Additive Manufacturing Scattering facility (AMS): A Tool for Process and Materials Certification

Klaus Attenkofer, Alessandra Colli, Eric Dooryhee, Gary Halada (Stony Brook)

Potential Partners or PI's:

Ron Jones (NIST), Dan Fischer (NIST), Todd Turner (AFRL, Materials and Manufacturing Directorate)

September 11, 2018

Rationale for the Proposed New Beamline

- Concise one-sentence on what is being proposed

A high energy (powder) diffraction facility as part of the certification process for additive manufacturing. The facility will focus on strain/stress and crystallite size and orientation determination of defined sample geometries using beam sizes of roughly 1mm. Its major competitive advantage will be a dedicated setup with fully integrated and automated data pipeline infrastructure compatible to NIST data basis (potentially also other defense partners), allowing data mining approaches for printing process optimization. The dedicated instrument will provide the continuous availability, necessary to satisfy the certification needs. Additionally, there will be a Rotational and Axial Motion System (RAMS) system integrated to allow operando fatigue measurements. The instrument will be fully integrated within the suite of high energy instruments at CHESS and APS.

- Science case, including national and/or global context

The main limitation of current additive manufacturing processes is the lack of certified printing protocols allowing to produce materials with defined materials qualities and ultimately a reliable printed part. This was recognized by NIST and other institutions who currently try to establish data basis for the different polymers, metallic alloys and ceramic materials and its printing and post-treatment parameters. High energy powder diffraction is an accepted and well understood tool to characterize strain/stress, crystallinity, and phase segregation for metallic and ceramic materials.

Using the data base and data mining approach, correlations between materials structure and fatigue effects can be established and finally investigated in detail by microscopic 3D tools. The NSLS-II instrument will focus on the data base aspects of this approach.

Rationale for the Proposed New Beamline

- User interests and demands (still in discussion with different communities)
  - NIST: Additive Manufacturing Benchmark Test Series (AM-Bench)
  - AFRL: as part of a large additive manufacturing institute
  - Potentially other defense organization
  - Industrial developers (for example in discussion with GE, Samsung)
  - Academic users in the field of engineering and materials sciences

Rationale for the Proposed New Beamline

- NSLS-II portfolio impact
  - Integrates fully into the suite high energy diffraction beamlines and builds on the strength of data management established at NSLS-II
  - Builds on the strong NIST-NSLS-II partnership established with SST and BMM (this includes data exchange, and data pipeline development)
  - Integrates with BNL additive manufacturing initiative
  - Builds on strong local academic network to provide critical mass
- Scope, performance, and feasibility, including implementation as a new end station or as a new branch line on an existing beamline

The beamline will be most likely a side station of an existing beamline (HEX). Currently, there are two versions under discussion:

- mono energy version (for example HEX B-hutch); this option will require a bragg monochromator in the energy range of roughly 100keV and a typical high energy end station equipment (Pilatus 6M with GaAs sensor). Development of the data infrastructure will be main effort.
- pink-beam version (for example HEX D-hutch); This option will require a filter box and potentially some focusing elements; main risk is the availability of an appropriate spatial resolving high energy detector system.

Soft X-Ray Scattering and Spectroscopy

Stuart Wilkins

U.S. DEPARTMENT OF ENERGY | Office of Science

BROOKHAVEN NATIONAL LABORATORY | National Synchrotron Light Source II

Program Description & Goals

- Scattering and Spectroscopy combined with Imaging to solve science problems in the fields of Quantum Materials and Catalysis & Energy Research.
  – Study heterogeneity at the nanoscale.

- Facilitate research in the area of Quantum Materials by developing and operating a cutting edge suite of experimental endstations for spatially resolved condensed matter probes.

- Facilitate research using x-ray spectroscopy to study real-life materials under real conditions for energy and catalysis research.

