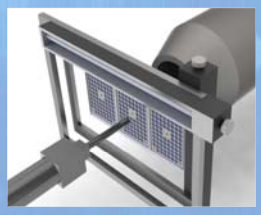


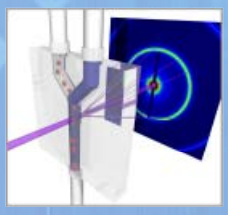


KEY CONCEPTS







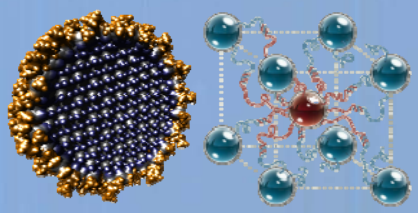


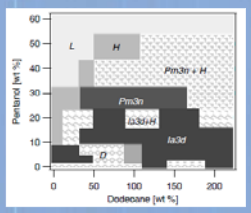
Rational materials design:
- New materials are hierarchical, nanoscale, and multi-component
- More sophisticated materials science requires *design*
- Need understanding at all length-scales

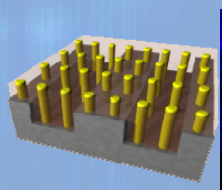
Non-equilibrium science:
- Path-dependent effects
- Processing history
- Stimuli/responsive
- Applied fields
- Engineering the energy landscape to *control order*

Automation and throughput:
- Robotic sample changer
- Explore vast parameter spaces using data feedback
- Integrated experimental controls (microfluidics, sample environments, mapping)

SELECTED APPLICATIONS







Synthesis: Using x-ray probes of structure to control reagent feeds, CMS will autonomously optimize synthesis (e.g. of nanoparticles)
Assembly: Tuning the self-assembly energy landscape with applied stimuli; understanding the resultant 3D hierarchical structures

Complexes and formulations: Massive parameter spaces will be explored to understand assembly and control formulation properties
Polymers: *In-situ* study of polymers under stress and flow will shed light on polymer crystallization

Devices: Studies of stimulated and direct self-assembly, e.g. in DNA lattices or block-copolymer nano-lithography, will pave the way for next-generation device architectures
Energy: High-performance materials for, e.g., organic solar cell, batteries, supercapacitors, fuel cells

FRONTIER SYNCHROTRON INFRARED SPECTROSCOPY BEAMLINE UNDER EXTREME CONDITIONS (FIS)

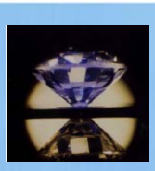
Proposal Team: L. Carr[1], D. Dolan[2], R. Hemley[3], S. Jacobson[4], S. Karato[5], **Z. Liu[3],** W. Panero[6], M. Pravica[7], and T. Zhou[8]

[1]Brookhaven National Laboratory, [2]Sandia National Laboratories, [3]Carnegie Institution of Washington, [4]Northwestern University, [5]Yale University, [6]Ohio State University, [7]University of Nevada, [8]New Jersey Institute of Technology

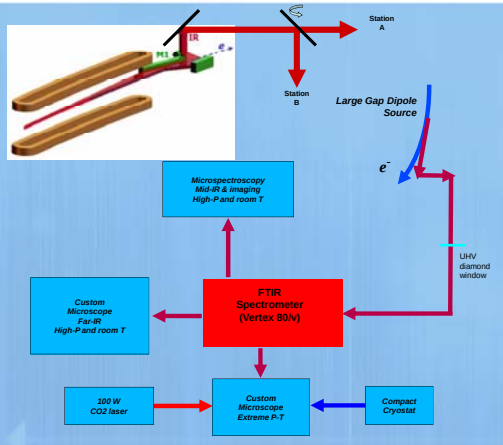
BROOKHAVEN NATIONAL LABORATORY

NATIONAL SYNCHROTRON LIGHT SOURCE II approved beamline

TECHNIQUES AND CAPABILITIES



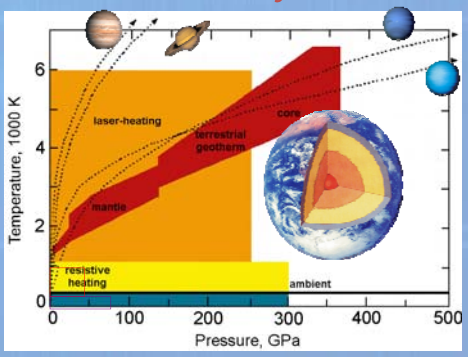
- **TECHNIQUE(S):**
  Fourier transform infrared spectroscopy;
  Raman and visible spectroscopy;
  Diamond anvil cell techniques for static high pressure;
  Gas-gun launchers for dynamic compression;
  Cryogenic techniques combined with DACs;
  Laser heating techniques combined with DACs.
- **SOURCE:** 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



Approved FIS large-gap IR beamline at NSLS-II: Beam extraction and endstations. Station A will be an integrated and dedicated infrared beamline for experiments at simultaneous high pressure and variable temperature with the following capabilities:
Infrared spectroscopy at high-pressure and ambient temperature
In-situ laser heating infrared spectroscopy at high P-T conditions
In-situ spectroscopy at high pressure and low temperature
Station B will be a multi-purpose station for measurements under dynamic compression.

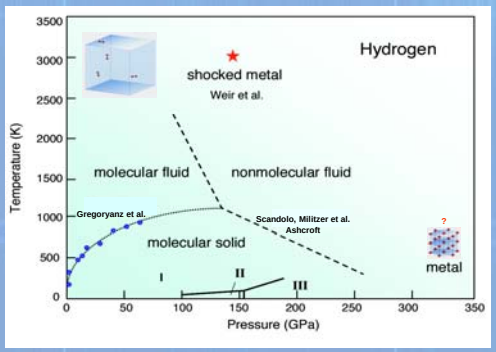
APPLICATIONS

Earth and Planetary Sciences



- Mimic the mantle extreme conditions and study the Earth's deep water cycle;
- Deep Carbon Cycle Research: study behavior of carbon-bearing materials in Earth's deep interior conditions.

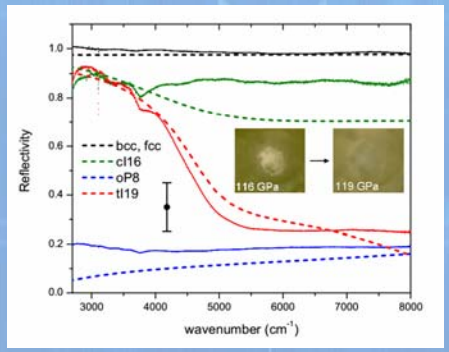
Hydrogen Metallization



Hydrogen phase diagram

FIS will enable *in-situ* optical studies of hydrogen metallization at multi-megabar pressure and low-T conditions due to the brighter and exceptionally stable infrared source at NSLS-II.

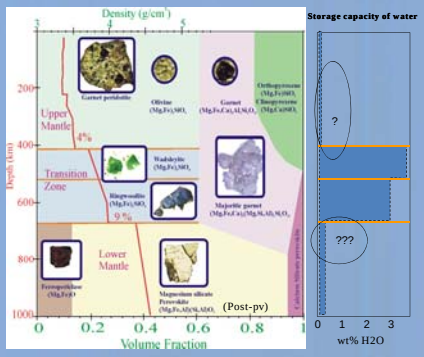
Material Sciences and Chemistry under Extreme Conditions



Anomalous properties of dense sodium

Synchrotron infrared spectroscopy on sodium shows a transition from a high reflectivity, nearly free-electron metal to a low reflectivity, poor metal in an orthorhombic phase at 118 GPa. An image of a sample across the cI16 → oP8 transition is shown.

SPECIFIC PROJECTS / ADDITIONAL INFORMATION



Earth's deep water cycle

- Infrared spectroscopy provides a key and often unique experimental approach with exceptional sensitivity to the O-H and C-O bonds and serves as an invaluable tool for studying Earth's deep water and carbon cycles. However, it is essential to extend diamond anvil cell techniques to higher temperatures and pressures with either external heating or laser heating techniques in order to directly probe thermodynamics, chemical diffusion, and molecular coordination under high *P-T* conditions. This requires brighter sources with diffraction-limited performance in the infrared range and superior stability. The capacity of the large-gap dipole at NSLS-II will provide an ideal infrared source for such studies to couple the high *P-T* DAC techniques with the FTIR spectroscopic measurements and to address these scientific questions and challenges related to Earth and Planetary Sciences;
- Metallization of hydrogen and hydrogen-rich materials such as methane, silane, and germane under extreme high pressure and low temperature;
- Optical studies of nano-crystalline materials and porous materials including BN nanotubes and zeolites/natrolites under extreme conditions for applications in energy efficiency, production, and storage.

HARD INELASTIC X-RAY SCATTERING (HIX)

Proposal Team: P. Abbamonte[1], E.E. Alp[2], A.Q.R. Baron[3], Y.Q. Cai[4], J.P. Hill[4], M. Greven[5], **Y.-J. Kim[6]**, H.-K. Mao[7], G.T. Seidler[8], L.J. Terminello[9], T. Thevuthasan[9]

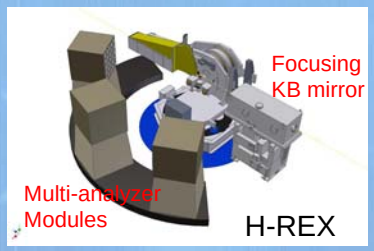
[1]Univ. of Illinois, [2]Argonne National Laboratory, [3]RIKEN SPring-8, [4]Brookhaven National Laboratory, [5]Univ. of Minnesota, [6]University of Toronto, [7]Carnegie Institution of Washington, [8]Univ. of Washington, [9]Pacific Northwest National Laboratory

BROOKHAVEN NATIONAL LABORATORY

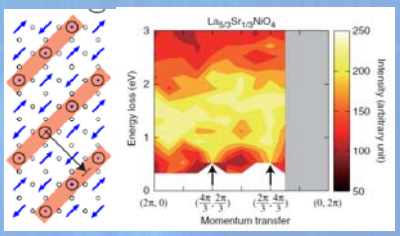
NATIONAL SYNCHROTRON LIGHT SOURCE II approved beamline

TECHNIQUES AND CAPABILITIES

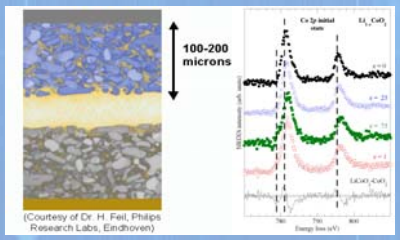
- Source: Undulator (high β)
- Energy Range: 2.9 keV – 15 keV
- H-REX: 10-50 meV resolution for Non-resonant IXS (NIXS) and Resonant IXS (RIXS)
- I-REX: 200-300 meV resolution for Non-resonant IXS (NIXS) and X-ray Raman Scattering (XRS)
- Excellent focusing, compatible with latest high-pressure diamond anvil cells (DAC).
- Sapphire and quartz analyzers
- High brightness of NSLS-II and multiple-analyzer system will allow measurement time comparable to current lower resolution instruments
- Analyzer temperature scan will be implemented



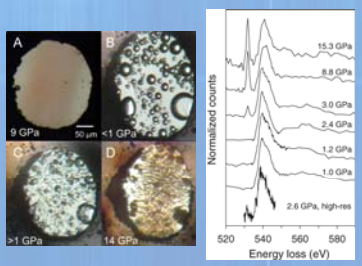
APPLICATIONS



Momentum dependence of charge excitations in $La_{5/3}Sr_{1/3}NiO_4$ due to charge stripes (shown on the left) measured with Ni K-edge RIXS. This measurement was done at the APS with the energy resolution of 130 meV. HIX will enable study of electronic (charge, orbital, and spin) excitations at much lower energy scale (< 50 meV) for much wider range of materials. [Wakimoto et al., Phys. Rev. Lett. 102 157001 (2009).]

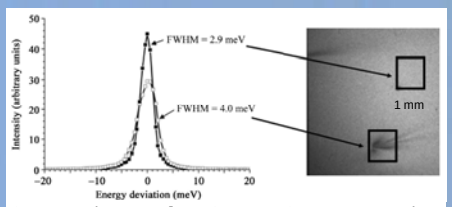


Complex morphology of battery electrodes, and the electronic structure change of Li-CoO_2 during charge-discharge cycling studied with NIXS, a bulk-sensitive in-situ probe. I-REX will enable NIXS/XRS studies of low-energy excitation spectra for a wide range of elements such as K-edges for C, and O and L-edges for 3d transition metals with energy resolution comparable to core-hole lifetime. [G. T. Seidler et al., unpublished.]

