Science Opportunities at NSLS-II

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Supercritical-CO_2 Materials Workshop

Brookhaven National Laboratory

March 22, 2011

U.S. DEPARTMENT OF ENERGY

BROOKHAVEN NATIONAL LABORATORY

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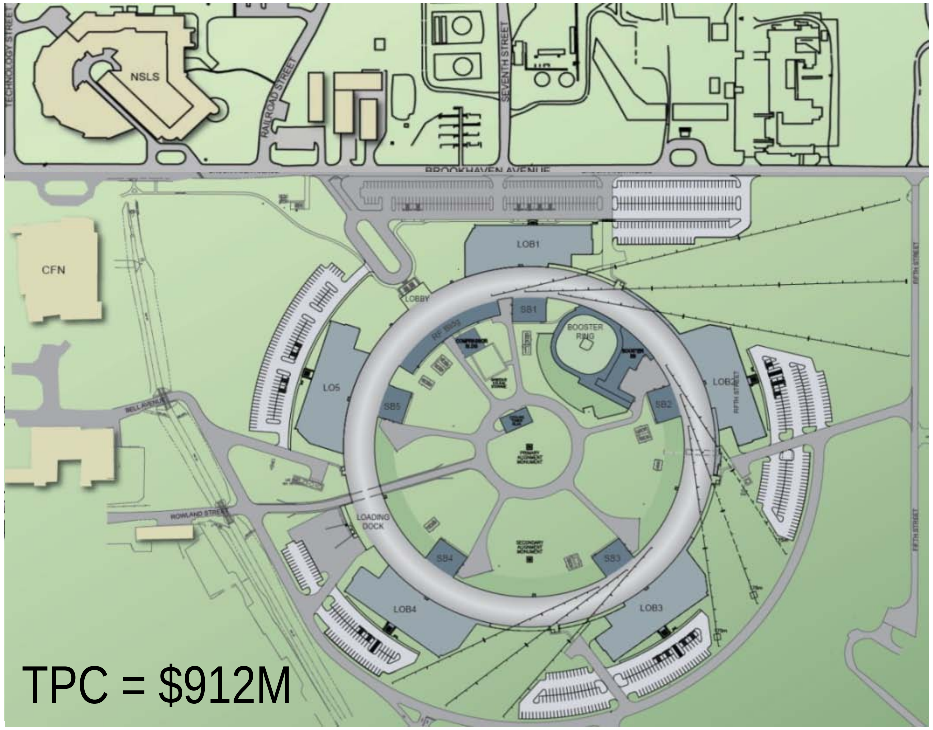
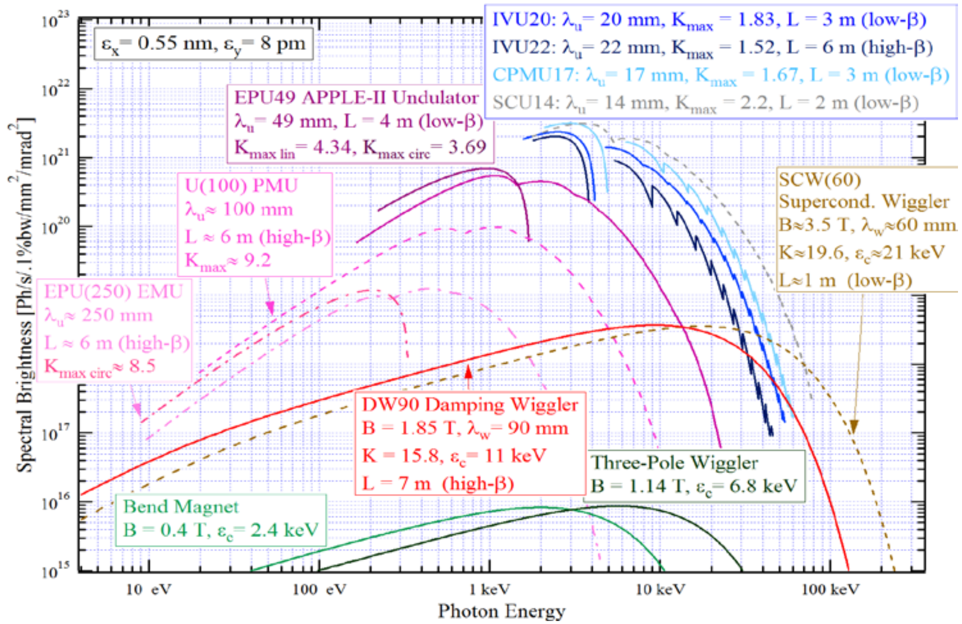
Outline

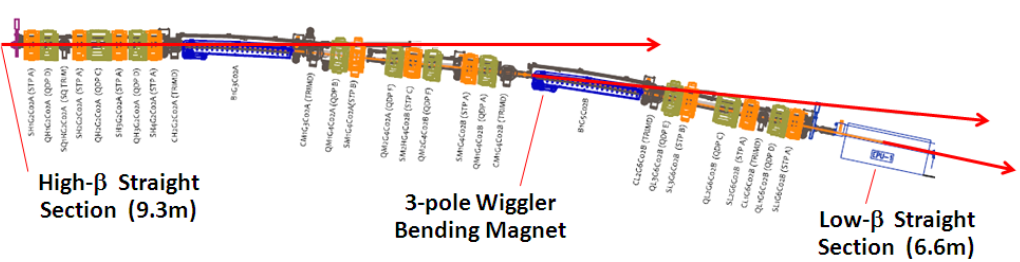
- Introduction to NSLS-II
  - Construction update
  - NSLS-II project beamlines
  - Development of additional beamlines
- In-situ techniques for sc-CO_2 studies
  - In-situ X-ray diffraction/scattering
  - In-situ X-ray spectroscopy
  - Real-time X-ray imaging
- Summary

NSLS-II: Optimized 3rd Generation SR





- 3 GeV, 500 mA, Circumference 791 m
- Emittance: ε_x= 0.55, ε_y= 0.008 nm-rad
- High brightness from soft to hard x-rays
- Small beam size: σ_y=2.6 μm, σ_x=28 μm
- Pulse length (rms) ~15 psec
- 27 insertion device beamlines
- 31 BM / 3PW / IR beamlines
- Full built-out includes at least 58 beamlines, plus canted IDs



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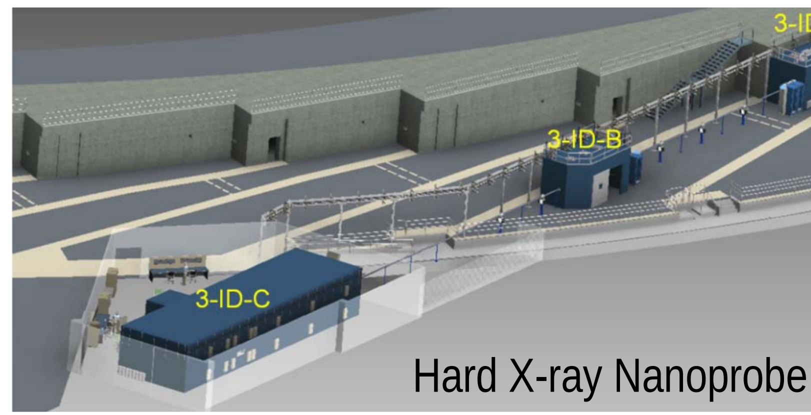
NSLS-II Construction Progress



Hard X-ray Nanoprobe

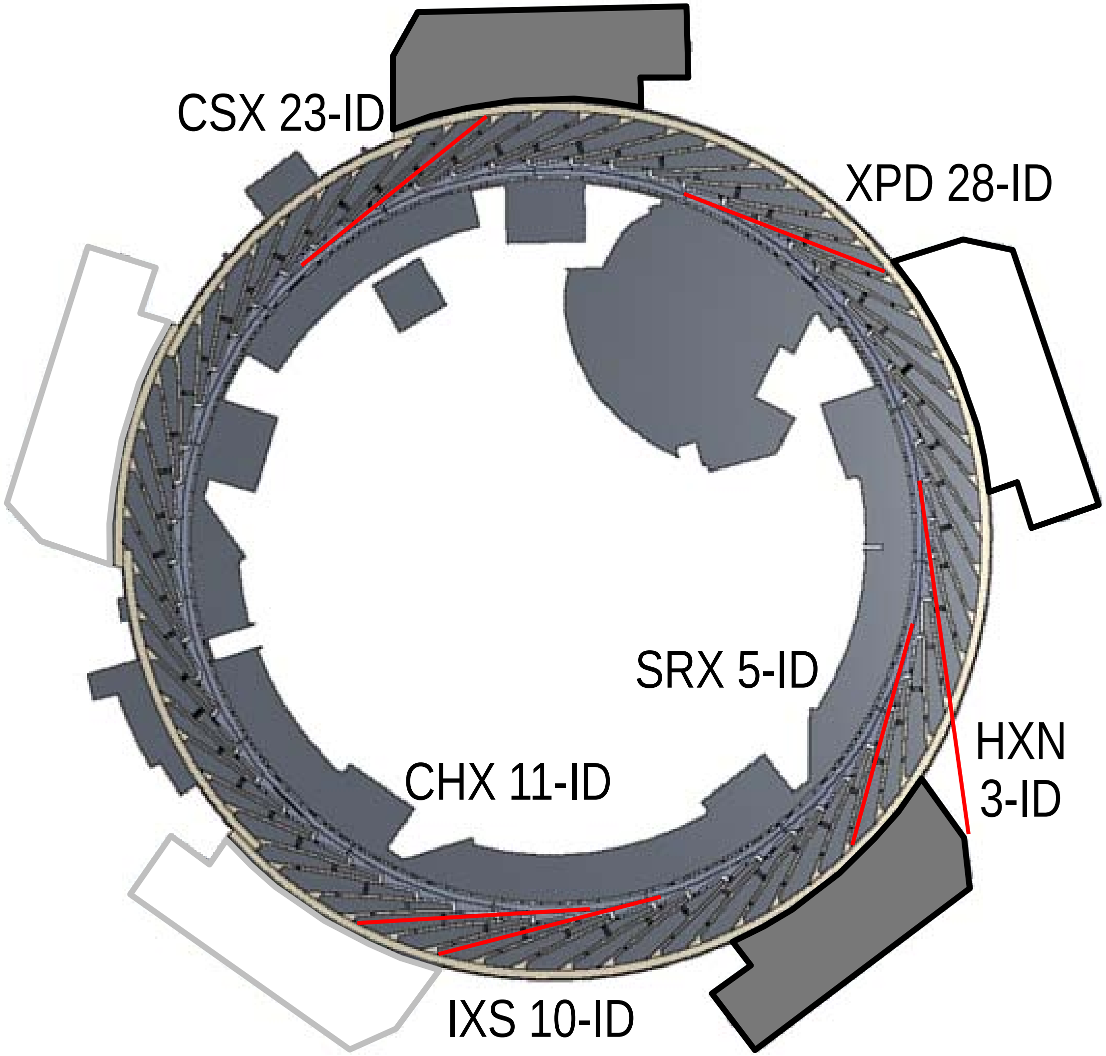
- NSLS-II project (DOE $912M) progressing on budget & on schedule
- Pentant 1 Beneficial occupancy received Mar 14. 2011; Accelerator installation and beamline procurements have begun
- Expected start of operations in Mar. 2014

Laboratory Office Bldg.