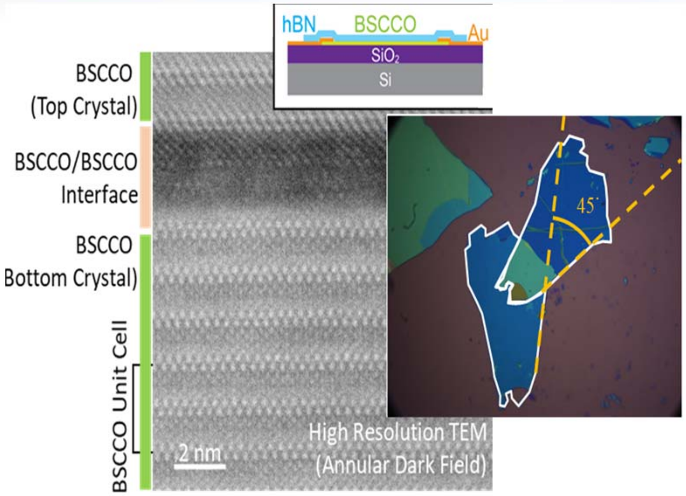
NSLS-II Soft X-ray Expertise and Needs

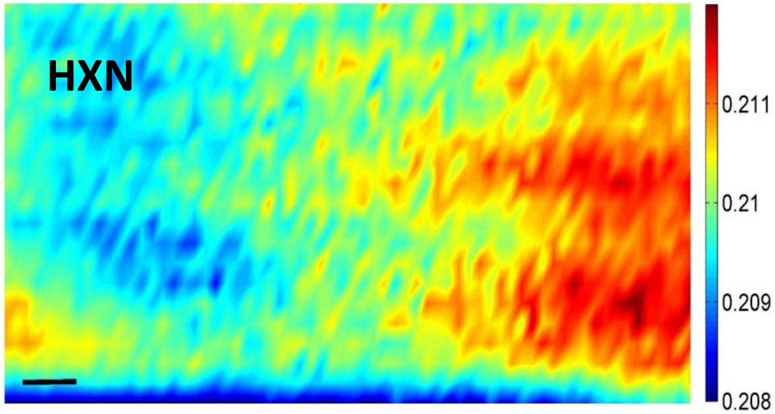
- NSLS-II has expertise in a number of soft x-ray science and techniques:
  - Optical Design of Soft X-ray Beamlines – (SIX very novel spectrometer)
  - RIXS (SIX, world leading resolution)
  - ARPES/PEEM (ESM, CFN, high intensity 1 um resolution)
  - Coherent Imaging / (Yong's Program, CSX)
  - Operando Spectroscopy (Klaus's Program, IOS)

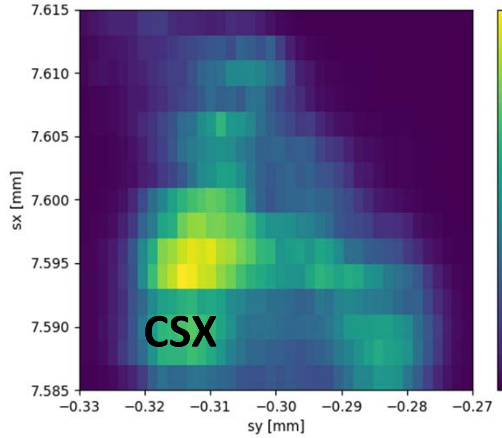
- In addition to these areas NSLS-II needs to build expertise in:
  - Sample handling / transport / automation for UHV
    - Solved problem in other domains, need to learn.
  - Detectors for soft x-rays (CP, CMOS, FastCCD2)
    - Usually not commercially available. Often more experimental than the experiment.
  - Data analysis for spatially resolved datasets.
    - How do we process data for spatially resolved ARPES measurements. What do they mean?
    - CDI when you are on an absorption edge – magnetic domain imaging.
  - True multimodal Experiments

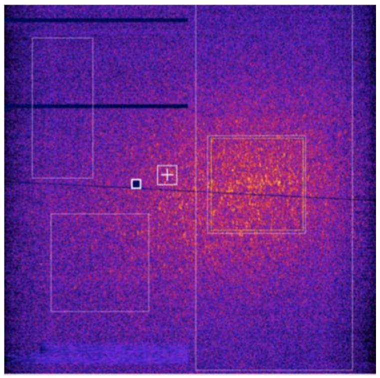
Why NSLS-II?