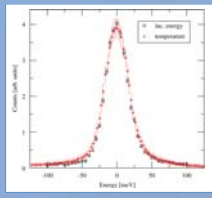


Photomicrographs of two diamond anvil cell (DAC) samples (left) and the XRS data of the H_2O sample at high pressure after 12 hours of irradiation, showing X-ray–induced dissociation of H_2O and formation of an O_2–H_2 alloy at high pressure. HIX will be compatible with >100GPa DAC, enabling the study of bonding changes in C and O, electronic structure of fluid/solid H/He [W. L. Mao et al., Science 314, 636 (2006)]

ADDITIONAL INFORMATION



Quartz analyzer test [J. P. Sutter, A. Q. R. Baron, et al. J. Synch. Rad. **13**, 278 (2006)



Sapphire analyzer test (31 meV FWHM over full analyzer of 20 mm diameter). [H. Yavas, E. E. Alp, et al., Nucl. Instr. and Meth. A **582**, 149 (2007).]

- HIX will also play a significant role in *actinide* spectroscopy. Its energy range (down to ~3 keV) will allow access to actinide M edges, which couple directly to the 5f electrons, enabling complementary 3d ➔ 5f RIXS or RXES studies. This will be also important for studying *fission materials* for energy research.
- HIX will enable in-situ studies of supercritical fluids, such as CO_2, which is crucial for carbon sequestration technologies.
- High-resolution required for HIX science case relies on the development of analyzers based on quartz (SiO_2) and sapphire (Al_2O_3) technologies; feasibility of using these materials for analyzers has been recently demonstrated as shown in the figures.

FULL-FIELD INFRARED SPECTROSCOPIC IMAGING (IRI)

Project Team: L. Carr,[1] O. Chubar,[1] G. Ellis, G. Flynn,[3] H.-Y. Holman,[4] S. Judex,[5] **L. Miller**,[1] J. Reffner,[6] G. Smith,[7] R. Smith,[1] E. Stavitski,[1] B. Weckhuysen,[8] P. Yu[9]

[1]Brookhaven National Lab, [2]Inst. Polymer Science & Technology Madrid, [3]SUNY Plattsburgh, [4]Lawrence Berkeley National Lab, [5]Stony Brook Univ., [6]John Jay College, [7]Indianapolis Museum of Art, [8]Utrecht Univ., [9]Univ. Saskatchewan

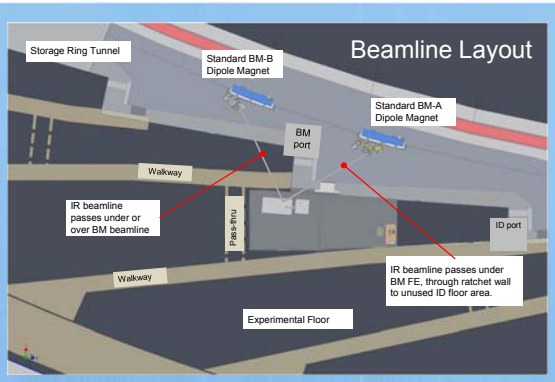
NATIONAL SYNCHROTRON LIGHT SOURCE II approved beamline

TECHNIQUES AND CAPABILITIES

- TECHNIQUE: Fourier transform infrared spectroscopic imaging with a 64x64 focal plane array detector
- SOURCE: Dual dipole magnets
- ENERGY RANGE / RESOLUTION: 500 – 4000 cm^{-1} / 1 cm^{-1}
- SPATIAL RESOLUTION: ~1 – 5 μm with pixel oversampling and image deconvolution

IRI at NSLS-II:

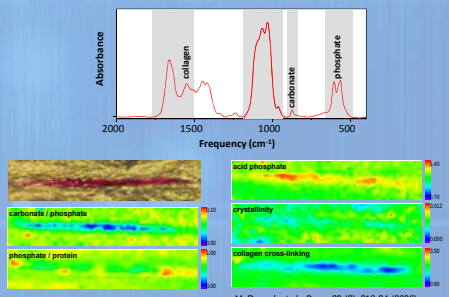
- Will enable in-situ studies of organic composition of materials by vibrational spectroscopy
- Measurements from microseconds to days with micromolar detection sensitivity and sub-micron spatial resolution
- The combination of high brightness and low noise of NSLS-II with a high throughput imaging system will be world leading



- Flexibility due to the unique extraction geometry of the M1 mirror, and the ease of steering infrared radiation.
- Since this beamline will utilize IR radiation from two dipoles, both a "BM-A" and "BM-B" dipole magnet will be used.

APPLICATIONS

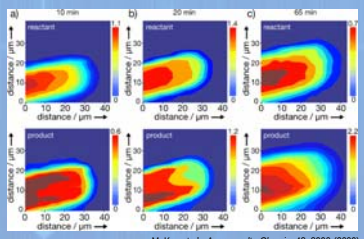
Bone Composition Differs in Microcrack Areas



M. Ruppel, et al., *Bone*, 39 (2): 318-24 (2006).

Bisphosphonates are current the most common treatment for osteoporosis but microdamage increases with high doses. Microcracks are 1 – 5 microns wide, requiring a synchrotron IR source to probe their composition. Results showed that bone composition was different in microcrack region. With too much accumulation of microdamage, the quality of bone may be reduced. With NSLS-II, lower drug doses, smaller cracks, earlier changes in composition.

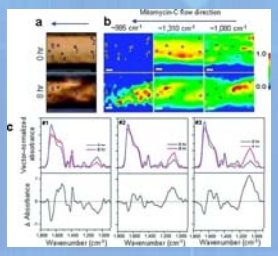
In Situ Synchrotron IR Imaging to Sulfur Removal in Fuel by Zeolites



M. Kox et al., *Angewandte Chemie*, 48, 8990 (2009).

The combination of label-free CARS and SIRMS were used to study the multidimensional distribution of thiophene reactants and products in zeolite crystals. The accumulation of active species in specific regions of the crystals illustrates the importance of diffusion barriers and capillary forces imposed by the pore network for the catalytic activity. This unique correlative approach is important for understanding of catalytic reactions on the micrometer scale under reaction conditions, thus introducing state-of-the-art chemical imaging techniques to the field of heterogeneous catalysis. With NSLS-II, reactions can be studied in real time to also determine rates as a function of crystal type, size, porosity, temperature, etc.

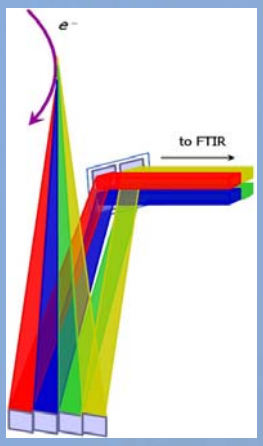
Real-Time Chemical Imaging of Bacterial Activity in Biofilms Using Microfluidics

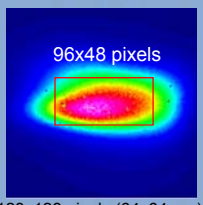


H.Y. Holman, et al., *PNAS*, 106, 12599 (2009)
H.Y. Holman, et al., *Anal. Chem.*, 81, 8564 (2009)

Real-time chemical imaging of bacterial activities can facilitate a comprehensive understanding of the dynamics of biofilm structures and functions. Open-channel microfluidic systems can circumvent the water-absorption barrier to study the developmental dynamics of bacterial biofilms. The spatial and temporal distributions of mitomycin-C uptake within a 1 day old *E. coli* biofilm at 985 cm^{-1} (DNA-MMC adducts), 1310 cm^{-1} (protein amide III), and 1080 cm^{-1} (polysaccharides). Scale bars = 10 μm. With NSLS-II, real-time studies can be performed on a large field of view with excellent spatial resolution to generate time-lapsed movies of reactions; better S/N will be necessary for small Δabsorbance for earlier time points and/or more subtle differences.

BEAMLINE PERFORMANCE: WHY NSLS-II?





128x128 pixels (64x64 μm)

Before PSF Deconvolution

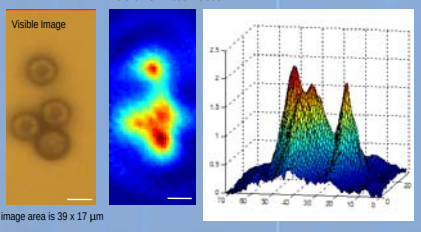
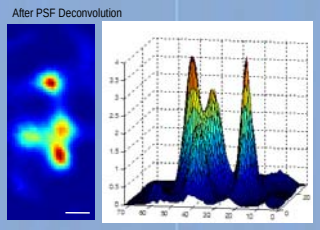


image area is 39 x 17 μm

After PSF Deconvolution



- With 90 mrad (H) at NSLS, we illuminated 4 distinct source points (Θ_v = 25 mrad at λ = 6 μm) or 96x48 pixels (36x16 μm area).
- With 50 mrad (H) at NSLS-II, a similar area is illuminated.
- With 2 NSLS-II dipoles, we will double the illumination area of the FPA to ~96x96 pixels (48x48 μm area).

- A 74X objective (0.65 NA) coupled to a 64x64 pixel, 40 μm pitch FPA yields a geometric effective pixel size of 0.54 μm. (approximately λ/10 at middle wavelengths => good for PSF deconvolution).
- High-brightness of synchrotron source should allow good S/N despite the small effective pixel area (can probably go another factor of 2 in magnification).
- (Left) FTIRI images of 6 μm-diameter polystyrene beads collected at Beamline U10B with a 74X objective (0.54 μm pixel size) and synchrotron IR source.
- (Top) Peak integration at 1450 cm^{-1}
- (Bottom) Same image after PSF Blind Deconvolution

POWDER DIFFRACTION BEAMLINE FOR *IN SITU* STUDIES OF STRUCTURAL AND CHEMICAL TRANSFORMATIONS (IXD)

Proposal Team: **J. Bai**[1], S.N. Ehrlich[2], J.C. Hanson[2], C.R. Hubbard[1], J.B. Parise[2,3], Jose A. Rodriguez[2], T.A. Tyson[4], J. Wang[2], X.-Q. Yang[2]

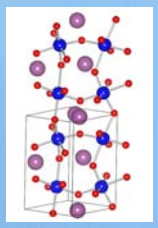
[1]Oak Ridge National Laboratory, [2]Brookhaven National Laboratory, [3]Stony Brook University, [4]New Jersey Institute of Technology

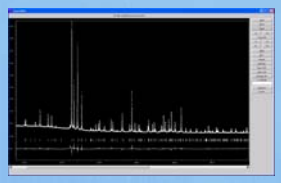
BROOKHAVEN NATIONAL LABORATORY

NATIONAL SYNCHROTRON LIGHT SOURCE II approved beamline

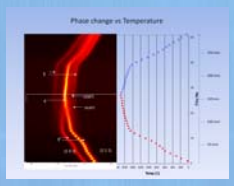
TECHNIQUES AND CAPABILITIES

- IXD is a sagittal focused powder diffraction beamline on a TPW port, with continuously tunable x-ray energy from 5 to 25 keV.
- Powder crystallography, including solving and refining crystal structures, quantitative analysis of phase fraction and size/strain analysis.
- *In situ* powder-diffraction measurements to study, with sub-minute time resolution, the structural, morphological and chemical transformations in materials following changes in temperature (10K to 2000K), pressure, reaction gas flow and charging states.
- Anomalous (or resonant) x-ray diffraction studies by tuning the x-ray wavelength near the absorption edges in order to distinguish neighboring elements and oxidation states.
- Diffraction at grazing incidence and reflectivity.
- Residual, thermal and growth-stress measurements.