LOB design: 33,600 ft^2

- 120 seats
- 10 laboratories
- machine shop
- conference rooms
- loading/storage area

Six Beamlines in Construction Project

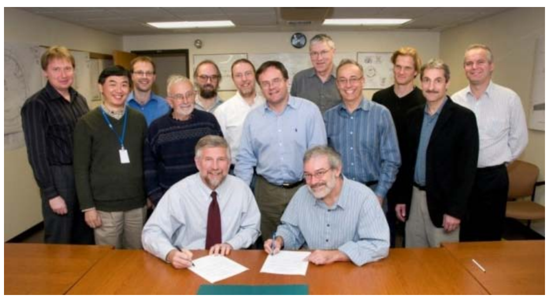


- Inelastic X-ray Scattering (IXS)
- Hard X-ray Nanoprobe (HXN)
- Coherent Hard X-ray Scattering (CHX)
- Coherent Soft X-ray Scattering & Polarization (CSX)
- Sub-micron Resolution X-ray Spectroscopy (SRX)
- X-ray Powder Diffraction (XPD)

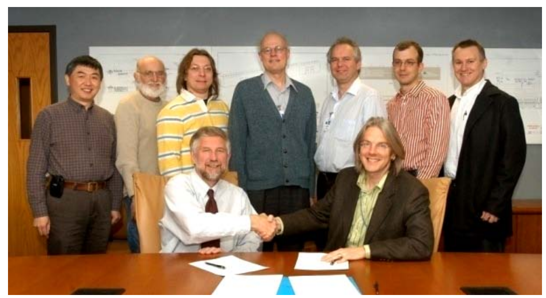
- *Beamline locations finalized for the six project beamlines*
- *Preliminary designs completed*
- *Procurement of long-lead-time components in progress*
- *Commissioning and ramp-up of operations to start March 2014*

Beamline Advisory Teams

- All NSLS-II beamlines are being developed using the concept of Beamline Advisory Teams (BATs).
  - BAT represents a segment of user community;
  - works close with facility to define scientific mission and technical scope;
  - meets regularly with facility staff in design, construction, and commissioning.



SRX BAT



XPD BAT

IXS BAT

CHX BAT

CSX BAT

HXN BAT

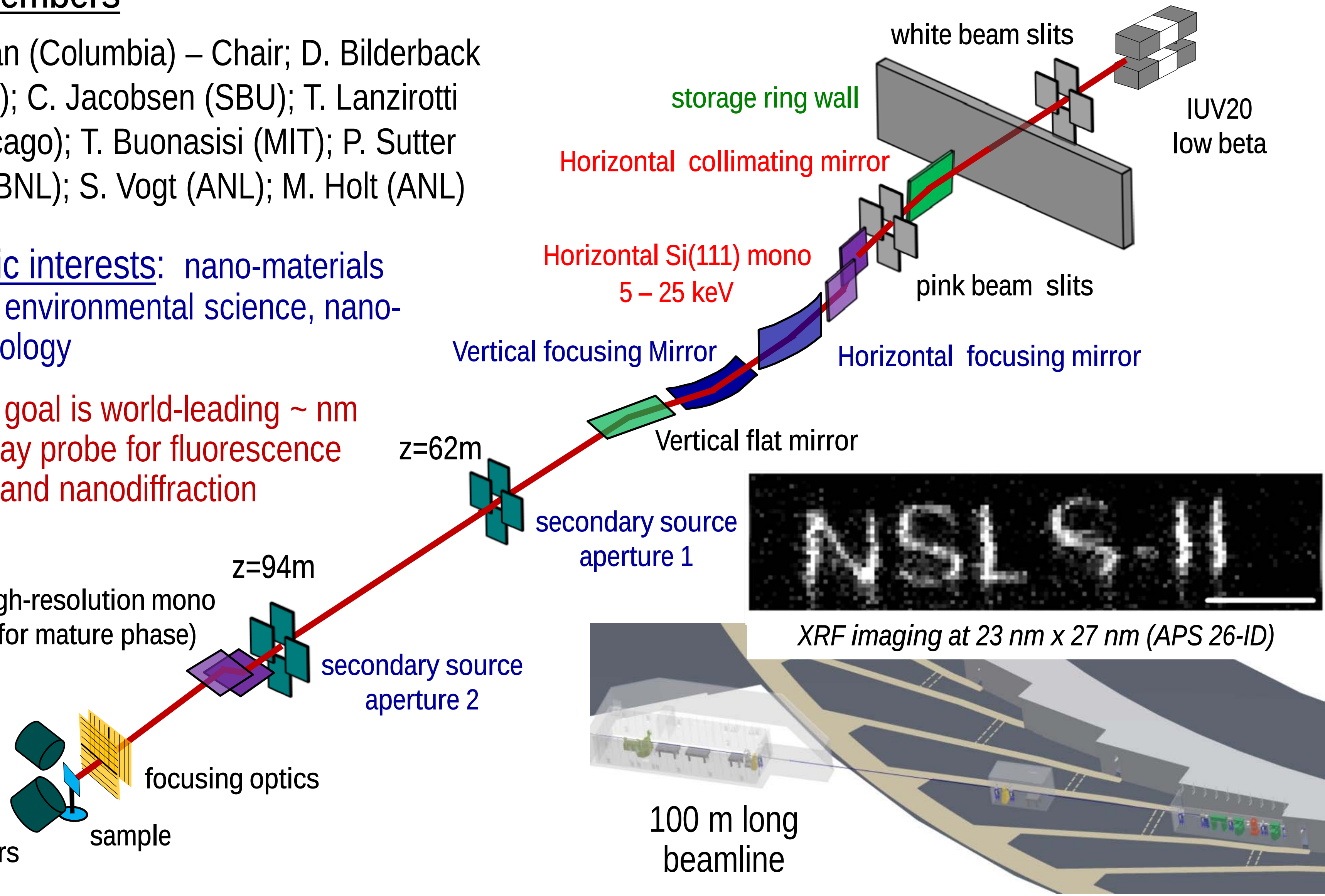
Hard X-ray Nanoprobe (HXN)

BAT members

C. Noyan (Columbia) – Chair; D. Bilderback (Cornell); C. Jacobsen (SBU); T. Lanzirotti (U. Chicago); T. Buonasisi (MIT); P. Sutter (CFN - BNL); S. Vogt (ANL); M. Holt (ANL)

<u>Scientific interests</u>: nano-materials science, environmental science, nano-biotechnology

Ultimate goal is world-leading ~ nm scale x-ray probe for fluorescence imaging and nanodiffraction



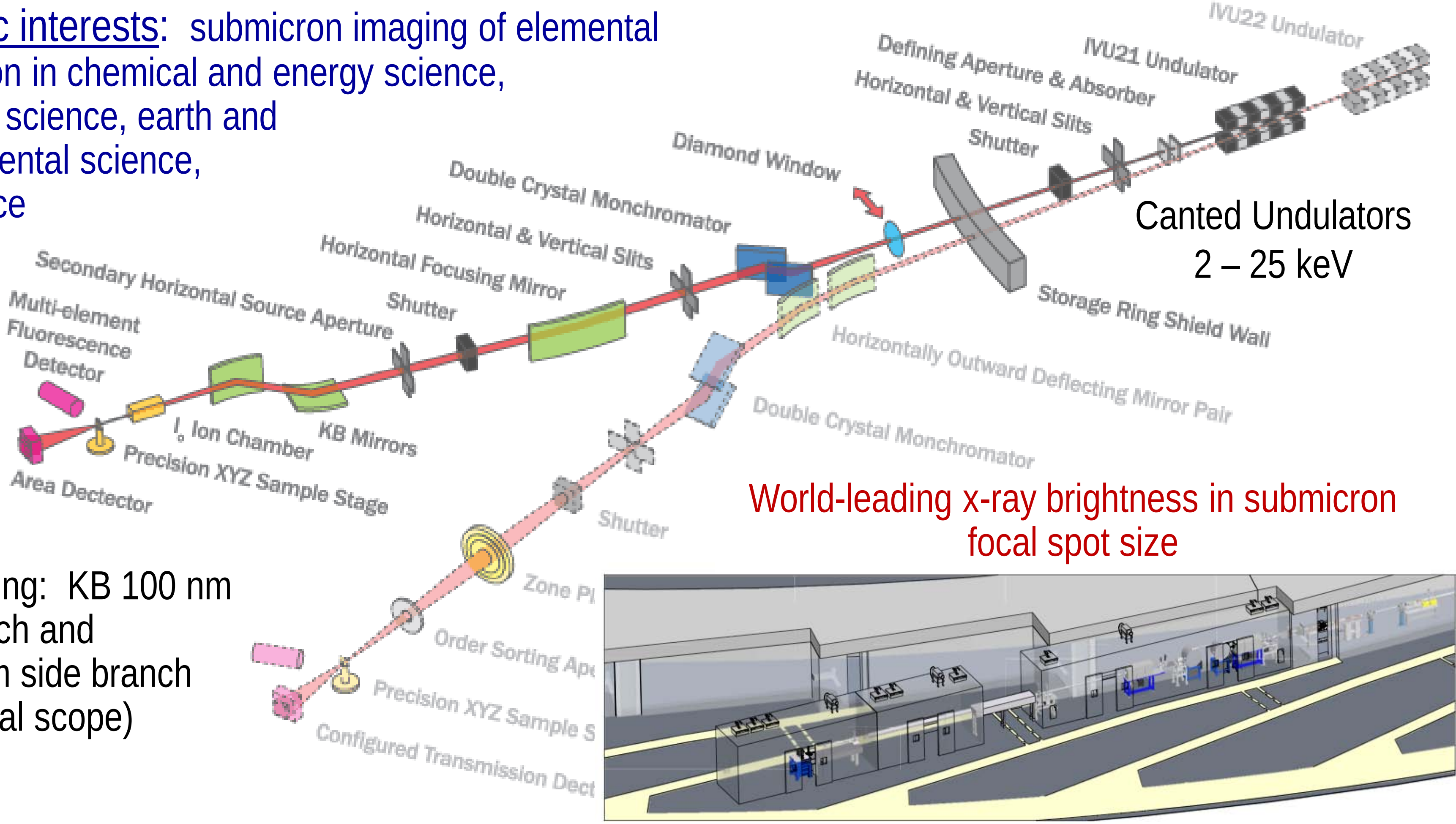
XRF imaging at 23 nm x 27 nm (APS 26-ID)