- NSLS-II (BNL) is a center for excellence in imaging (HXN) and RIXS (SIX)
- Multimodal approaches will be essential as performance depends on many factors (crystalline structure [HXN], electronic structure [CSX, ARI], excitations [SIX, ARI])



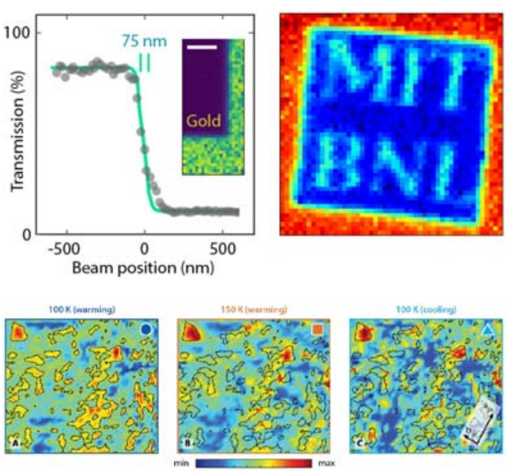


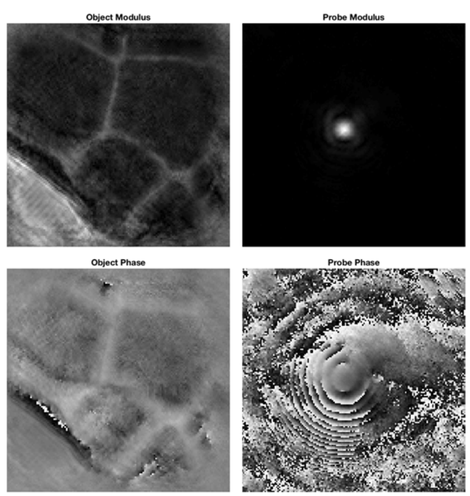




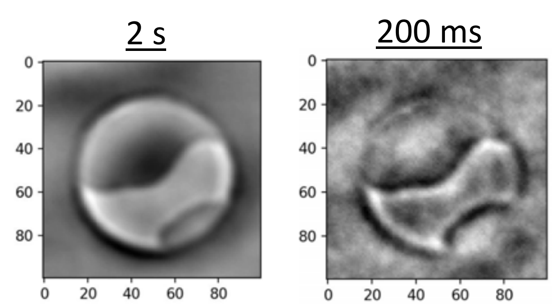
Recent Development with Imaging

- Recent imaging directions @ CSX
  - Magnetic Domain Imaging in $NdNiO_3$ Imaging Skyrmions by soft x-ray Holography
  - Bragg CDI from ASI
  - Biomineralization in oyster shells (Ca L-edge)





Holography @ CSX



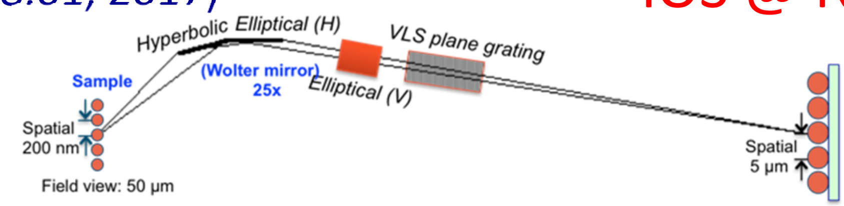
ALS Plans

From status report by Roger Falcone (ALS Division Director), Sept. 2017:

Current projects are enhancing ALS capabilities and preparing for ALS-U: *Catalysis & energy materials*

- COSMIC – coherent imaging (Beamline 7.0.1, 2017) CSX @ NSLS-II
- AMBER – renewable energy spectroscopies; in-situ soft x-ray tools (6.0.1, PNNL, 2018)
- wetRIXS, iRIXS, qRIXS (Beamline 8.01, 2017) IOS @ NSLS-II

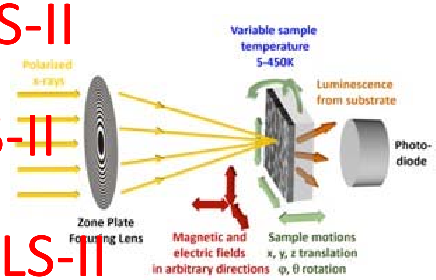
Next up: nano RIXS (NSLS-II)



Current projects are enhancing ALS capabilities and preparing for ALS-U: *Spin & quantum materials*