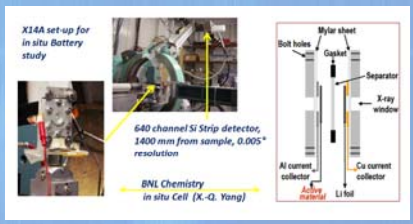


Monoclinic $ScMnO_3$: A New Multiferroic Material

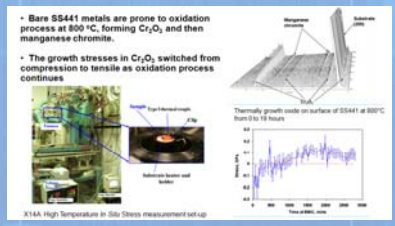


Quadrupole lamp furnace, up to 2000 K

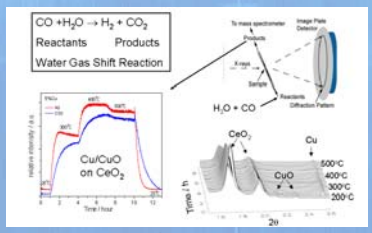
APPLICATIONS



Li-ion Battery Studies



In Situ Stress Measurement



Catalysis: Water Gas Shift reaction

In Situ X-ray Powder Diffraction Studies on Li-ion Battery Materials

One of the major science drivers for the proposed IXD beam line is to continue and enhance the current cutting-edge Li-ion battery studies at the NSLS. Lithium-ion batteries are the most promising candidate for use in hybrid electric vehicles (HEVs) and plug-in hybrid electric vehicles (PHEVs). Research topics in this field include the structure and morphology transformations in cathode materials during charge/ discharge processes, kinematics of chemical Lithium extraction, and *in situ* processing and synthesis of battery materials.

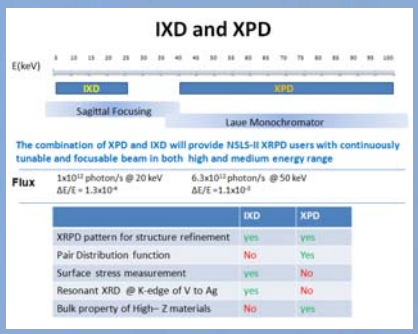
In Situ Surface Stress Analyses of High Temperature Alloys

High-temperature alloys have many important industrial applications in assuring a better performance of fossil-energy systems. Such alloy systems usually have multicomponent structures that undergo complicated physical and chemical processes in their surfaces in high temperature processes. New stress analyses method will be developed at IXD to simultaneously monitor the phase growth and the evolution of stress.

Real-Time Catalysis: *In-Situ* Structural Studies for Producing and Storing Hydrogen Fuel

Catalysis is the most critical field for sustainable energy production. Since real catalysts and catalytic processes are extremely complex, the availability of techniques for the real time characterization of catalytic systems as they evolve in time with a changing chemical environment is a high priority. The *in-situ* XRD capability of the IXD beamline affords the effective solution to such research needs, and will ensure the continuity of service to existing user groups.

SPECIFIC PROJECTS / ADDITIONAL INFORMATION



IXD and XPD

E(keV)

IXD — Sagittal Focusing

XPD — Laue Monochromator

The combination of XPD and IXD will provide NSLS-II XRPD users with continuously tunable and focusable beam in both high and medium energy range

Flux: 1×10^{12} photon/s @ 20 keV, $\Delta E/E = 1.3\times10^{-4}$; 6.3×10^{12} photon/s @ 50 keV, $\Delta E/E = 1.1\times10^{-3}$

	IXD	XPD
XRPD pattern for structure refinement	yes	yes
Pair Distribution function	No	Yes
Surface stress measurement	yes	No
Resonant XRD @ K-edge of V to Ag	yes	No
Bulk property of High– Z materials	No	yes

- IXD aims to satisfy the practical needs for *ex situ* and *in situ* XRPD measurements by a wide user community, such as the user groups conducting studies on batteries, fuel cells, catalysis, high-temperature materials, light-metal production, and other energy industry-related and EERE-sponsored researchers.
- The medium x-ray energy range of IXD is in complementary to the already funded high energy (> 40 keV) x-ray powder diffraction beamline XPD.
- The High Temperature Materials Laboratory (HTML) at Oak Ridge National Laboratory is a DOE User Facility dedicated to solving materials problems that limit the efficiency and reliability of systems for power generation and energy conversion, distribution, and use. HTML supports the proposed IXD beamline and will join with other EERE-sponsored programs to pursue support from within EERE for scientific personnel and instruments in operating the IXD.
- IXD will work with the *Industrial User Program* of the Photon Science Directorate to explore new opportunities in industrial applications using synchrotron radiation.

INFRARED BEAMLINE for MAGNETOSPECTROSCOPY, ELLIPSOMETRY and TIME-RESOLVED OPTICAL SPECTROSCOPIES (METRO)

Proposal Team: D.N. Basov[1], C. Bernhard[2], K. Burch[3], **G. L. Carr[4]**, T.F. Heinz[5], C.C. Homes[4], R.P.S.M. Lobo[6], J. Musfeldt[7], M. Sfeir[4], A.A. Sirenko[8], C.S. Stanton[9], D.B. Tanner[9], and J.J. Tu[10]

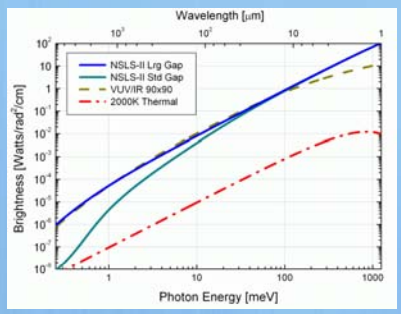
[1]Univ. Cal. San Diego, [2]Univ. Fribourg, [3]Univ. Toronto, [4]Brookhaven Nat'l Laboratory, [5]Columbia Univ., [6]CNRS, [7]Univ. Tennessee, [8]New Jersey Inst. Tech., [9]Univ. Florida, [10]City College New York

BROOKHAVEN NATIONAL LABORATORY

NATIONAL SYNCHROTRON LIGHT SOURCE II approved beamline

TECHNIQUES AND CAPABILITIES

- The METRO infrared beamline will provide advanced infrared spectroscopic capabilities for condensed matter physics and materials science.
- High-brightness, ultra low-noise and pulsed infrared synchrotron radiation source from NSLS-II Large Gap bending magnet.
- Continuous spectral coverage from <5 cm^{-1} to >30,000 cm^{-1} (0.5 meV to 4 eV)
- Diffraction-limited spatial resolution, time resolution to 10s of picoseconds.



High magnetic field & low-temp. spectroscopy

- Fields to 10T (16T feasible)
- Temperatures down to 1.6K
- Optical access for synchrotron radiation and laser.
- Polarization control.

Spectroscopic ellipsometry

- Unique capability into THz w/cryogenic sample space.
- Full Müller-matrix system: => anisotropic & magnetic materials.

THz microprobe of small crystals and structures

- Spotsize of 2λ (diff. limited)
- Temperatures down to 5K.

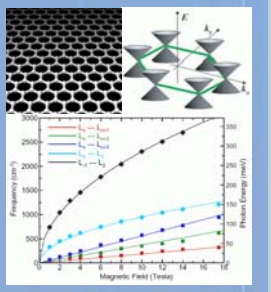
Time-resolved, photo-induced spectroscopies

- Synchronized optical excitation with Ti:S laser.
- Probe with complete spectral coverage.
- Time resolution to ~10ps.

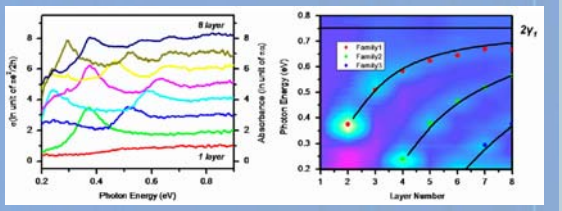
APPLICATIONS

Electronic properties of novel materials such as graphene.

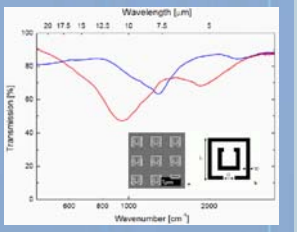
(H.L. Liu et al, NJP)



(K.F. Mak et al, PNAS)

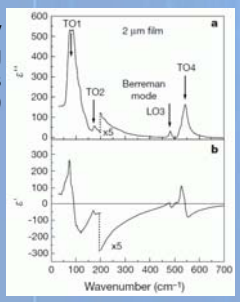


Plasmonic structures and metamaterials for negative refractive index.
(J. Hwang et al).



Spectroscopic studies of complex oxides and emergent materials.

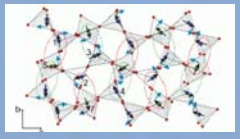
Ellipsometric study of mode softening in $SrTiO_3$ films
(A. Sirenko, Nature)

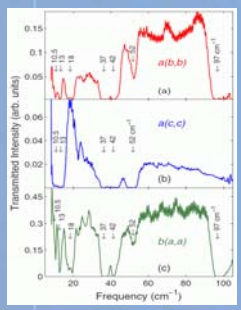


Conductivity through M-I transition in vanadium oxide single microcrystals
(M. Qazilbash & D. Basov)

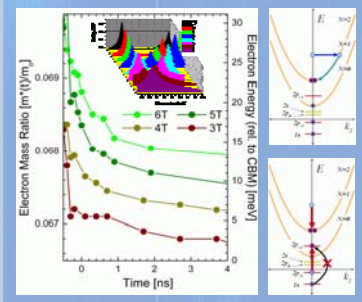
295 K | 343 K | 360 K

Multiferroic phonon and magnon modes
(A. Sirenko, PRB)

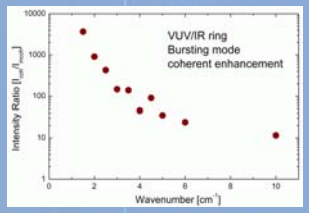




Materials for harvesting light energy: photoelectron relaxation dynamics in III-V semiconductors *(S.N. Gilbert).*



Light source & accelerator physics: coherent THz produced by electron bunch instability *(G.L. Carr)*



INSTRUMENTATION DEVELOPMENT (MID)

Project Team: L. Berman, G. Carini, K. Evans-Lutterodt, M. Honnicke, S. Hulbert, M. Idir, **J. Keister**, R. Reininger, D.P. Siddons, P. Takacs, Z. Zhong
Brookhaven National Laboratory

BROOKHAVEN NATIONAL LABORATORY

NATIONAL SYNCHROTRON LIGHT SOURCE II approved beamline

TECHNIQUES AND CAPABILITIES

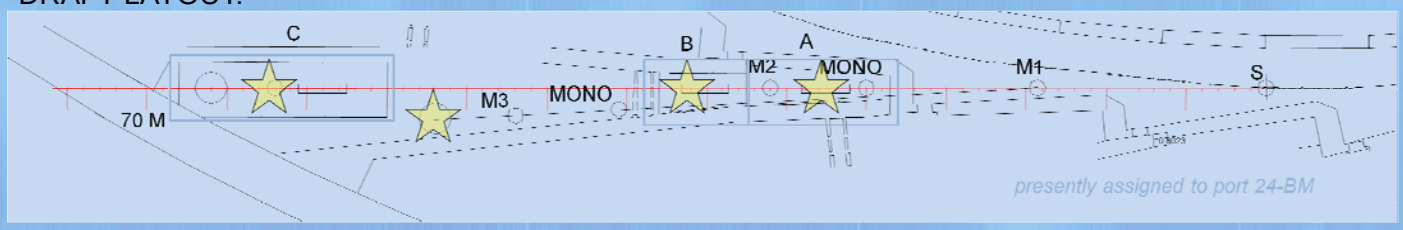
- 50 eV – 50 keV available from NSLS-II BM port
- Four highly configurable experimental stations to handle different energies and geometries:

1. Optics table in hutch A (first optics enclosure)
2. Optical table and chamber in hutch B
3. Optical table & diffractometer in hutch C
4. Vacuum reflectometer on soft branch

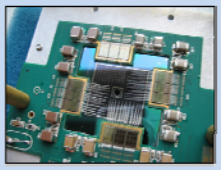
- Highly flexible beamline layout to allow white, pink and monochromatic beams:

1. Main branch:
   a) DCM/DMM Monochromator
   b) Focusing mirror
2. Soft x-ray side branch:
   a) Horizontal collection mirror
   b) Grating mono
   c) Refocusing mirror

DRAFT LAYOUT:

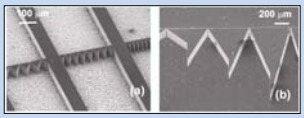


APPLICATIONS

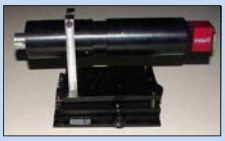


Maia detector developed at BNL for microbeam x-ray spectroscopic imaging

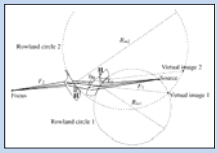
640-channel strip detector; development of germanium sensors for high energy applications in progress