Sub-μm Resolution X-ray Spectroscopy (SRX)

<u>BAT members</u>

T. Lanzirotti (Chicago) – Chair; S. Sutton (Chicago); S. Vogt (ANL); G. Woloschak (NU); M. Rivers (Chicago); P. Eng (Chicago); L. Miller (NSLS); J. Fitts (BNL); P. Northrup (BNL); K. Jones (BNL)

<u>Scientific interests</u>: submicron imaging of elemental distribution in chemical and energy science, materials science, earth and environmental science, life science



Canted Undulators
2 – 25 keV

World-leading x-ray brightness in submicron focal spot size

XRF imaging: KB 100 nm main branch and FZP 30 nm side branch (not in initial scope)

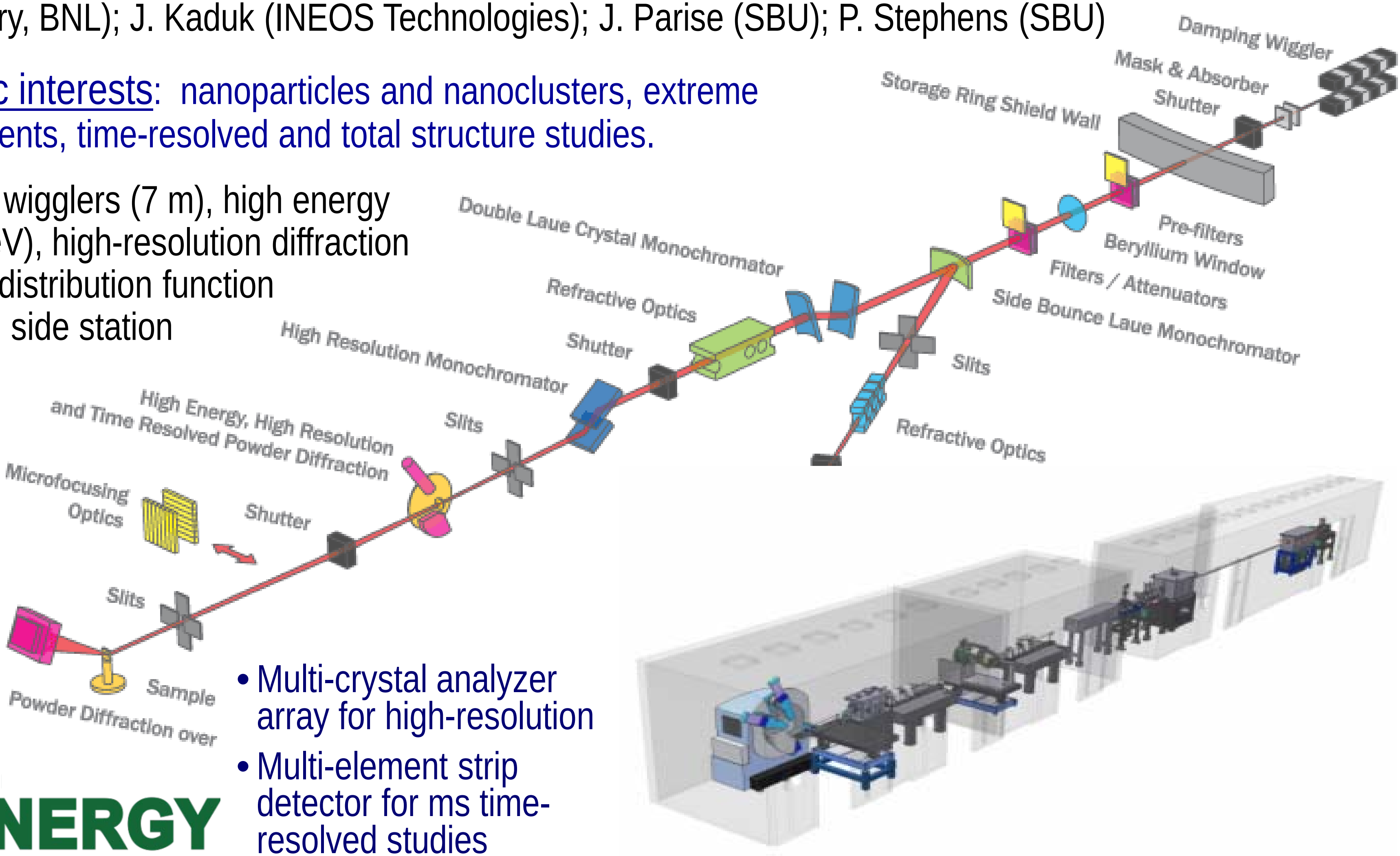
X-ray Powder Diffraction (XPD)

<u>BAT members</u>

S. Billinge (Columbia/BNL) – Chair; P. Chupas (APS, ANL); L. Ehm (SBU/BNL); J. Hanson (Chemistry, BNL); J. Kaduk (INEOS Technologies); J. Parise (SBU); P. Stephens (SBU)

<u>Scientific interests</u>: nanoparticles and nanoclusters, extreme environments, time-resolved and total structure studies.

Damping wigglers (7 m), high energy (30-80 keV), high-resolution diffraction plus pair-distribution function studies in side station



- Multi-crystal analyzer array for high-resolution
- Multi-element strip detector for ms time-resolved studies

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Development Process of Additional Beamlines

2010 Call for Beamline Development Proposals
National Synchrotron Light Source II

March 26, 2010

- Scope
  - All areas of science and beamline types – ID, BM, 3PW, IR
  - Independent of funding source or implementation approach
- Science case and technical requirements
- Results
  - 54 Beamline Development Proposals received by Jun 21
  - Reviewed by Science Advisory Committee & Study Panels
  - 34 BDPs approved; Results announced Oct 4, 2010

Critical Dates

Call for proposal issued
March 26, 2010

Informational session
Wednesday, April 14, 2010
Agenda | Video (RealPlayer)

Letter of Intent due Monday,
April 26, 2010
(submitted LOIs)

Beamline development workshops
April-June 2010

Beamline proposal due Monday,
June 21, 2010

SAC review
Summer 2010

Related Materials

Proposal template

NSLS-II Beamline Development Policy

NSLS-II Source Properties

NSLS & NSLS-II User Access Policy (draft)