- MAESTRO – nanoARPES, PEEM (w/Molecular Foundry, 2016) ESM / ARI @ NSLS-II
- QERLIN – resonant inelastic x-ray scattering (w/Moore Foundation, 2018) SIX @ NSLS-II
- Spin-resolved ARPES (2016) ESM @ NSLS-II
- COSMIC – coherent scattering (2017) CSX @ NSLS-II

Proposed quantum materials STXM STXM @ NSLS-II



Gap Analysis – Current Soft X-ray Beamlines

- For quantum materials move from 20th century science (big crystals) to 21st century materials which requires the understanding and control non-heterogeneity and the functioning of nanostructured devices.
- Understanding these will require nanostructure determination of:
  - Crystalline structure -> Nanoprobe (HXN…)
  - Electronic order -> REXS (CSX)
  - Electronic structure -> ARPES (ESM)
  - Electronic Excitations -> RIXS (SIX) NFIR (INF)
- Current tools are pushing into the imaging regeme, but need to develop these instruments further.

Gap analysis

- Build on expertise in RIXS / ARPES and Imaging to produce simultaneous imaging of electronic inhomogeneities (ARI) pushing the ultimate spatial resolution .
- Develop STXM to provide much needed imaging capabilities in the soft region to obtain contrast for QM, ES, SM and Bio?
- Near-field (IR) spectroscopy provides very high spatial resolution (nm) to an important probe of electronic structure / excitations for QM, ES
- Need to address the decaying endstation at IOS. Opertunity to combine RIXS expertise and AP-PES into a novel instrument directly relevant for ES.
- Work on providing multimodal access combining electronic and structural imaging.
- TIN would compliment SIX.

- **ARI**
  - Nano RIXS / ARPES
- **SMF** (Yong) / **STX** (Ron)
  - STXM ES, QM, BIO, SM
- **INF (3 beamlines)**
  - Nearfield for QM and ES
  - IR for spectroscopy at INSPIRE
  - Other Imaging?
- **INSPIRE**
  - Rebuild of IOS
- **CDI / MRN**
  - ?
- **TIN**
  - Hard x-ray RIXS

Future Needs

- ARI
  - Develop endstation for nano-ARPES / nano-RIXS
  - Directly addresses the future needs for quantum materials research
  - Synergistic with SIX and ESM (and possibly CSX, INF, HXN ….)
- Quantum Materials STXM
  - Quantum STXM for quantum materials research, spintronic devices.
  - Cryo – B-Field (<0.1 T) – E-Field
  - Couples into CSX, ARI and INF programs.
- Energy Materials STXM
  - Battery / Catalyst through low energy STXM
  - Soft Matter
  - Sample Cells
- INSPIRE (QIX/AP-XPS)
  - Catalyst research by combining XPS, IRRAS and RIXS
  - Can operate at high >100 Torr pressure to address the pressure gap.
- INF
  - Near field IR imaging with ideal resolution for non-heterogeneity in Quantum Materials and energy relevant materials (catalysts).
  - Serves as IR delivery to INSPIRE when needed.

ARI – A Nano-ARPES/RIXS Facility for NSLS-II

A. Walter, P. D. Johnson, S. Wilkins

September 11, 2018

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Rationale for the Proposed New Beamline

- **What is proposed:** A high flux, nano-focused beamline is proposed to provide unprecedented nano-scale ARPES & RIXS spectro-microscopies.
- **Science case, including national and/or global context:**
  - A spatial resolution of 100 nm approaches the characteristic electronic correlation lengths. Therefore it will be invaluable in studying the interplay between spin, charge and lattice degrees of freedom which lead to quantum electronic phase instability.
  - All current and planned nano-ARPES beamlines worldwide with energy ranges ~40 -1000eV energy range will have on-sample fluxes 3-4 orders of magnitude less than ARI majorly limiting momentum and real-space mapping.
  - No nano-RIXS beamline currently exists or is in planning, and the combination of ARPES and RIXS has also not been proposed to the best of our knowledge. The low emittance, high flux and high stability of NSLS-II makes ARI technically possible, and so it will be a unique capability to NSLS-II.
- **User interests and demand:** The large number of high profile co-proposers on the beamline proposal, as well as the high score given in 2015 by the SAC and future beamline review panel, indicate clearly the user interest and demand for this beamline and endstation.