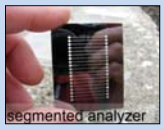
A bi-prism interferometer used in for coherence metrology of synchrotron beams



Hartmann Wavefront sensor developed atSOLEIL for wavefront metrology of adaptive optics



A fixed-exit monochromator using two sagitally bent Laue crystals developed at BNL for high energy experiments



A prototype monolithic analyzer crystal for multi-element powder diffraction in development at BNL

Detector systems
Characterization and calibration of arrayed spectroscopic detectors for high-throughput diffraction, spectroscopy and imaging

Optics
- X-ray profilometry and reflectometry of mirrors and gratings
- Wavefront analysis
- Diffractometry
- Topography (main branch)

Integrated systems
- Monochromators, spectrometers and polarimeters
- Environments
- Precision positioning and temperature control
- Automation

OUTLINE OF PROJECTED ACTIVITIES

Program Priorities

Development of new beamline devices and methods

- In-house detector and spectrometer development, high-performance crystal optics, positioning and temperature control, coherence and brightness-preserving reflective, refractive, and diffractive optics, integrated sample / optics / collection environments, polarimeters etc.
- Additional needs expected to arise but which cannot fully be projected

Characterization of beamline optical components and detectors

- New commercial devices to be installed on other beamlines, including mirrors, monochromators (including both crystals and gratings), detectors, phase plates, zone plates, etc.
- Other “standard” devices designed and built in-house

Collaboration with and support of users with similar development needs

- MID will support facility instrumentation needs, to keep science at NSLS-II on the cutting edge
- Beamline operation to commence as early as possible in the life of NSLS-II
- Internal development and commissioning needs presently being collected

MATERIALS PHYSICS AND PROCESSING (MPP)

Project Team: **J.L. Jordan-Sweet[1]**, V. Kiryukhin[2], C. Lavoie[1], C.E. Murray[1], C.S. Nelson[3], I.C. Noyan[4]

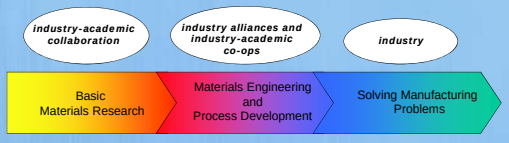
[1]IBM Research Division, [2]Rutgers University, [3]Brookhaven National Laboratory, [4]Columbia University

BROOKHAVEN NATIONAL LABORATORY

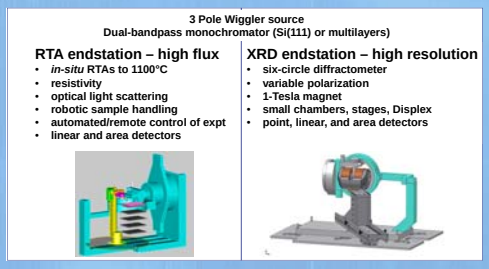
NATIONAL SYNCHROTRON LIGHT SOURCE II approved beamline

TECHNIQUES AND CAPABILITIES

- High-throughput, real-time, *in-situ* rapid thermal annealing (RTA) studies of structural changes in thin films, film stacks, and nanopatterned samples:
  - Phase transformations, texture changes, barrier failure, interfacial roughening, *etc*.
- X-ray diffraction (XRD) and scattering techniques on solids, including thin films, stacks, nanopatterned samples, magnetic and strongly correlated systems, and bulk materials. Capability for mounting small environmental chambers, bending jigs, magnets, and Displexes.
  - Resonant scattering, magnetic scattering, pole figures, phase ID, strain, reflectivity, *etc*.

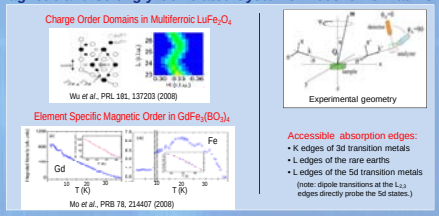


Industrial materials research continuum



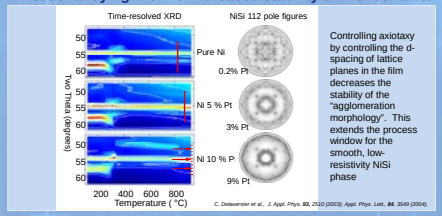
APPLICATIONS

Magnetic and Strongly-Correlated Systems: Recent work at NSLS



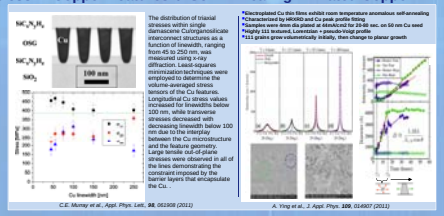
Resonant and non-resonant x-ray scattering (XRS) will be used to study electronic ordering phenomena such as magnetic, orbital, charge, and multipole order. Nonresonant XRS is well-suited to characterize the weak lattice distortions that often accompany electronic ordering, while resonant XRS is element specific and provides additional information through measurements of energy, polarization, and azimuthal dependencies.

Effect of alloying with Pt: increased stability of NiSi contacts



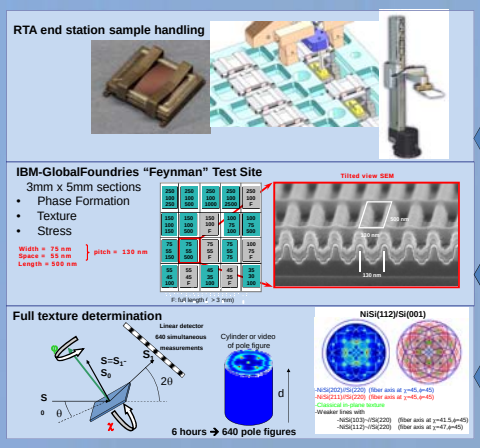
Real-time, *in-situ* annealing is used to study phase transformation sequences and kinetics of materials used in chip manufacturing and those being developed for future devices, such as Phase-Change Memory. Texture and strain also play an important role in device performance. Pole figures are particularly useful for understanding the behavior of metal silicides used as electrical contacts to the device layer in chips.

Stress in Copper Features / Self-Annealing in Plated Copper Films



High-resolution XRD is a valuable technique for determining stress/strain, grain size, lattice defects, and other structural subtleties that can strongly affect the behavior of chip components. BEOL (back-end-of-the-line) processes such as metal deposition, trench lining and capping, dielectric formation, chemical-mechanical polishing, and annealing can all lead to mechanical and chemical effects that require in-depth structural characterization.

SPECIFIC PROJECTS / ADDITIONAL INFORMATION



- Rapid switching between the RTA and XRD endstation hutches and between high-flux and high-resolution monochromator crystals will enable the efficient use of beam time.
- IBM currently is developing an automated RTA endstation for the CLS that will enable remote operation and fast turnaround.
- The NSLS has plans for a unique 1 Tesla magnet that can be mounted on a standard Huber diffractometer (see figure 2 above).
- IBM has a new test site that uses EUV lithography to make samples having arrays of nanometer-scale features for measuring scaling effects on phase formation, stress, and texture.
- IBM is collaborating with students at E. Poly Montreal and U. Ghent to develop software for sophisticated texture measurement and analysis.

MULTI-SCALE X-RAY DIFFRACTION (MXD)

Project Team: Y. Chu[1], P. Evans[2], **K. Evans-Lutterodt[1]**, G.E. Ice[3], D. A. Ivanov[4], B.C. Larson[3], W. Liu[4], W. Mao[5], C.E Murray[6], E. DiMasi[1], I. Takeuchi[7], W. Yang[8]
[1]Brookhaven National Lab, [2]Univ. Wisconsin, [3]Oak Ridge National Lab, [4]ISM de Mulhouse, [5]Stanford Univ., [6]IBM, [7]Univ. of Maryland, [8]Carnegie Inst.

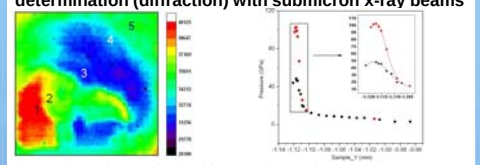
BROOKHAVEN NATIONAL LABORATORY

NATIONAL SYNCHROTRON LIGHT SOURCE II approved beamline

NANO/MICRO BEAM X-RAY DIFFRACTION

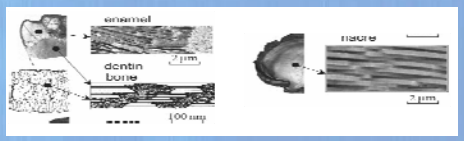
- Adjustable 30nm to 3 microns **stable** spot sizes
- Larger spots sizes can collect most of the light from undulator
- Diffraction friendly, i.e. maximize reciprocal space access
- Long working distance focusing optics which enable:
  - a) Heating and cooling of samples while thermally shielding optics
  - b) Other sample environments, UHV, humidity, DAC, …
  - c) Easy integration of optical microscopes other diagnostics
  - d) Coherent illumination of sample possible
- Diffractometer design to allow new modes with required stability
- Extensions such as the novel coherence based methods can be built on top of this platform

Chemical composition (absorption) and structural determination (diffraction) with submicron x-ray beams

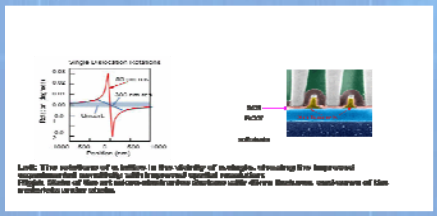


Ref: "Nanoprobe at Megabar", PNAS (2010); "Calcium at HP and LT", PNAS (2010)

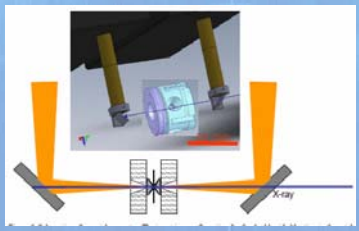
APPLICATIONS



Natural materials, such as shells, tooth, and bone, exhibit many levels of hierarchical micro-structure, starting with building blocks in the nanoscale, and often with improved properties. Monolithic $CaCO_3$ has a fracture strength ~3000 times weaker than nacre, a natural shell material composed of 200 nm sized crystals of the $CaCO_3$.

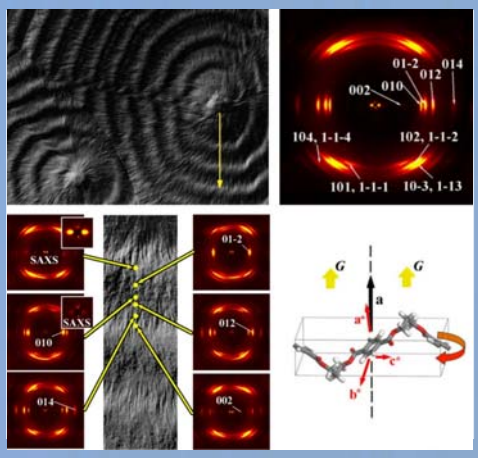


With the proposed beamline, we would be able to map out the diffraction from individual dislocations and study the evolution of grain-to-grain and intra-grain strain distribution inside of each grain with all kinds of external fields (E, M, P, T) at real time. This capability will have applications in the study of the long term battery reliability.



The extreme environment typical of the earths core and mantle is a problem rich phase space. The most recent example is the suggestion of a deep carbon component to the carbon cycle. The small beam sizes and long working distance at MXD allows diffraction from materials in ultra high pressures and temperatures in a laser heated diamond anvil cell.

SPATIALLY RESOLVED DIFFRACTION



The basic capability is spatially resolved diffraction. As another example of the importance of spatially resolved diffraction, we show on the left the work of Ivanov et. al., on the nature and origin of spherulitic banding in poly(trimethylene terephthalate), PTT. Scrolling and twisting of crystals at crystal growth fronts has been documented for both organic and in-organic sytems. The complex shape of these non-planar crystals can sometimes be deduced from microscopy, however the case of spherulitic banded polymer crystals was not amenable to these microscopy methods. Recent micro-focus X-ray scattering measurements provide strong support to the seminal model of *Keith* and *Padden*. In this model, lamellar twist was posited to result from a tilt of the chains relative to the basal lamella plane, which gives rise to a differential congestion of the chain folds at the lamella surfaces.