Conceptual Design Report for Six Project Beamlines

Background Beamline Information

34 Approved Proposals Posted On-Line

http://www.bnl.gov/nsls2/beamlines/2010BeamlineProposal-Approved.asp

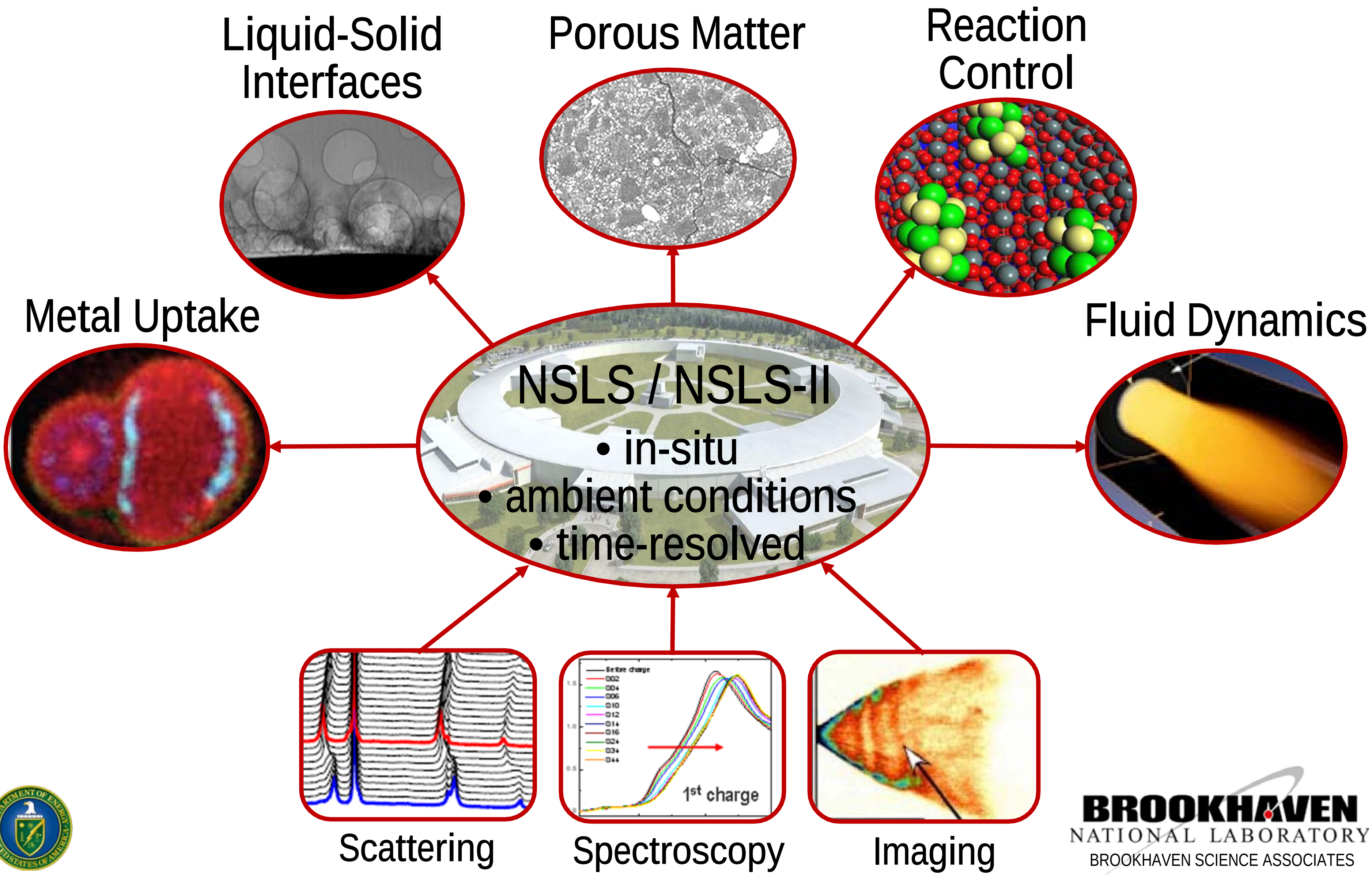
2010 Beamline Development Proposals — Approved Proposals

Proposal Results Announcement

Acronym	Title	Spokesperson	Type	Information
4DE	4-Dimensional Studies in Extreme Environments	Donald J. Weidner, Stony Brook University	1	Slide
ABS	A Highly Automated Instrument for Static X-ray Scattering Measurements of Biological Molecules in Solution	Lin Yang, BNL	1	Slide
AIM	Advanced Infrared Microspectroscopy	Lisa Miller, BNL	1	Slide
AMX	Flexible Access Macromolecular Crystallography at an Undulator Beamline	Dieter Schneider, BNL	1	Slide | Proposal
BMM	Hard X-ray Absorption Spectroscopy and Diffraction - Beamline for Materials Measurements	Daniel Fischer, NIST	2	Slide | Proposal
CDI	Coherent X-ray Diffraction	Ian Robinson, University College London	1	Slide | Proposal
CMS	Complex Materials Scattering	Kevin Yager, BNL	1	Slide | Proposal
ESM	Electron Spectro-microscopy for Fundamental Studies of the Physics and Chemistry of Materials	Elio Vescovo, BNL	1	Slide
FIS	Frontier Synchrotron Infrared Spectroscopy Beamline Under Extreme Conditions	Zhenxian Liu, Carnegie Institution of Washington	1	Slide
FMX	Frontier Macromolecular Crystallography at an Undulator Beamline	Robert Sweet, BNL	1	Slide | Proposal
FXI	A Superconducting Wiggler Long [illegible] field Imaging at [illegible]	[illegible]	1	Slide
HXI	[illegible]	[illegible]	1	Slide
IRI	[illegible]	[illegible]	1	Slide
ISR	[illegible]	[illegible] Ludwig, Boston University	1	Slide | Proposal
ISS	[illegible] Spectroscopy	Bruce Ravel, NIST	1	Slide | Proposal

2011 Call Beamline Development Proposals
Letter of Intent: due March 28, 2011
Proposal: due June 6, 2011

sc-CO_2/Materials Research at NSLS/NSLS-II



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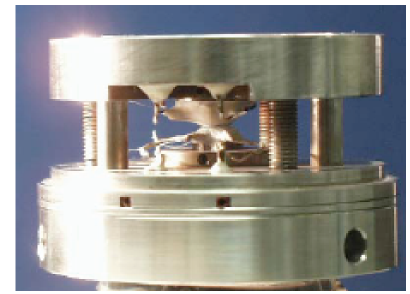
Four Dimensional Extreme Environments ($4DE^2$)

$4DE^2$ at NSLS-II

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

Examples of Science Areas & Impact

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



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

Beamline Capabilities

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

SOURCE: Superconducting wiggler

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

SPATIAL RESOLUTION: ~1 – 5 μm

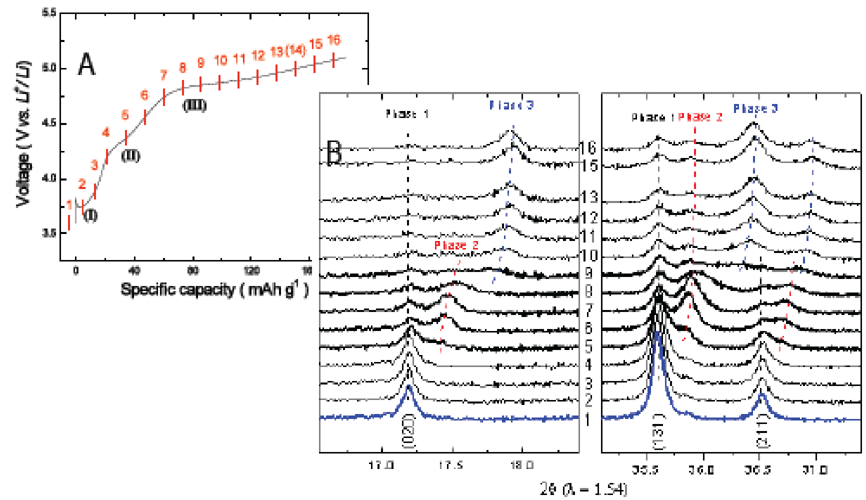
Powder Diffraction for In-Situ Studies of Structural and Chemical Transformations

IXD at NSLS-II

- As a powder diffraction beamline with medium x-ray energy, IXD will enable *in situ* XRD measurements with time scale of seconds and decent Q-space resolution, and is suitable for element selective and surface sensitive XRD methods.
- The state of art capabilities for *in situ* XRD measurements under various conditions will find broad applications in advanced energy material studies and industrial R & D.

Examples of Science Areas & Impact

- BATTERIES: The *in situ* XRD measurements on cathode and anode materials of Li-ion batteries make it possible to monitor the structural transformation during the charge-discharge cycles, help in understanding the electrochemistry process and the search for better materials for energy storage.
- FUEL CELLS: Grazing angle XRD to reveal the composition and morphology of the surface layers of the Ni-YSZ anode as applied in novel full cell design
- HIGH TEMPERATURE ALLOYS: *In situ* residual stress measurements under high temperatures on the surface and coating materials of high temperature alloys such as used in solid oxide fuel cell and gas turbines



(A) 1st charge curve of **C**-$LiFe_{1/4}Mn_{1/4}Co_{1/4}Ni_{1/4}PO_4$ **(B)** *In situ* XRD patterns of **C**-$LiFe_{1/4}Mn_{1/4}Co_{1/4}Ni_{1/4}PO_4$ during 1st charge. A partially delithiated solid solution phase (phase 2) is identified between the two normally observed end phases in $LiFePO_4$ cathodes. K Nam et al, Electrochem. Commun., 11, 913 - 916 (2009).

Beamline Capabilities

TECHNIQUE(S): *in situ* powder diffraction studies under high temperature, high pressure, reaction gas flow and charge-discharge cycles

SOURCE: three-pole wiggler

ENERGY RANGE / RESOLUTION: 6 to 25 keV / 1.3 x10^{-4}

BEAM SIZE: vertically ~ 200 μm , horizontally 0.2 to 2 μm

Quick X-ray Absorption and Scattering (QAS)

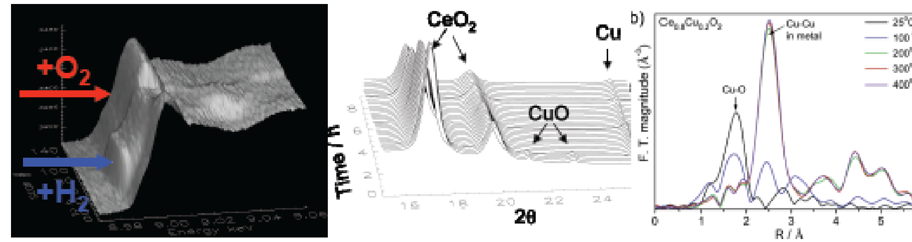
QAS at NSLS-II

- 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
- Will probe complex interactions in nanoscale systems at the time scale from tens of ms to hours and length scale from Å to µm

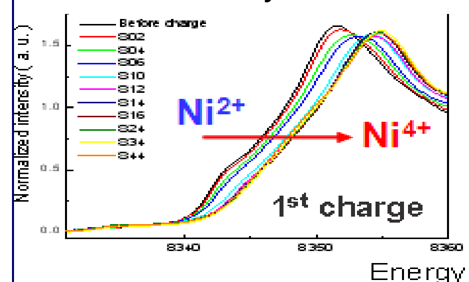
Examples of Science Areas & Impact

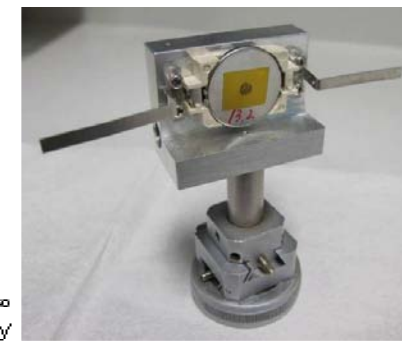
- CATALYSIS: Investigations of structure, kinetics, dynamic and reactivity during in situ transformations with 10 ms time resolution
- GLASSES AND MEMORY ALLOYS: Understanding correlations between glass-forming ability and structure o novel glasses and phase-change materials
- ENVIRONMENTAL SCIENCE: Kinetics of rapid chemical processes on mineral surfaces and soils
- ENERGY GENERATION AND STORAGE: Understanding physical and chemical processes in batteries and fuel cells

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



Ni K-edge in-situ XAS in battery cell





➡ Nickel is responsible for most of the capacity.