Rationale for the Proposed New Beamline

- **NSLS-II portfolio impact:**
  - The ARI beamline will build off of the existing ARPES and RIXS capability at NSLS-II. However it will go far further than any existing or planned beamline by using mirrors to focus. Zone-plate focusing limitations of other planned nano-ARPES beamlines also prevents photon energy scans and effective real-space scanning.
  - The ARI beamline lends itself well to the 'multi-modal' direction of research at NSLS-II. By definition it is a multi-modal beamline (combining ARPES and RIXS). As these two primary techniques require an electron and a soft X-ray detector respectively, careful design of the endstation would allow for XPS, X-ray photoelectron diffraction and X-ray absorption 'spectroscopy' with no design change; X-ray diffraction, X-ray transmission and X-ray reflection 'measurements' with minor design changes (but no appreciable cost increase).
- **Scope, performance, and feasibility:** Detailed reports on the scope, performance and feasibility have been provided previously. Recent NSLS-II funded R&D has shown that all aspects of the design are challenging but feasible and that the performance parameters outlined previously can be met.

Infrared Near-FIeld Nanospectroscopy for Interface Technology and Emergence (INFINITE)

Mengkun Liu (*Stony Brook*), Chris Homes (*BNL/CMPMSD*), Dimitri Basov (*Columbia*), Dario Stacchiola (*BNL/CFN*), Sam Tenney (*BNL/CFN*), Jose Rodriguez (*BNL/Chem*), J.A. Boscoboinik (*BNL/CFN*), Ira Waluyo (*BNL/NSLS-II*), **Larry Carr (*BNL/NSLS-II*)**

September 11, 2018

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Rationale for the Proposed New Beamline

- **What is being proposed:** A suite of **THREE** independently operating infrared beamlines for 1) cryo-nanospectroscopy of heterogeneity and emergence in complex quantum mat'ls (5K to 400K), 2) vibrational nanospectroscopy of catalytic nanostructures, 3) microspectroscopy & surface vibrational spectroscopy, with each beamline spanning the 2 to 125 mm wavelength range (10 meV to 0.6 eV). *Note:* INF provides the IR for **INSPIRE** (sep. 2-slider)

- **Science case, including national and/or global context:** IR spectroscopy directly senses both collective and localized excitations associated with electrons and atomic/molecular bonding -- quantum transport, phonons, plasmons, magnons, CDWs, SDWs, SC gaps, etc. Nanospectroscopy couples to some $q \neq 0$ excitations.

- **User interests and demand:** IR nanospectroscopy is a new method with a growing community. The ALS has recently commissioned a 2nd beamline (2.4) for this technique to accommodate user needs. The mid-IR microspectroscopy beamlines at NSLS were typically over-subscribed.

Rationale for the Proposed New Beamline

- **NSLS-II portfolio impact:** INF provides a suite of three beamlines reaching 10 meV energies, two for achieving 10 nm spatial resolution. This will be unique to NSLS-II while also complementing x-ray probes capable of sensing at the nanoscale (e.g. nano XAS, nano ARPES). One beamline provides IR microspectroscopy, serving a large and diverse science community, thus filling a major gap in the NSLS-II portfolio. INSPIRE adds surface vib. spectroscopy.

- **Scope, performance, and feasibility:**
  - These are full/complete beamlines from source through endstations. A single extraction of IR from a standard dipole serves as source for all three INF beamlines. Newly developed cryo+near field will provide unique sample environments.
  - The combination of high beam current (500ma) and very high stability make NSLS-II an ideal, and likely world-leading, facility for nano and microspectroscopy.
  - The technology for nanospectroscopy (apertureless scattering from an AFM type tip), though recently developed for synchrotron infrared, has been successfully demonstrated at ALS and a few other facilities around the world.