QUICK ABSORPTION SPECTROSCOPY (QAS)

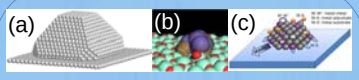
Project Team: S.R. Bare[1], J.G. Chen[2], S. Ehrlich[3], **A. Frenkel[4]**, J. Graetz[3], J.C. Hanson[3], S. Khalid[3], J.A. Rodriguez[3], X.-Q. Yang[3]
[1]UOP LLC, [2]University of Delaware, [3]Brookhaven National Laboratory, [4]Yeshiva University

BROOKHAVEN NATIONAL LABORATORY

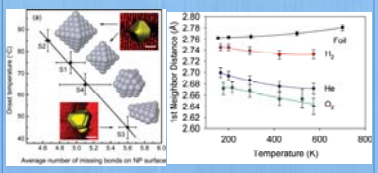
NATIONAL SYNCHROTRON LIGHT SOURCE II approved beamline

TECHNIQUES AND CAPABILITIES

- Will enable in-situ and operando studies of complex nanoscale systems undergoing real-time transformations
- Will enable synchronous measurements of nanocatalysts by complementary techniques including IR, XAS, XRD, DAFS and Mass Spectrometry with high energy resolution
- Will probe complex interactions in nanoscale systems at the time scale from tens of ms to hours and length scale from Å to µm



Bridging complexity gap between model (a,b) and real (c) catalysts



Effects of size, shape, support and adsorbates on physical/chemical properties and reactivity

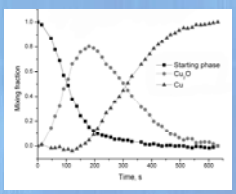
Kinetics of nucleation/growth

XAFS/XRD/ DAFS/MS

DRIFTS/ XAFS/MS

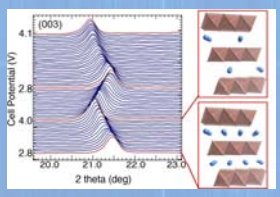
QEXAFS

APPLICATIONS



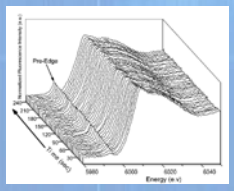
Investigation of $Cu^{2+}/CeO_2 \rightarrow Cu^0$ reaction kinetics by QEXAFS.

CATALYSIS: Investigations of structure, kinetics, dynamics and reactivity during in situ transformations with 10 ms time resolution



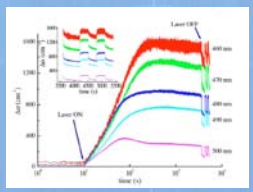
$LiCoO_2$ during a charge-discharge-charge cycle:

ENERGY GENERATION AND STORAGE: Understanding the physical and chemical processes in batteries and fuel cells



Cr(III) oxidation kinetics ([Cr(III)] = 100 mM)

ENVIRONMENTAL SCIENCE: Kinetics of rapid chemical processes on mineral surfaces and soils

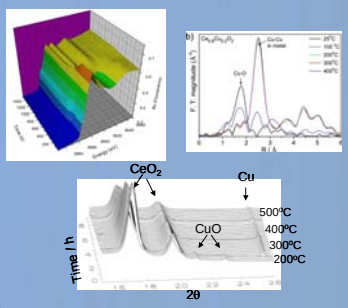


Photoinduced changes in amorphous chalcogenide films

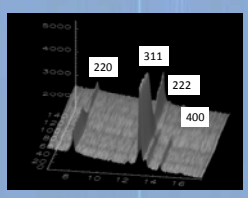
GLASSES AND MEMORY ALLOYS: Understanding correlations between glass-forming ability and structure of novel glasses and phase-change materials

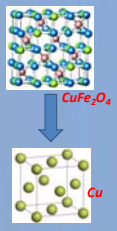
SPECIFIC PROJECTS / ADDITIONAL INFORMATION

Water-Gas Shift catalyst: $Cu_{0.2}Ce_{0.8}O_2$



Water-Gas Shift catalyst: $CuFe_2O_4$





TECHNIQUES: X-ray absorption spectroscopy and x-ray diffraction with 10ms time resolution, combined with multiple complementary techniques: IR, MS, DAFS, HERFD

MONOCHROMATORS: Double Crystal (slow scanning) and Channel Cut (Quick EXAFS)

SOURCE: Three pole wiggler

SPECTROSCOPY + CRYSTALLOGRAPHY (SM3)

Project Team: M. Allaire[1], L.E. Berman[1], W.A. Hendrickson[2], A. Héroux[1], L.M. Miller[1], **A. Orville[1]**, H.H. Robinson[1], D.K. Schneider[1], W. Shi[3], A.S. Soares[1], D. Stoner-Ma[1], M.R. Sullivan[3], R.M. Sweet[1]

[1]Brookhaven National Laboratory, [2]Columbia University, [3]Case Western Reserve University

BROOKHAVEN NATIONAL LABORATORY

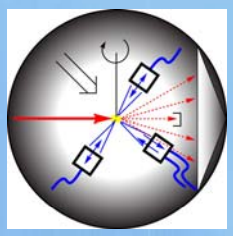
NATIONAL SYNCHROTRON LIGHT SOURCE II approved beamline

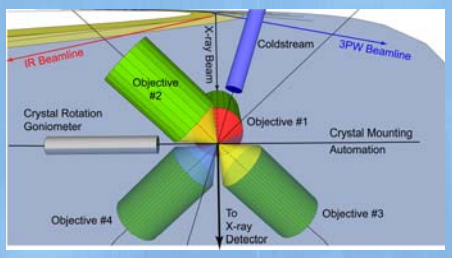
MULTIDISCIPLINARY STUDIES OF MACROMOLECULAR CRYSTALS

The SM3 missions are aligned with those of the NIH and the DOE.

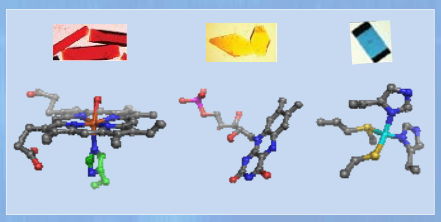
We will build an integrated infrastructure at the NSLS-II to support the nearly simultaneous, correlated measurements of data for:

- X-ray diffraction to high resolution (through objective #1)
- UV/Vis optical absorption (with objectives #2 and 3)
- Steady-state and time-resolved fluorescence spectroscopy (with objectives #2, 3, and 1 or 4)
- Non-resonance & resonance Raman spectroscopy (with lasers and backscatter mode through objectives #1, 3 and/or 4)
- FTIR spectroscopy from an adjacent IR beamline (e.g. with objectives #2 and 3)
- XAS/XANES/EXAFS viewing a 3PW and spectroscopy access through regions 1 and 3 or 4)

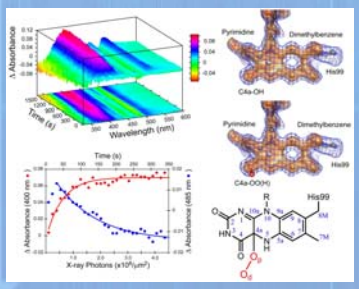




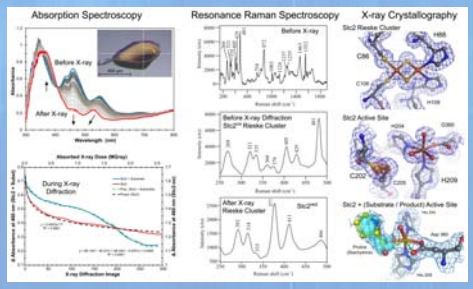
APPLICATIONS



- About ½ of all enzymes contain cofactors and/or metal ions.
- The electronic properties of these types of cofactors provide more strategies to achieve catalysis than H, C, N, O, P, or S atoms.
- Therefore, fundamental mechanistic insights into biology will come from understanding the relationship between atomic and electronic structure.

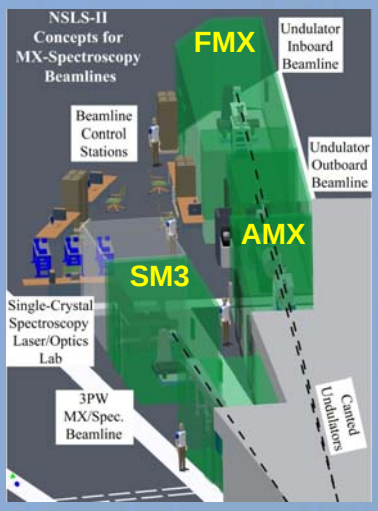


- Complementary data from choline oxidase (CHO) crystals
- Spectroscopic changes in a CHO crystal of upon X-ray exposure at 100 K
- Crystal structure of possible reactive oxygen species
- The electronic and atomic structures correlate well
- If only one type of data, then the interpretation remains uncertain



- Stachydrine demethylase (Stc2) impacts plant-microbe symbiosis
- Absorption spectra after each X-ray diffraction image show that the sample changes (left)
- Resonance Raman spectra (center) before and after X-ray diffraction indicates that the Rieske cluster is reduced
- Crystal structures (right) reveal X-ray promoted catalysis *in situ*

SM3 WILL INTERACT WITH NEIGHBORING BEAMLINES



- FMX: Frontier macromolecular crystallography (undulator, to produce a micro-beam in the 1 – 50 μm range)
- AMX: Highly automated macromolecular crystallography (undulator, to produce a mini-beam in the 5 – 300 μm range)
- Photons may also come from a nearby IR beamline
- **SM3 Techniques**: Macromolecular crystallography (MX) with spectroscopy integrated into the beamline and an off-line laser optics lab (UV/Vis Absorption, Fluorescence, FTIR, Raman, XAS/XANES/EXAFS)
- Source: Three-pole wiggler
- Flux: 10^{11} ph/s at 12 keV
- Energy Range: 5 – 20 keV
- Beam Size: 25 – 300 μm
- Dedicated to full-time correlated studies of spectroscopy with MX

TIME-RESOLVED X-RAY DIFFRACTION AND SPECTROSCOPY UNDER EXTREME CONDITIONS (TEC)

Project Team: E.H. Abramson[1], M.R. Armstrong[2], A.V.G. Chizmeshya[3], Y. Fei[4], **A.F. Goncharov[4]**, J. A. Hawreliak[2], R. Jeanloz[5], K.K.M. Lee[6], J.-F. Lin[7], H.-K. Mao[4], R. McDonald[8], V. Prakapenka[9], E. Reed[10], M. Rivers[9], M.S. Somayazulu[4], S.T. Stewart[11], V.V. Struzhkin[4], Stanley W. Tozer[12], Pavel Zinin[13]

[1]Univ of Washington, [2]Lawrence Livermore National Lab, [3]Arizona State Univ, [4]Carnegie Inst of Washington, [5]Univ of California Berkeley, [6]Yale Univ, [7]Univ of Texas Austin, [8]Los Alamos National Lab, [9]Univ of Chicago, [10]Stanford Univ, [11]Harvard Univ, [12]National High Magnetic Field Lab, [13]High Press Minerals Physics & Mat'ls Science Lab SOEST

BROOKHAVEN NATIONAL LABORATORY

NATIONAL SYNCHROTRON LIGHT SOURCE II approved beamline

TECHNIQUES AND CAPABILITIES

- 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

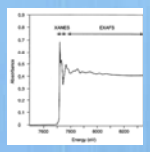
TECHNIQUES: micro-XRD and SAXS, XAS, XFS, XES, x-ray Raman

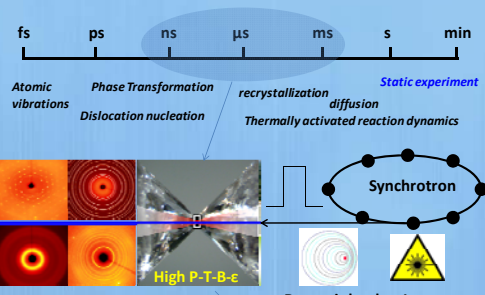
SOURCE: High beta undulator

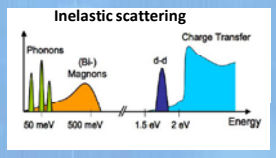
ENERGY RANGE / TIME RESOLUTION: 5-20 keV (up to 40 keV) / down to 10 ps, 1 ms typical