Beamline Capabilities

TECHNIQUES: x-ray absorption spectroscopy and x-ray diffraction with 10 ms time resolution

MONOCHROMATORS: double crystal (slow scanning) and channel cut (Quick EXAFS)

SOURCE: three-pole wiggler

Spokesperson: Anatoly Frenkel, Yeshiva University

X-ray Fluorescence Microscopy (XFM)

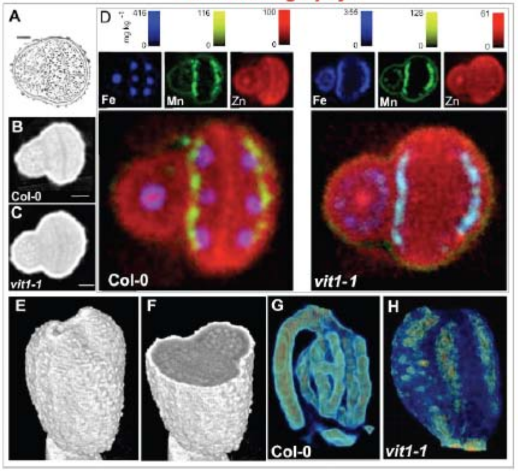
XFM at NSLS-II

- Will provide spatially-resolved characterization of elemental abundances and speciation in “as-is” samples at µm scale with high throughput. Crucial for biological screening.
- Optimized for microfocused extended x-ray absorption fine structure (µEXAFS) spectroscopy; 4 to 20 keV.
- Capabilities for NSLS-II’s three pole wigglers excellent sources for µEXAFS and XFM will provide in a 1-10 µm beam flux densities **two orders of magnitude higher** than at the NSLS. This will be world-leading for full µEXAFS.

Examples of Science Areas & Impact

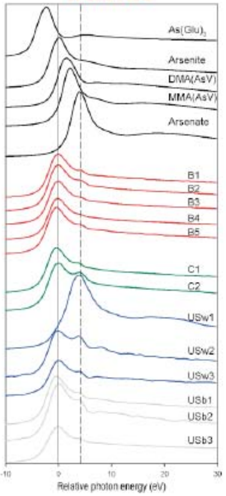
- Molecular speciation of contaminants in the environment at the microscale
- Genetic control of metal ion uptake, transport and storage in plants relevant to agriculture and bioenergy
- Biogeochemistry of nanotoxins in the environment
- Metal ions in health and disease
- Mineral-fluid interface reactions relevant to carbon sequestration
- Early solar system properties inferred through analysis of extraterrestrial materials
- Characterization of paleontological, archeological and cultural heritage artifacts

3D in-vivo imaging of metals in Arabidopsis using fluorescence microtomography



Kim, et al., Science, 2007

As XANES spectroscopy from rice



Meharg et al., ES&T, 2008

XFM is well-suited for evaluation of how specific genes influence the uptake of nutrients and contaminants in plants. It will provide non-destructive, three dimensional characterization in-vivo with high throughput. XFM’s strengths in µEXAFS can evaluate how chemical form influences bioavailability or toxicity.

Beamline Capabilities

TECHNIQUE(S): µm x-ray fluorescence (XRF), x-ray absorption fine structure (XAFS) spectroscopy, x-ray diffraction (XRD) and fluorescence computed microtomography (FCMT)

SOURCE: three-pole wiggler

ENERGY RANGE / RESOLUTION: 4 to 20 keV / 1 eV

SPATIAL RESOLUTION: 1 – 10 µm variable

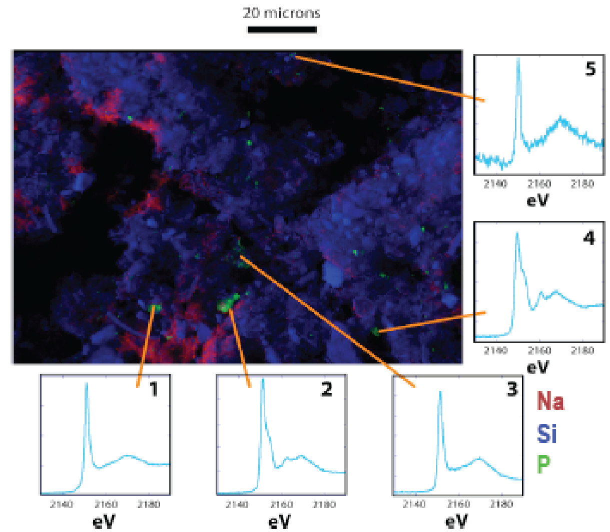
Tender Energy X-ray Absorption Spectroscopy

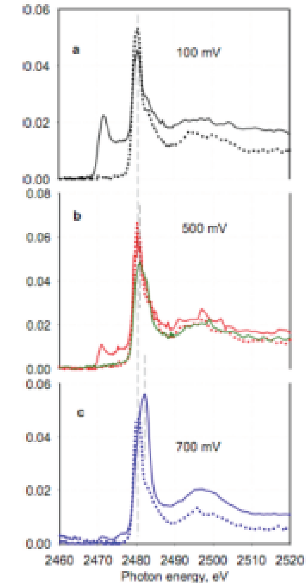
TES at NSLS-II

- Will enable **spatially-resolved** and ***in-situ*** studies of speciation and local structure by x-ray absorption spectroscopy in a *non-vacuum environment*
- Chemical sensitivity to **key lighter elements** Mg through Ti, and advantageous heavier-element L and M edges
- Optimized for the **NSLS-II dipole bend** source -- high brightness over a tunable spatial resolution and energy scanning across 1.2-8 keV will be world-class

Examples of Science Areas & Impact

- **Catalysis**: Materials (zeolites, thin films, nanomaterials), reaction mechanisms and intermediate species, poisoning
- **Energy Materials**: Photovoltaic, fuel cell, battery and superconducting (nano)materials
- **Environmental/Earth Science**: Biogeochemical and redox processes, contaminant behavior and remediation
- **Climate**: Terrestrial and marine C cycling, carbonate (bio) mineralization, geologic record of climate change
- **Sustainability**: Nutrient (P, S, K, Ca, Mg, Fe) cycling, transport and bioavailability, biofuel/biomass productivity





Example applications: Above: natural systems, e.g. P in sediments, exhibit species and distribution heterogeneities at the submicron to mm scale. Diaz et al., *Science*, 320, (2008). At right, combined *in-situ* XAS and electrochemistry examines S speciation during poisoning of fuel-cell catalyst (Baturina et al., unpublished). TES will enable fast-scanning high-quality EXAFS and XANES at tunable spatial resolution to better address these and other real systems.

Beamline Capabilities

Techniques: x-ray fluorescence and spectroscopic imaging, high-performance and *in-situ* EXAFS

Source: dipole bend magnet

Energy Range: 1.2 to 8 keV (optimized for 1.2-5 keV)

Spatial Resolution, Flux: 1x1 mm to 1x1 μm; up to $3x10^{12}$ ph/sec flux

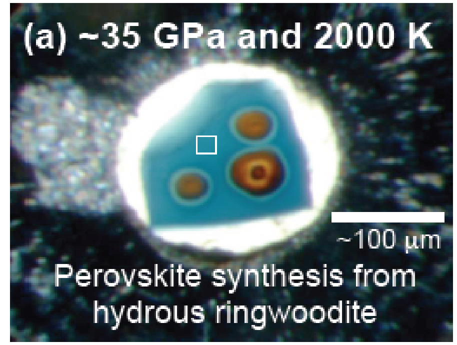
Frontier IR Spectroscopy at Extreme Conditions

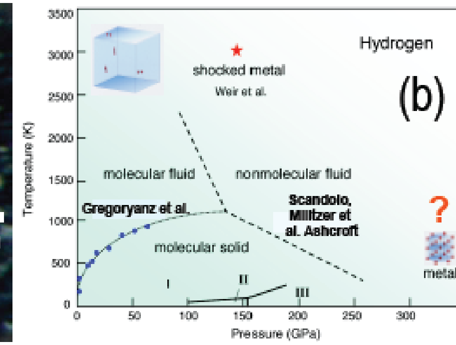
FIS at NSLS-II

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

Examples of Science Areas & Impact

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





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

Beamline Capabilities

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

SOURCE: large-gap (90 mm) dipole magnets

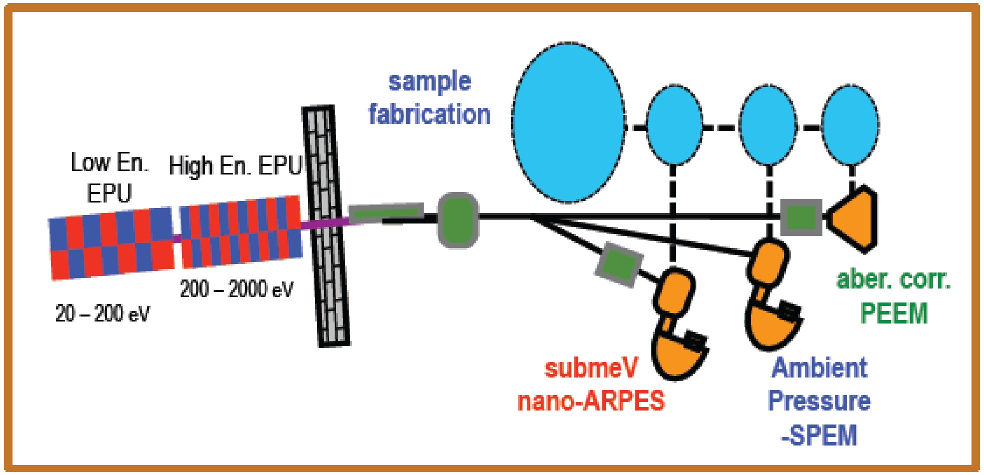
ENERGY RANGE / RESOLUTION: 50 – 10000 cm^{-1} / 1 cm^{-1}

SPATIAL RESOLUTION: diffraction limited resolution in the entire energy range

Electron Spectro-Microscopy (ESM)

ESM at NSLS-II

- A suite of instruments to probe, characterize and design functional materials based on their electronic structure
- Overcoming traditional barriers: spatial averaging & ambient pressure
- Advancing photoemission to make contact with real devices and applications



Schematic of proposed ESM beamline

Examples of Science Areas & Impact

- ARPES from ~ 300 nm samples (inside polycrystalline grains)
- SP-ARPES from multi-domain samples in zero applied field (sampling of single magnetic domains)
- Catalysis @ relevant partial pressures
- Chemical composition maps at ambient pressure
- Combinatorial investigation of multi-parameter samples
- Photoemission from real (micro-)devices (spintronics, fuel cells, etc.)