SMF: Soft X-ray Spectro-Microscopy Facility

K. Kaznatcheev, J.Thieme, Y. Chu, S. Wilkins

September 11, 2018

Rationale for the Proposed New Beamline

- **What is being proposed**:
  - Soft X-ray scanning microscope (STXM) as analytical instrument and full-field x-ray microscope (TXM) for very fast imaging in 2D and 3D on an ID-based high-flux, moderate energy resolving power (~3000) beamline with a wide energy range (250eV-3.5keV).
  - A combined low-energy TEM and STXM capability in a single experimental station, to be built at the downstream end of the ARI beamline (50 eV-1.5 keV).

- **Science case, including national and/or global context**:
  - Nano-imaging for geo- and planetary sciences, and bio- and medical sciences
  - Compositional analysis and functional performance of soft matter compounds under operando conditions (aqueous environment, holder for cryogenic specimens)
  - Chemical imaging of working catalysts
  - Holographic imaging of spin textures in quantum materials

- **User interests and demands**

Extensive user demand in a wide range of scientific areas. Existing instruments in other facilities are heavily oversubscribed. Geographical argument: No instrument available on the East Coast, although many user groups based here.

Rationale for the Proposed New Beamline

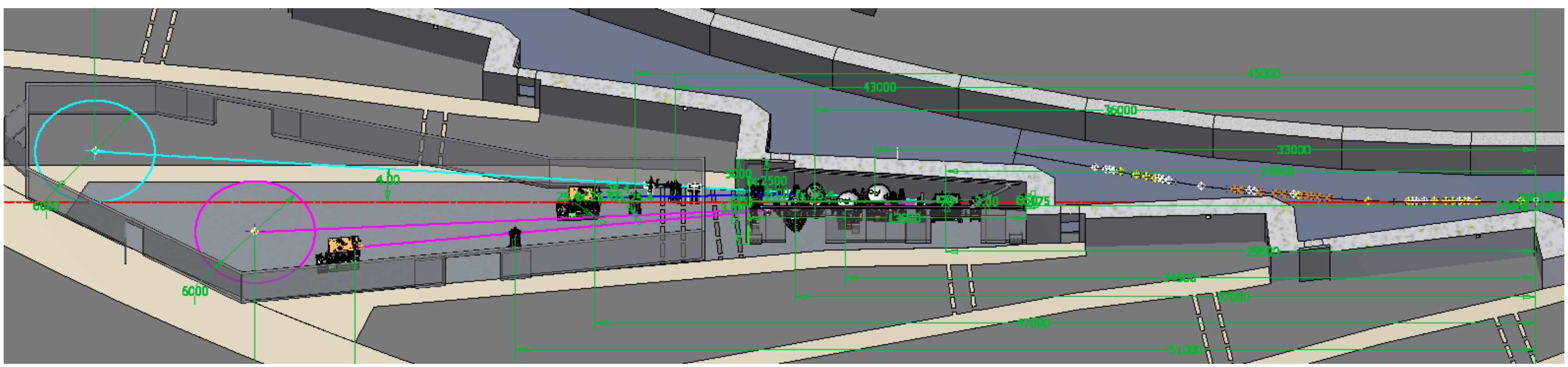
- **NSLS-II portfolio impact:**

SMF will provide a seamless transition with SRX, closing the availability gap for low-Z elements of X-ray spectromicroscopy with spatial resolutions of 100 nm or less. HXN offers higher resolution but no access to low-Z elements. Multi-modal imaging measurements requiring the study of e.g. transition metals and organic materials will be possible within NSLS-II.

- **Scope, performance, and feasibility:**

Two undulators in canted geometry, each serving one branch:

  - Higher-energy branch: high-E STXM (day 1), full-field TXM (later)
  - Lower-energy branch (ARI): low-E STXM in combination with TEM (later)



Catalysis & Growth Village (CGV)
Structure Identification Along the Reaction Path:

Klaus Attenkofer, Steve Ehrlich, Eli Stavitski, Oleg Tchoubar

September 11, 2018

Rationale for the Proposed New Beamline

- Concise one-sentence on what is being proposed

A combination of high throughput **soft** and **tender** X-ray Absorption (focused on in-situ/operando) stations, a set off 3 (or more) ambient pressure XPS chambers (including High Energy XPS) dedicated to "dirty chemistry", and a soft X-ray resonant coherent scattering chamber dedicated to operando and in-situ experiments; this suit of instruments can be realized in