SPATIAL RESOLUTION: 0.2 – 5 μm

Probing fundamental ultrafast processes





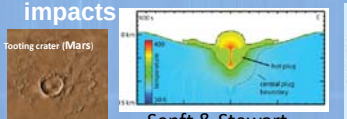


- o phase transition kinetics
- o structural dynamics & deformation
- o chemical reaction dynamics
- o transport properties (diffusion)
- o electronic properties

APPLICATIONS

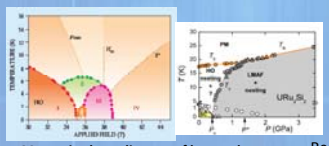
- Materials properties (e.g., strength) under extremes including dynamic response
- Synthesis of new materials by "Going Beyond Static Phase Diagrams"
- Control over fast chemical reactions (e.g., in energetic materials)
- Carbon sequestration technology
- Deep planetary interior dynamics & composition

Understanding planetary impacts



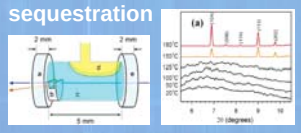
Senft & Stewart

Hidden Magnetic Orders



Magnetic phase diagram of heavy electron compound URu_2Si_2

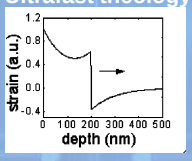
Understanding CO_2 sequestration



Reaction chamber: maintains constant chemical activity

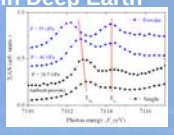
Formation of $MgCO_3$ detected by XRD

Ultrafast theology



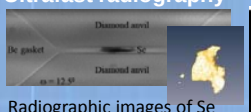
Laser induced strain profiled by pump-probe techniques
Lindenberg *et al.*, Phys. Rev. Lett. (2000)

Spin transitions in Deep Earth



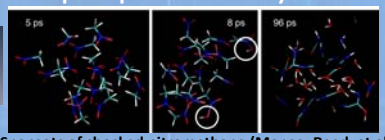
Pressure effect on pre-edge XAS
Struzhkin et al

Ultrafast radiography



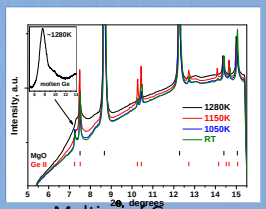
Radiographic images of Se at 10.7 GPa Liu *et al.*, PNAS (2008)

First principle molecular dynamics



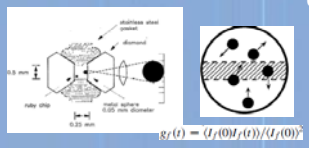
Snapsots of shocked nitromethane (Manaa, Reed et al).

Melting dynamics



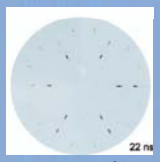
Melting of Ge,
Prakapenka et al., 2008

Transport phenomena



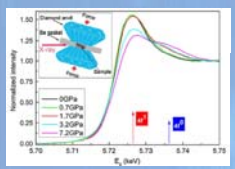
From rolling spheres to time-resolved correlation spectroscopy to determine the viscosity in the DAC
Thurn-Albrecht *et al.*, Phys. Rev. Lett. (1996)
Wang *et al.*, Phy. Rev. Lett., (1998)

Understanding plastic deformation mechanism



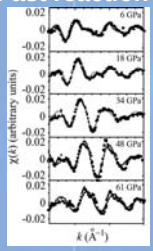
Laue pattern of laser shocked CdS single crystal
Ichiyanagi *et al.*, Appl. Phys. Lett (2007)

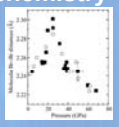
Controlling the synthesis of new materials



Ce L_3-edge XAS spectra: delocalization of 4f electron in $Ce_{75}Al_{25}$ Metallic Glass
Zeng *et al.*, Phys. Rev. Lett. (2010)

Fast reaction chemistry





Intramolecular Br-Br distance

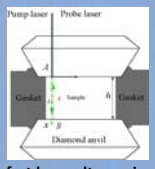
EXAFS oscillations at the bromine K-edge

San Miguel *et al.*, PRL (2007)

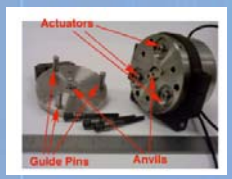
SPECIFIC PROJECTS / ADDITIONAL INFORMATION

Major challenge: Bridge the gap between static and dynamic experiments in P-T-strain rate conditions reached & probed

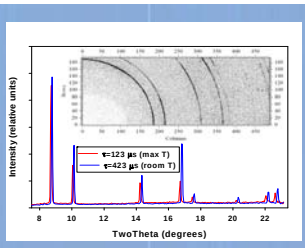
- Laser driven shock compression in the DAC
- Pulsed laser & res. heating in the DAC (>10,000 K)
- Dynamic DAC (10^{-2}-10^4 strain rate)
- Pulsed and static magnetic fields
- Laser ultrasonics in the DAC
- Optical spectroscopy (Raman, CARS, broadband)



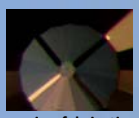
Ultrafast laser ultrasonics (Chigarev, Zinin et al)

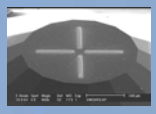


Dynamic DAC(Evans et al)

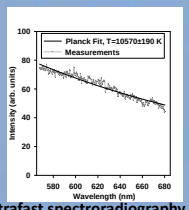


X-ray diffraction in pulsed laser heated DAC (Goncharov, Prakapenka et al.)



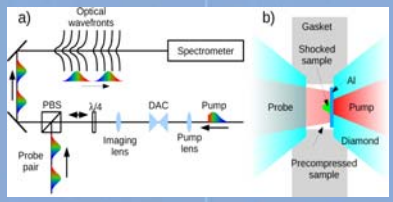


New microfabrication tools for DAC probes (photolithography and FIB contacts (Struzhkin et al)



Ultrafast spectroradiography In pulsed laser heating (Goncharov et al)

Plastic DAC for pulsed and dc magnetic field studies at high pressure and cryogenic temperatures (S. Tozer)



Laser driven shock compressions in the DAC (Armstrong, Crowhurst et al.)

TENDER ENERGY X-RAY ABSORPTION SPECTROSCOPY (TES)

Project Team: S. Bare[1,2], J. Brandes[3], T. Buonassisi[4], J. Chen[5,2], M. Croft[6], E. DiMasi[7], A. Frenkel[8,2], D. Hesterberg[9], S. Hulbert[7,2], S. Khalid[7], S. Myneni[10], **P. Northrup[7,11]**, E.T. Rasbury[11], B. Ravel[12], R. Reeder[11], J. Rodriguez[7,2], D. Sparks[5,13], V. Stojanoff[7], G. Waychunas[14]

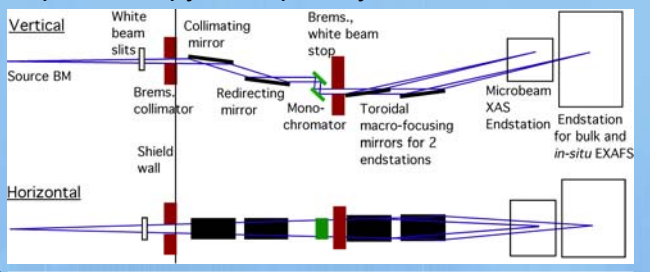
[1]UOP LLC, [2]Synchrotron Catalysis Consortium, [3]Skidaway Inst. of Oceanography, [4]MIT Laboratory for Photovoltaics Research, [5]Univ. of Delaware, [6]Rutgers Univ., [7]Brookhaven National Lab, [8]Yeshiva Univ., [9]North Carolina State Univ., [10]Princeton Univ., [11]Stony Brook Univ., [12]NIST, [13]Delaware Environmental Inst., [14]Lawrence Berkeley National Lab

BROOKHAVEN NATIONAL LABORATORY

NATIONAL SYNCHROTRON LIGHT SOURCE II approved beamline

TECHNIQUES: High performance and *in-situ* X-ray absorption spectroscopy and spatially-resolved XAS of structured materials; optimized for the "tender" energy range from 1.2 up to 8 keV.

- Will enable **spatially-resolved** and ***in-situ*** studies of speciation and local structure by XAS, X-ray fluorescence (XRF) and spectroscopic imaging, in a *non-vacuum environment.*
- Chemical sensitivity to **key lighter elements** Mg through Ti, and advantageous heavier-element L and M edges such as Cd, Pd, U.
- Optimized for the **NSLS-II dipole bend** source: high brightness over a tunable spatial resolution and energy scanning across 1.2-8 keV.



CAPABILITIES:

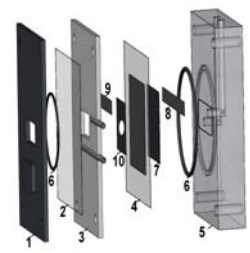
Source: dipole bend magnet, E_c 2.39 keV. Two optimized endstations.
Energy Range: 1.2 to 8 keV (optimized for 1.2-5 keV).
Spatial Resolution: 1x1 mm to <1x1 µm; **Flux**: up to $3x10^{12}$ ph/sec.
Detection: high- and low-countrate XRF from 0.9 to 8.3 keV.
Speed: on-the-fly scanning for ~1 minute EXAFS and/or rapid imaging.

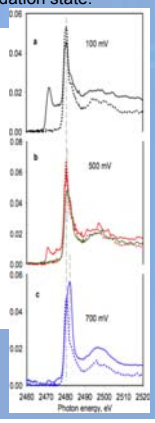
	High-performance and *in-situ* XAS endstation	Microbeam XAS and spectroscopic imaging endstation		
Energy (keV)	Flux, at 0.2x0.2 to 1.2x0.5 mm (ph/s at sample)	Flux, at 19x23 microns	Flux, at 6x7 microns	Flux, at 1x1 micron
1.2	1.15×10^{12}	5.4×10^{11}	2.85×10^{11}	6.8×10^{9}
2	3.5×10^{12}	2.3×10^{12}	1.0×10^{12}	2.4×10^{10}
3	2.8×10^{12}	1.9×10^{12}	7.7×10^{11}	1.8×10^{10}
4	1.9×10^{12}	1.2×10^{12}	4.5×10^{11}	1.1×10^{10}
5	1.3×10^{12}	7.2×10^{11}	2.7×10^{11}	6.4×10^{9}
7.5	5.1×10^{11}	2.15×10^{11}	8.1×10^{10}	1.9×10^{9}

APPLICATIONS: Focus on *Energy*, *Climate*, *Soil* and *Earth* Sciences

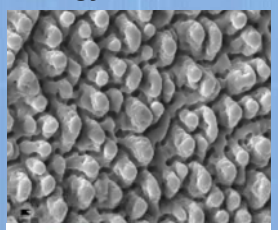
Sulfur poisoning of Fuel-Cell catalysts: *in-situ* XAS

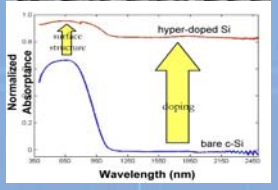
- Novel **Spectroelectrochemical cell** design for *in-situ* measurements of fuel-cell catalyst at controlled electrochemical potential and under gas flow, all compatible with low-energy fluorescence-mode XAS measurements.
- Real fuel-cell materials and geometry.
- Sample spectra under varying potentials, indicating changes in S speciation and oxidation state.





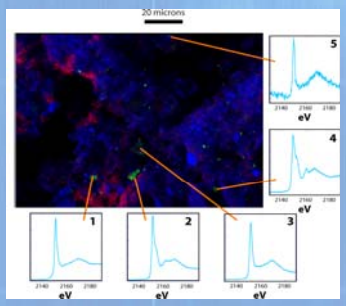
O. Baturina, B. Gould, Y. Garsany, R. Stroman and P. Northrup, "Identification of adsorbed SO2 species on fuel cell electrocatalysts by sulfur K-edge XANES combined with electrochemistry" in review.





Sulfur-doped silicon: Enhanced efficiency photovoltaics.

Surface structure (SEM) image of micro-textured and hyper-doped Si. Surface texture increases intrinsic absorption of the Si at the usual wavelengths; hyper-doping dramatically increases absorption at higher wavelengths.
B. Newman, T. Buonassisi, P.Northrup, in prep

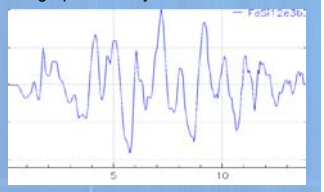


Phosphorus distribution and speciation: Key nutrient cycling and bioavailability influence global photosynthesis and biofuel/agricultural productivity.