Beamline Capabilities

TECHNIQUE(S): nano-ARPES, SP-ARPES, AP-SPEM, XPEEM

SOURCE: two EPUs

ENERGY RANGE / RESOLUTION: 20 – 2000 eV
< 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

SPATIAL RESOLUTION: ~200x300 nm (KB), ~50 nm (ZP)

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Spokesperson: Elio Vescovo, Brookhaven National Laboratory

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NEXT Beamlines

ESM Electron Spectro-Microscopy

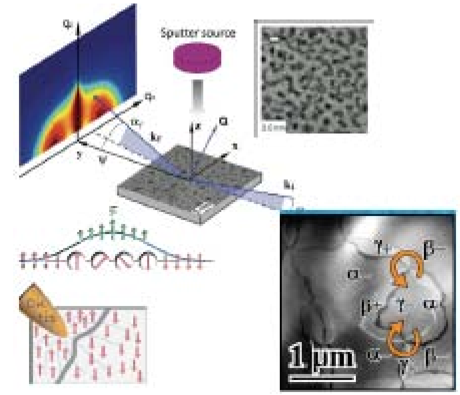
FXI Full-field X-ray Imaging from μm to nm

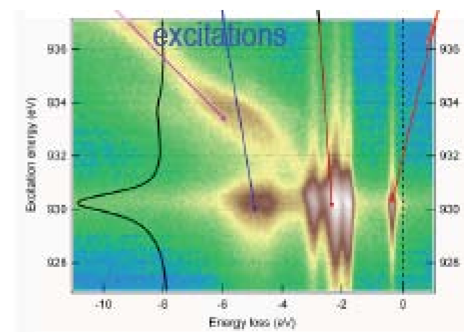
ISS Inner Shell Spectroscopy

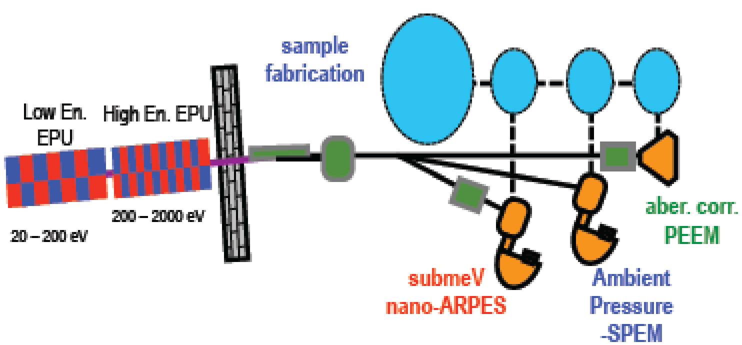
ISR Integrated In-Situ & Resonant X-Ray Studies

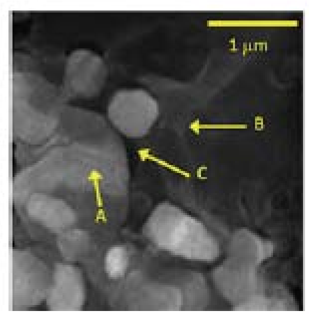
SIX Soft Inelastic X-ray Scattering

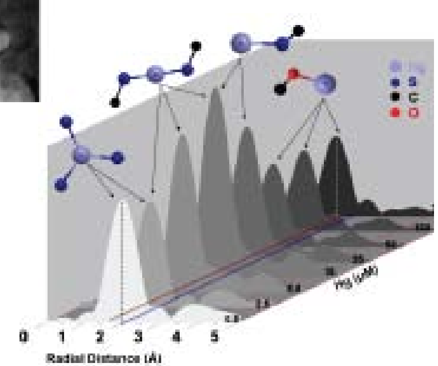
SMI Soft Matter Interfaces



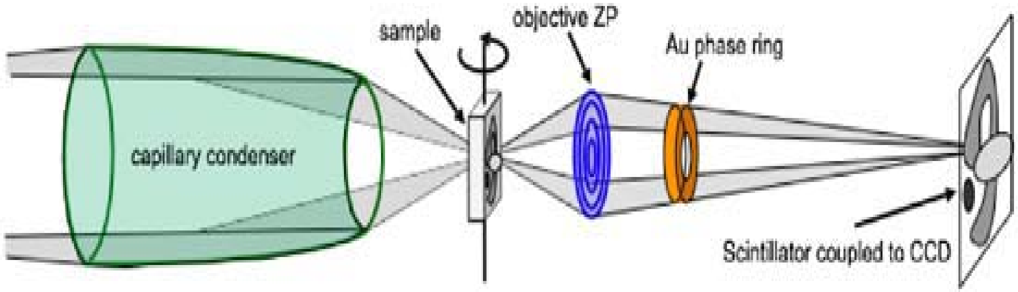






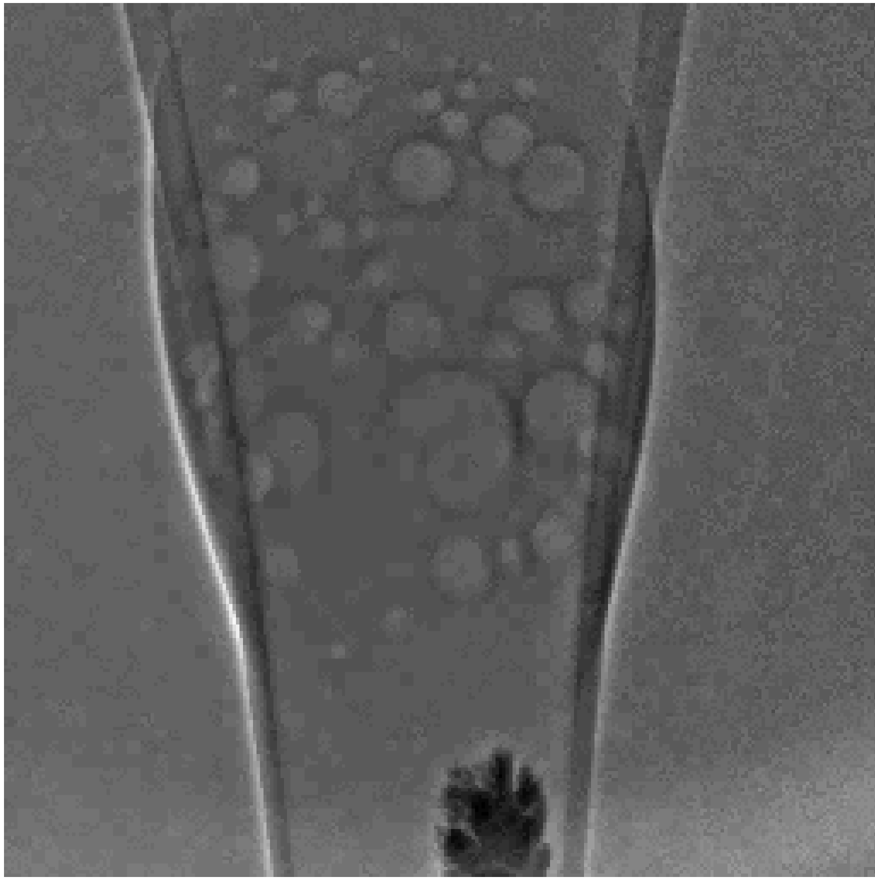


Real Time Nano-Imaging of Electrochemical Growth



- Modern x-ray lenses (Fresnel zone plates) and bright x-ray sources enable x-ray microscopic imaging at nano-scale spatial resolutions

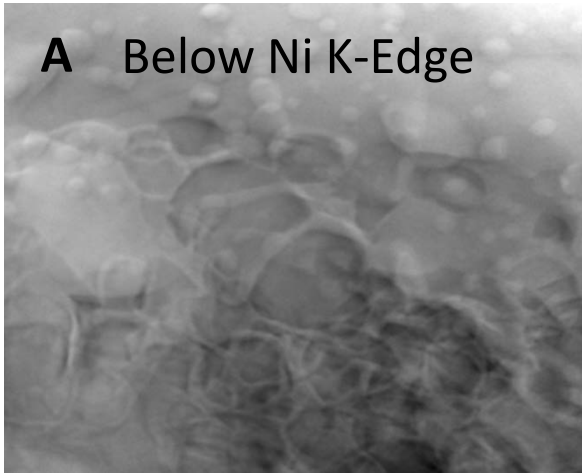
- Example: In-situ studies of dendritic growth of Cu in $CuSO_4$ solution by electrical potential (APS 32-ID, 8 keV)
  - Frame rate: 100 msec/frame
  - TXM FOV: 22 μm