Spatial and species heterogeneity of phosphorus in a natural sediment. Colors represent P (green), Si (blue) and Na (red). Point spectra indicate a variety of species.
Ingall, Brandes, Diaz, deJonge, Paterson, McNulty, Elliott, Northrup, "Phosphorus K-edge XANES spectroscopy of mineral standards," J. Synch. Rad., 18 (2011).
JDiaz, Ingall, Benitez-Nelson, Paterson, de Jonge, McNulty, Brandes, "Marine Polyphosphate: A Key Player in Geologic Phosphorus Sequestration" Science, 320, (2008).

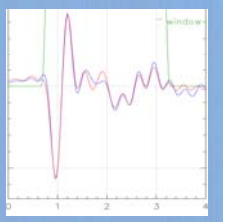
Investigation of site disorder in high-pressure silicate phases: Si K-edge EXAFS.

EXAFS data (k^2-weighted) suitable for determination of site disorder in microgram-size sample from high-pressure synthesis run.
P.Northrup, L. Li, D. Weidner, in prep



Nutrient sorption processes in poor and highly-leached soils: P, S, Ca, Mg, and K EXAFS.

S K-edge EXAFS (Fourier transform, real) data and fit for sulfate adsorbed to gibbsite mineral surface. Fit includes S-O, multiple scattering, and distances to two Al and one O in the substrate.



P. Northrup, M. Alves (USP, Brazil), D. Sparks, in prep.

SCIENTIFIC IMPACT:

- Energy Materials: Photovoltaic, fuel-cell, battery and superconducting (nano)materials.
- Catalysis/Chemistry: Materials (zeolites, thin films, nanomaterials), reaction mechanisms and intermediate species, poisoning of catalysts.
- Environmental/Earth Science: Biogeochemical and redox processes, contaminant behavior and remediation; Ca-Mg-Si high-pressure phases.
- Climate: Terrestrial/marine C cycling, carbonate (bio)mineralization, geologic record of climate change, ocean chemistry, CO_2 sequestration.
- Sustainability: Nutrient cycling, transport and bioavailability, biofuel/biomass productivity, especially in poor and leached tropical soils.

DEVELOPMENT STRATEGY:

- Our goal is *high productivity at the earliest possible date*.
- Strategy combines in-house and external aspects to create world-class capabilities and develop cutting-edge research programs -- and be ready to go on Day One.
- Utilize upgraded facilities at NSLS **X19A** Facility Beamline and **X15B** User Consortium-operated beamline.
- Design, commission TES microprobe endstation at X15B.
- Synchrotron Catalysis Consortium and BNL Chemistry: ongoing development of *in-situ* programs at X19A.
- Pre-test and commission optical components at NSLS.
- Collaborate closely with **ISS** (NEXT beamline for high-flux hard X-ray spectroscopies), **XFM** (hard X-ray micro-spectroscopy beamline), and **SRX** (Project Beamline: submicron hard X-ray probe) development teams.

X-RAY ABSORPTION SPECTROSCOPY FOR BIOLOGICAL, ENVIRONMENTAL, AND ENERGY SCIENCES (XAS)

Project Team: S. Bare[1], J. Bohon[2], **M. Chance[2]**, E. Farquhar[2], J. Fitts[3], M. Maroney[4], L. Que[5], I. Sagi[6], W. Shi[2], M. Sullivan[2], D. Tierney[7]

[1]UOP LLC, [2] Case Western Reserve University, [3]Brookhaven National Laboratory, [4]University of Massachusetts, [5]University of Minnesota, [6]Weizmann Institute of Science, [7]Miami University of Ohio

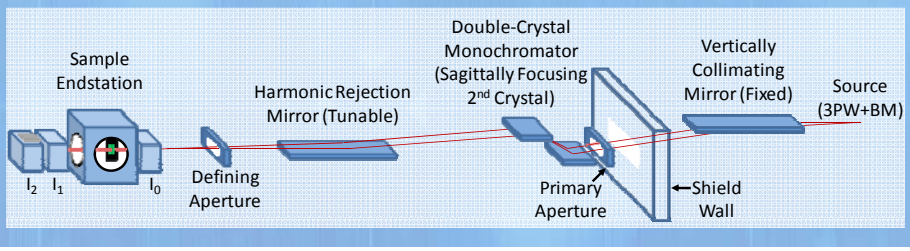
BROOKHAVEN NATIONAL LABORATORY

NATIONAL SYNCHROTRON LIGHT SOURCE II approved beamline

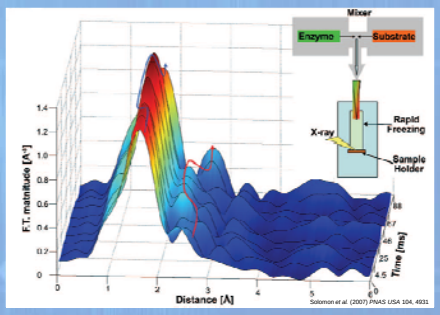
BEAMLINE CAPABILITIES

X-ray absorption spectroscopy beamline emphasizing studies of dilute samples from biological, environmental, and energy sciences-related fields.

- 3-pole wiggler source in the 5-25 keV energy range; provides continuity of service and expanded capabilities for an extensive, highly-productive user community.
- Enables EXAFS/XANES studies of dilute (<100 μM) samples.
- Sagittally focusing monochromator providing flexible beam size & tunable flux density (~0.2mm x 0.5mm to 2mm x 10mm, maintaining flux), with modern 31-element (or better) solid state Ge fluorescence detector.
- Multiple endstations (cryogenic, *in situ*, high throughput) with rapid changeover capabilities.
- Will be the #1 facility of its kind in the US and only such facility on the East Coast.



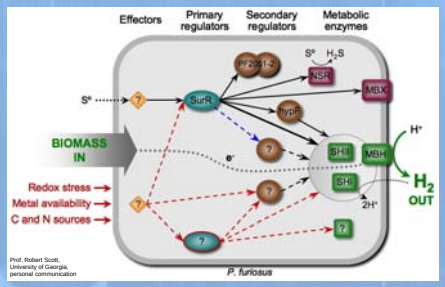
APPLICATIONS



Time-Resolved RFQ XAS

Flux and detector capabilities will further enable XAS studies of dilute samples from time-resolved freeze-quench trapping of very short-lived intermediates formed during enzyme reactions, providing new chemical insights.

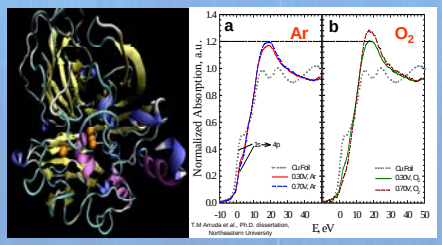
(above) Time-resolved XAS spectra of TNFα converting enzyme during catalysis.



HT XAS for Systems Biology

High throughput (HT) determination of the metal content of fractionated biological materials and proteins from large scale proteomic and metabolic studies will be feasible, including detailed XAS studies of metalloproteins identified from HT screening efforts.

(above) Metabolic pathways in *P. furiosus*

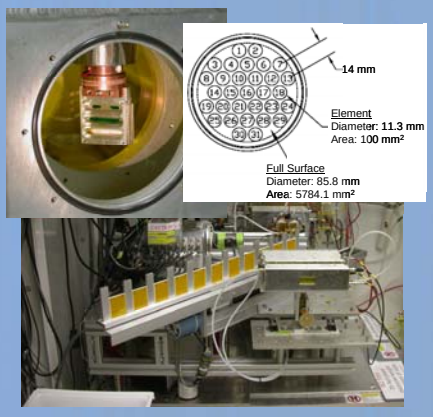


In Situ Studies of Biological Catalysis

Increase scope of *in situ* studies of enzymes, including probing structure-property relationships for metalloproteins with electro-active sites through tandem XAS and electrochemical experiments.

(above) *In situ* electrochemical/XAS studies of laccase in the presence of Ar or O_2 carried out at NSLS X3B.

ADDITIONAL INFORMATION



Many elements of the highly utilized and successful NSLS X3B beamline endstation will be implemented and further improved upon at the completed NSLS-II XAS beamline (see photos at left). These include cryogenic multicell sample holders, an automated high-throughput metal screening apparatus with space for 220 samples, and an advanced 31-element solid-state germanium fluorescence detector that is currently being commissioned at NSLS X3B.

With its focus on studies of dilute samples in biological, environmental, and energy sciences fields, XAS will fill an important niche within the community of X-ray absorption spectroscopy beamlines that have been approved for NSLS-II, while exhibiting synergy with numerous life-sciences oriented beamlines that comprise a "Biology Village."

X-RAY FLUORESCENCE MICROPROBE (XFM)

Project Team: P. Bertsch[1], E. Dooryhee[2], G. Flynn[3], J. Flynn[4], M. Ginder-Vogel[5], **A. Lanzirotti[6],** M. Marcus[7], L. Miller[2], P. Northrup[8], T. Punshon[9], M. Rivers[6], B. D.P. Siddons[2], G. Smith[10], S. Sutton[6], R. Tappero[2], T. Tokunaga[7]

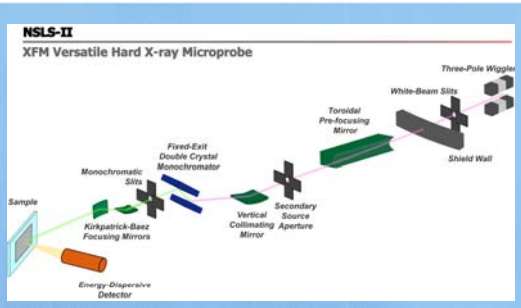
[1]Univ. Kentucky, [2]Brookhaven National Lab, [3]SUNY Plattsburgh, [4]American Museum of Natural History, [5]Calera Corp., [6]Univ. Chicago., [7]Lawrence Berkeley National Lab, [8]Stony Brook Univ., [9]Dartmouth College, [10]Indianapolis Museum of Art

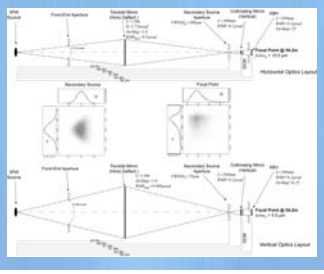
BROOKHAVEN NATIONAL LABORATORY

NATIONAL SYNCHROTRON LIGHT SOURCE II approved beamline

TECHNIQUES AND CAPABILITIES

- XFM is an optimized three-pole wiggler beamline for the characterization of materials in an "as-is" state that are chemically heterogeneous at the micrometer scale via synchrotron induced X-ray fluorescence.
- XFM includes instrumentation for microbeam X-ray fluorescence (μXRF), diffraction (μXRD) and fluorescence computed microtomography (FCMT) . However, it is optimized to provide users state-of-the–art microfocused Extended X-ray Absorption Fine Structure (μEXAFS) spectroscopy between 4 to 20 keV.
- XFM will trade-off beam size and flux for sample configuration flexibility. This includes more readily achievable stability constraints for spectroscopies (μEXAFS), accommodating large sample sizes (up to meters), and provisions for customized environmental chambers.

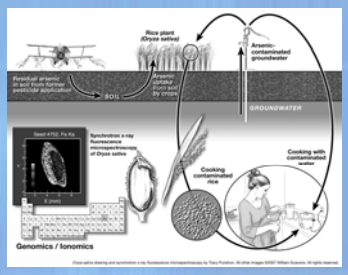


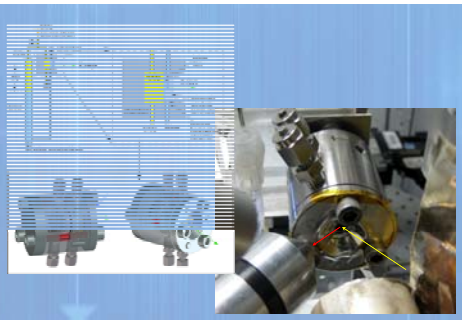


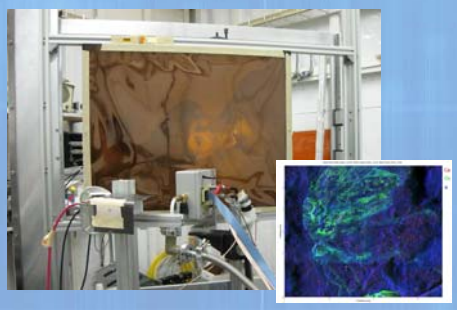
Energy	Source flux (0.33%E/E)*	Flux at sample position*		
keV	Ph/s/0.1%bw	10x5 μm	5x5 μm	1x1 μm
4	1.1E+13	2.E+11	8.E+10	3.E+09
5	9.5E+12	2.E+11	1.E+11	4.E+09
6	8.8E+12	2.E+11	1.E+11	4.E+09
7	8.0E+12	2.E+11	9.E+10	4.E+09
8	7.2E+12	2.E+11	8.E+10	4.E+09
9	6.5E+12	2.E+11	8.E+10	3.E+09
10	5.8E+12	1.E+11	7.E+10	3.E+09
11	5.1E+12	1.E+11	6.E+10	3.E+09
12	4.6E+12	1.E+11	6.E+10	2.E+09
13	4.1E+12	1.E+11	5.E+10	2.E+09
14	3.6E+12	9.E+10	5.E+10	2.E+09
15	3.3E+12	8.E+10	4.E+10	2.E+09
16	3.0E+12	8.E+10	4.E+10	2.E+09
17	2.6E+12	7.E+10	3.E+10	1.E+09
18	2.4E+12	6.E+10	3.E+10	1.E+09
19	2.2E+12	6.E+10	3.E+10	1.E+09
20	2.0E+12	5.E+10	3.E+10	1.E+09

*Fixed aperture (0.112 x 0.25 mrad); Si(111) bandpass; 12.5 μm Be window; 5 cm air absorption

APPLICATIONS







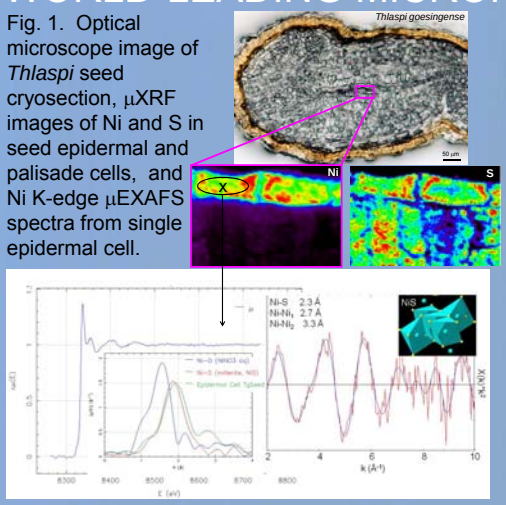
- XFM will provide the NSLS-II user community an optimized beamline for studying the genetic control of metal ion uptake, transport and storage in plants relevant to agriculture and bioenergy.
- The only beamline in the world designed to directly support plant biochemistry, XFM will provide high-throughput and high-resolution whole-plant fCMT.
- fCMT provides a 3D non-invasive, spatially resolved and multi-elemental analysis technique that images the metal concentration of specific cell layers and organelles in plants as close to their natural state as possible.

- XFM's unique optical design, which allows it to maintain a small spot size at long working distances with high flux and at high energies, will provide an ideal platform for microfocused analysis of samples within environmental cells.
- For example, we are designing cells for XFM that allow users to analyze materials under $scCO_2$ confinement (T > 31°C, P > 7.4 MPa), while controlling ΔP.
- XFM will utilize in-situ μXRF, μEXAFS and μXRD to quantify hydraulic/transport properties of brine films confined by CO_2 under geologically relevant conditions.

- Imaging large samples at X-ray microprobes is impractical, but XFM with its variable focus and collimation and advanced ultrafast, large solid-angle EDS detectors being developed at BNL, is ideal for these studies.
- XFM will allow for signals from 100's of elements to be read at milliseconds per pixel for bidirectional scans covering meters!
- Large format translation stages and environmentally-controlled hutches will allow for analysis of whole objects including panel paintings, sculpture, paleontological and archaeological materials.
- XFM will be the focus of such work for museum scientists in the northeastern U.S.

WORLD-LEADING MICROFOCUSED EXAFS SPECTROSCOPY

Fig. 1. Optical microscope image of *Thlaspi* seed cryosection, μXRF images of Ni and S in seed epidermal and palisade cells, and Ni K-edge μEXAFS spectra from single epidermal cell.



- XFM's unique optical design is optimized to enhance NSLS-II's source stability to provide unmatched μEXAFS data quality. Specifications include:
  - Use of a toroidal focusing mirror to illuminate a virtual source on the experimental floor that will be collimated, monochromated and imaged with KB microfocusing mirrors.
  - μ(E) oscillations measured to <0.1% of the edge-step signal for transition metals at >10 ppm concentration in a 1-10 μm spot.
  - Focused beam stability of <5% of the beam area while scanning a 1 keV energy range.
  - Fixed-exit DCM with user-selectable Si(111) and Si(311) crystal-pairs on a broadband source, will permit general user's to seamlessly and efficiently configure the beamline for spectroscopy in the 4-20 keV range.

X-RAY FOOTPRINTING FOR *IN VITRO* AND *IN VIVO* STRUCTURAL STUDIES OF BIOLOGICAL MACROMOLECULES (XFP)

Project Team: S.C. Almo[1], J. Bohon[2], **M.R. Chance[2]**, S. Gupta[2], M.R. Sullivan[2], T.D. Tullius[3], G.P. Williams[4], S. Woodson[5]

[1]Albert Einstein College of Medicine, [2]Case Western Reserve University, [3]Boston University, [4]Jefferson Laboratory, [5]Johns Hopkins University

BROOKHAVEN NATIONAL LABORATORY

NATIONAL SYNCHROTRON LIGHT SOURCE II approved beamline

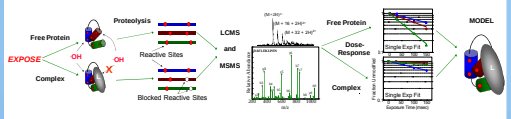
TECHNIQUES AND CAPABILITIES

X-ray Footprinting: hydroxyl-radical mediated covalent labeling via synchrotron x-ray radiolysis of water

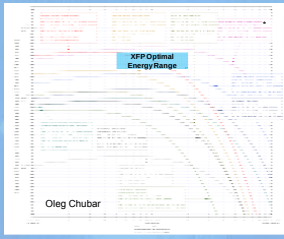
- Tool for structural biology to allow observation of complex macromolecules in the solution state
- Near-physiological conditions *in vitro* and *in vivo studies*
- Flow cell experiments for rapid exposure and time-resolved measurements on biologically relevant timescales
- High flux density focused "white beam" to achieve exposure times of less than a ms and high quality data for complex samples

The NSLS-II Damping Wiggler source will provide world-leading flux in the energy range of interest (5-20keV); promises to make XFP the premier facility in the world for synchrotron footprinting

Synchrotron X-ray Footprinting



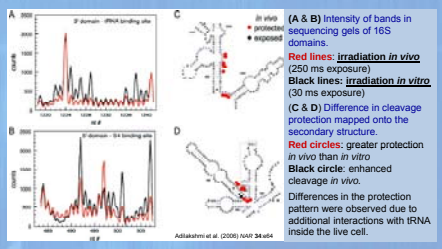
Samples are exposed to x-rays for a range of times (ms time scale); the hydroxyl radicals created in the solution covalently modify solvent-exposed reactive amino acid side-chains, but buried residues are protected. Proteins are digested with proteases and analyzed via mass spectrometry (LCMS and MS/MS) for quantitation and verification of the modified residues. Dose-response plots are created to determine the rate constants; variation in the modification rates of a peptide reflects the change of solvent accessibility of its probe residues, and can allow structural modeling of conformational changes and binding interfaces. Footprinting of nucleic acids involves hydroxyl-radical mediated cleavage of solvent-exposed backbone regions, observed via gel electrophoresis.



NSLS-II Damping Wiggler: Premier Source for X-ray Footprinting

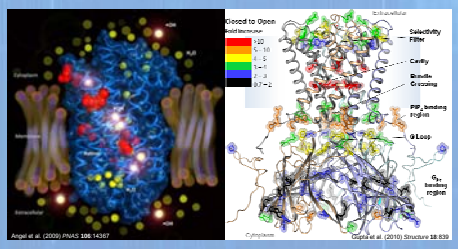
Comparison of source properties (far-field estimation) of a variety of "wiggler" sources around the world (W, Wiggler; SCW, Superconducting Wiggler; IVW, In-Vacuum Wiggler; DW, Damping Wiggler). Additional notes: the APS U33 source is an undulator used in 'tapered' mode for a broader energy band; although the PETRA III DW200 source appears to have the highest available flux, the information provided here is per horizontal mrad; this source will maximally provide 0.1 horizontal mrad of usable radiation, thus the NSLS II DW100 will provide greater flux in the energy range of interest.

APPLICATIONS



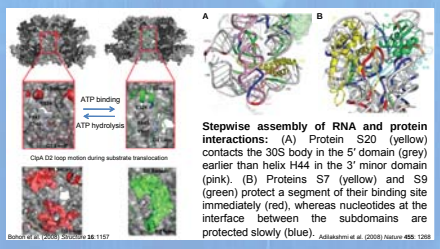
(A & B) Intensity of bands in sequencing gels of 16S domains.
Red lines: irradiation *in vivo* (250 ms exposure)
Black lines: irradiation *in vitro* (30 ms exposure)
(C & D) Difference in cleavage protection mapped onto the secondary structure.
Red circles: greater protection *in vivo* than *in vitro*
Black circle: enhanced cleavage *in vivo*.
Differences in the protection pattern were observed due to additional interactions with tRNA inside the live cell.

Adilakshmi et al. (2006) NAR **34**:e64



Angel et al. (2009) PNAS **106**:14367

Gupta et al. (2010) Structure **18**:839



Bohon et al. (2008) Structure **16**:1157

Stepwise assembly of RNA and protein interactions: (A) Protein S20 (yellow) contacts the 30S body in the 5′ domain (grey) earlier than helix H44 in the 3′ minor domain (pink). (B) Proteins S7 (yellow) and S9 (green) protect a segment of their binding site immediately (red), whereas nucleotides at the interface between the subdomains are protected slowly (blue). Adilakshmi et al. (2008) Nature **455**: 1268

In Vivo Studies

Reduction of exposure times *in vivo* to the range of single digit milliseconds will reduce sample damage due to overexposure.

Proof of principle with 16S rRNA shown above. Current studies: ribosome biogenesis, receptor-protein cell surface interactions.

Membrane Protein Dynamics

The high flux density beam at XFP will overcome the effect of strong hydroxyl radical scavengers in membrane fractions & liposomes.

Shown above: (left) observation of bound water in Rhodopsin; (right) novel gating residues identified in the K^+ channel.

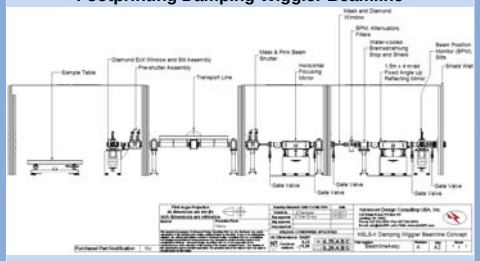
Mega-Dalton Complexes

XFP beam will overcome radical scavengers in complex buffer systems and enable probing of residues deep inside of cavities.

Above: (left) structural studies of proteosome assembly (ClpAP); (right) resolving early stages of ribosome assembly in real time.

ADDITIONAL INFORMATION

Preliminary Conceptual Design of the XFP X-ray Footprinting Damping Wiggler Beamline



The XFP beamline will produce a focused "white beam" of tunable size (minimum of 1x1 mm^2 due to flow cell diameter requirements), with more than 50X higher usable flux density than the current world-leading source (NSLS X28C). This will allow exposure times in the 100s of μs, enabling time-resolved experiments limited only by mixing times.

The "Biology Village": synergy with other synchrotron-based structural biology methods

- X-ray crystallography: atomic resolution static structures of macromolecular components form the basis for structural models
- Small-angle x-ray solution scattering: global molecular envelopes in solution provide overall observation of changes in macromolecular conformation; time-resolved experiments
- X-ray footprinting: local probe of solvent accessibility in solution provides observation of local conformational changes and identification of binding interfaces; time-resolved experiments

Together, these techniques promise to provide highly detailed molecular movies of the functions of complex biomolecules