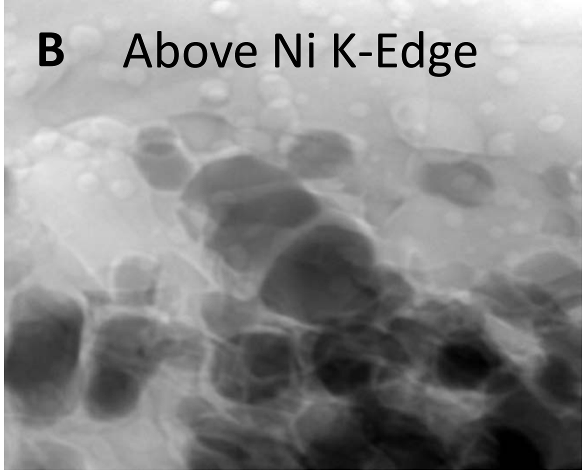


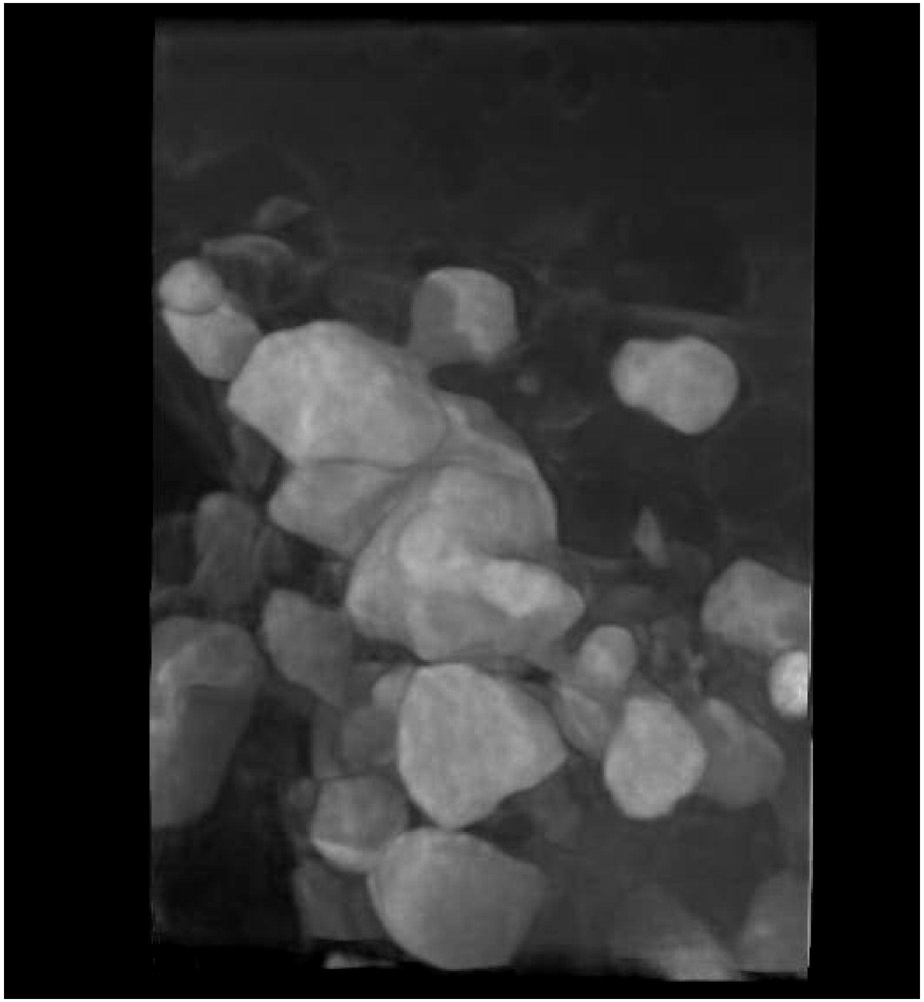
J. Yi, S. Wang (ANL), Y-K. Hwu (Acad. Sinica, Taiwan), J. H. Je (POSTECH, Korea), Y. S. Chu (NSLS-II, BNL)

Element-Specific Nano-Tomography

Solid Oxide Fuel Cell (SOFC) Pore Structures: Yittria Stablized Zirconia YSZ (electrolyte) / NiO (anode)



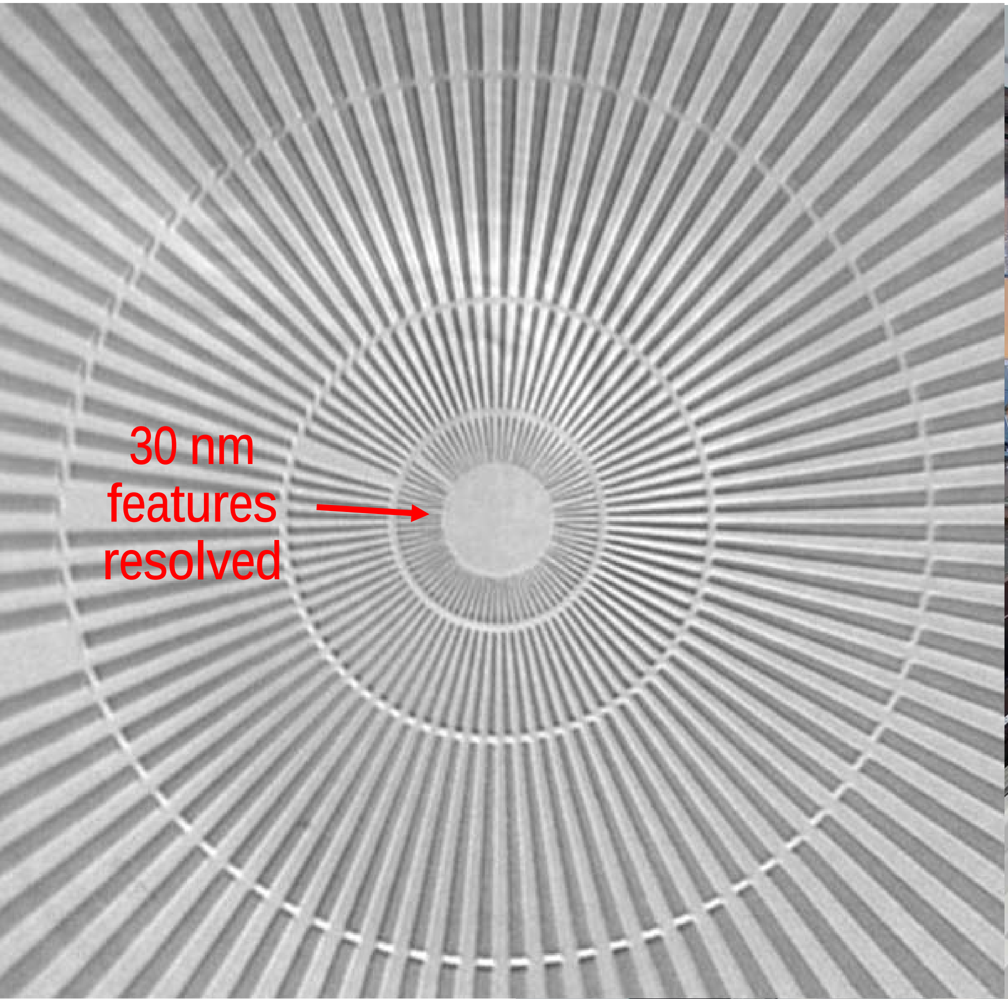


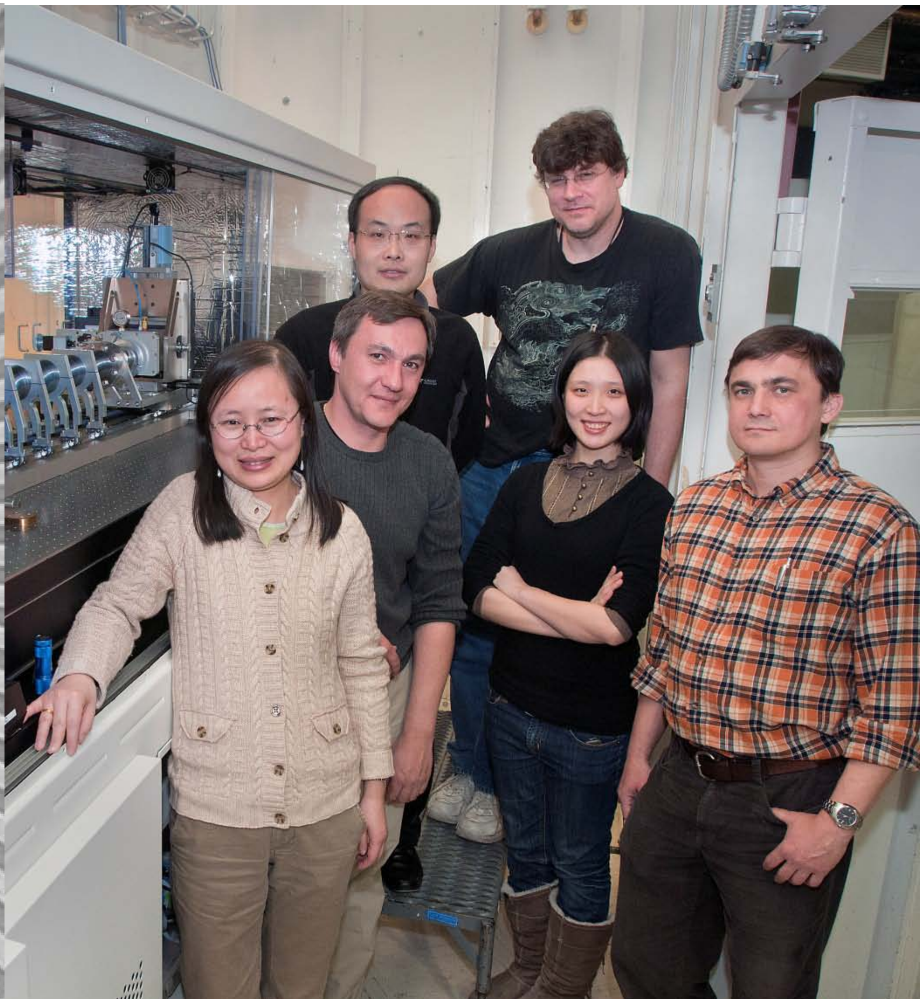


W. Chiu (U. Conn.)
Y.S. Chu (NSLS-II, BNL)

New Transmission X-ray Microscope at NSLS

- New TXM (~$2M funded by DOE ARRA) has been installed at X8C and is being commissioned
- TXM will be transitioned to the Full-field X-ray Imaging (FXI) beamline at NSLS-II

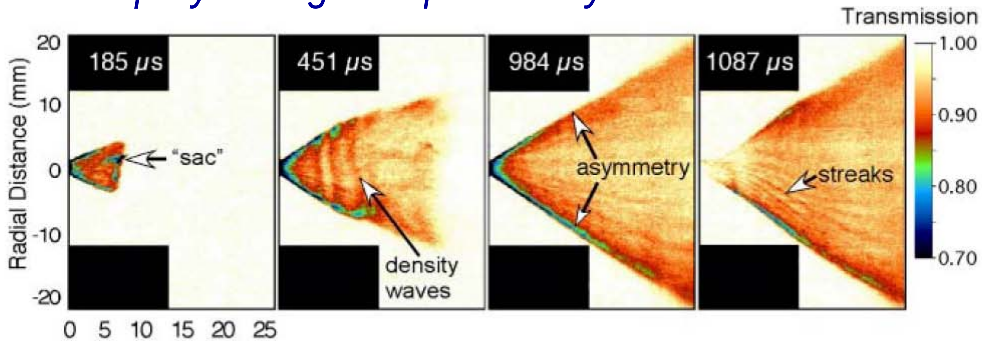


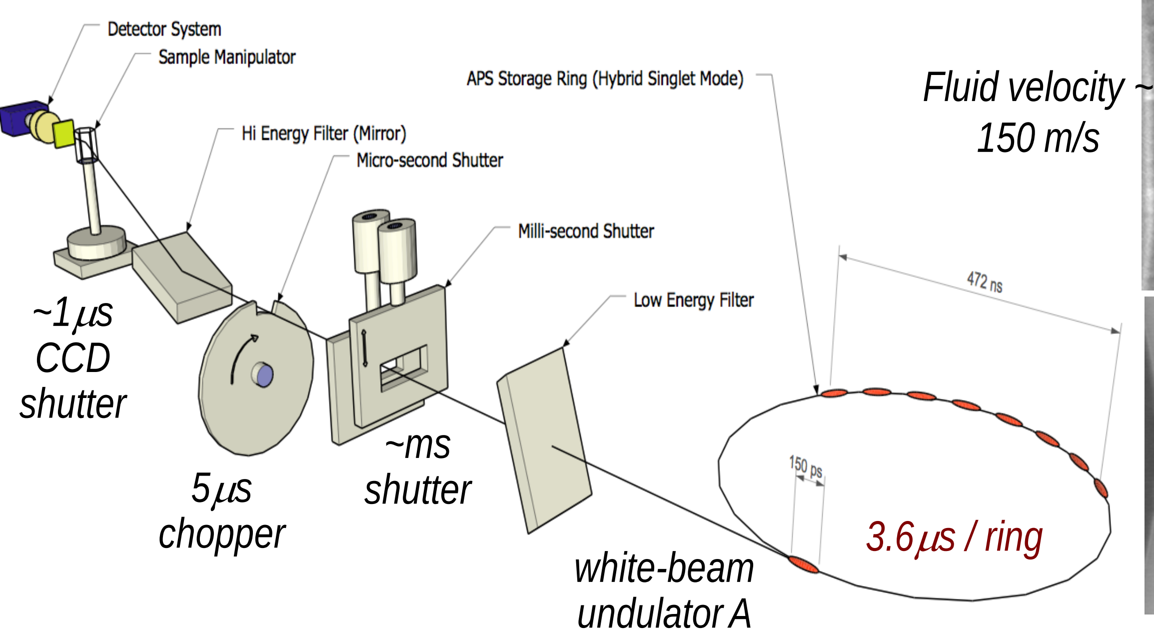


High-speed Imaging and Single-Pulse Flash Imaging

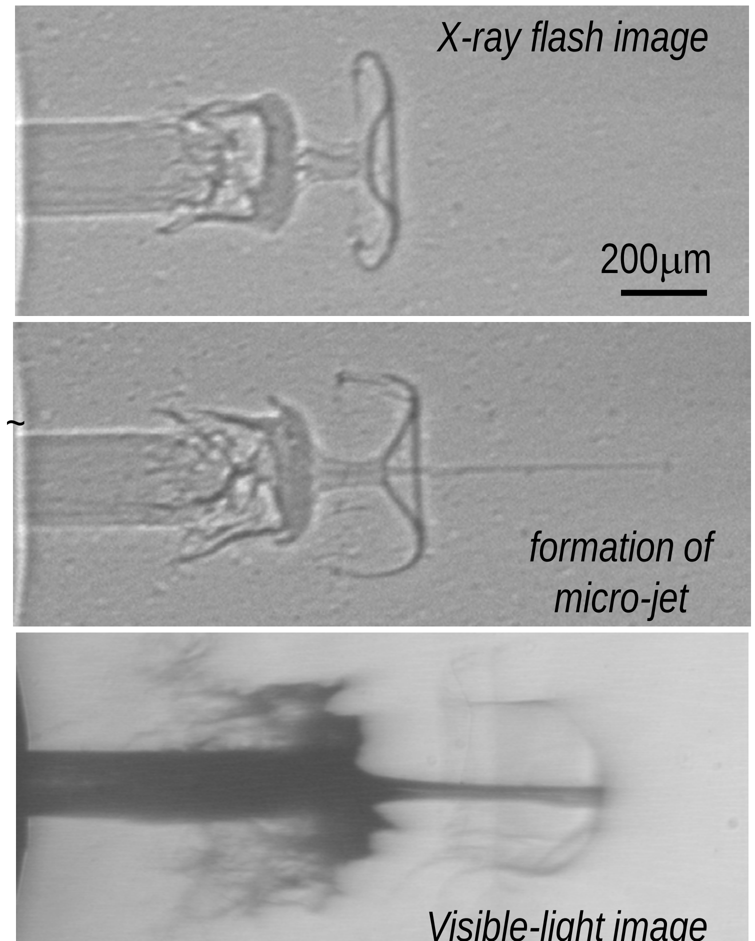
Kamel Fezzaa, Wah-Keat Lee, Jin Wang, et al. (APS)

Fuel spray in engines: pixel array detector





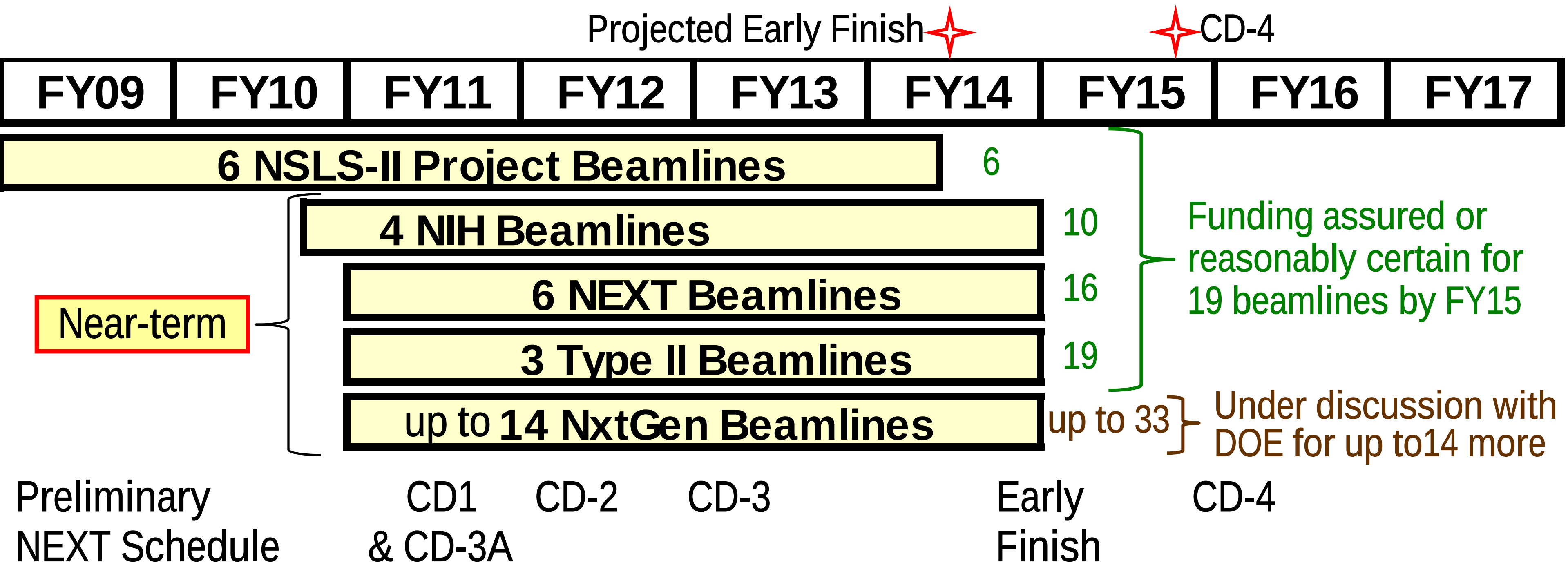
Single-pulse X-ray flash imaging at 150ps



U.S. DEPARTMENT OF ENERGY

BROOKHAVEN NATIONAL LABORATORY
BROOKHAVEN SCIENCE ASSOCIATES

Beamline Development Schedule and Summary



Summary

- NSLS-II is pursuing an aggressive schedule to ramp up cutting-edge experimental facilities in broad areas of science and technology
- Many experiments may be pursued at existing NSLS to jump start research programs discussed here at this Workshop
- We are excited to work with scientific community to develop science programs