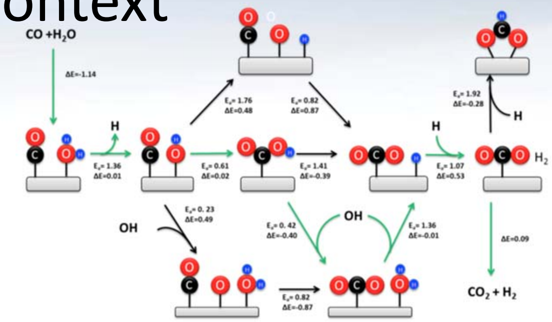
> a **Full Sector** using the **bending magnet port** (with optimized 3PW/5PW) for high throughput absorption and some of the ambient pressure XPS systems. These instruments will work with moderate focusing (~50-100μm) and cover the soft X-ray part up to 2500eV. The **ID port** is instrumented with a **canted tender and soft X-ray device with full polarization control**. The soft x-ray branch is providing 10μm beam size and is optimized for coherence; it hosts the soft X-ray resonant coherent scattering chamber and at least one specialized ambient pressure XPS chamber. The tender line is hosting an absorption chamber and will have an extension option for a second specialized ambient pressure XPS chamber. This option may also host other instruments like the INSPIRE instrument.

or

> A **combination** of a **new bending magnet port** and **integration of new instruments at existing beamlines**. One new **bending magnet port** identical to the described beamline above. The soft X-ray line could be combine with one of the CSX/IOS branches. The tender line may be integrated in one of the two tender X-ray lines (in both cases there may be significant impact on the existing programs).

Rationale for the Proposed New Beamline

- Science case, including national and/or global context



One of the great challenges in catalysis, and in materials growth (using precursor materials) is the structure development of the reactant and the catalyst during the reaction pathway. Visualizing and finally understanding (quantitative quantum chemical calculation) these structure changes and their impact on the reaction pathway are the key to rational designed catalytic materials and complex materials structures. Four steps are necessary to achieve this understanding:

1. Identify number and energetic conditions of intermediates along the reaction path; this is typically done using kinetic experiments. (Done by the user)

2. Identifying the intermediates in the spectroscopic response; fast data acquisition and a large data set is essential to apply statistical analysis methods which allow not only to identify the various spectral contributions but also to quantify its contribution to the signal.

3. Identifying structures which are consistent with the observed spectroscopic features using quantum chemical calculations. This step will not only result in structure hypothesis but also can separate between “active=necessary for the reaction” and “parasitic=without or with negative impact on conversion efficiency” changes in the catalyst.

4. Verifying structures with an imaging tool

Combining the proposed tools with the existing tools within the program will allow to characterize

- not only the catalyst but also the reactant;
- the XPS/HEXPS combination will provide additional depth sensitivity;
- the soft X-ray resonant coherent scattering will provide the final imaging tool.

Combining the proposed probes in concert with the existing probes of the program and with the computational and data analytics efforts at the laboratory will provide a new “answer oriented approach”.

Rationale for the Proposed New Beamline

- User interests and demands
  - Nearly the complete user community of ISS, QAS, IOS, and parts of BMM will be interested on the new toolset. In addition there is a strong connection to existing programs at chemistry division and at CFN with existing user communities.
  - We estimate about
    - 30 potential groups in the North-East area
    - Additional 50 potential groups nation wide
    - Additional 20 groups world-wide
    - Total: about 100 groups
  - The combination of low-risk and high risk experiments will guarantee high productivity (measured by number of publications) as well as high impact.

Rationale for the Proposed New Beamline

- NSLS-II portfolio impact
  - Complements the existing hard X-ray high throughput capabilities for in-situ and operando experiments.
  - builds on data analysis and quantum chemical computation efforts
  - builds on HEXPS expertise at NIST
  - builds on ambient pressure expertise at IOS, chemistry division, and CFN
  - complements the BCDI, and RSOX activities

  <u>Makes NSLS-II to a synchrotron catalysis center</u>

- Scope, performance, and feasibility, including implementation as a new endstation or as a new branchline on an existing beamline
  - will require sample environment developments (building on existing efforts)
  - low risk for high throughput efforts
  - soft X-ray resonant coherent scattering has to be demonstrated and established as tool for catalytic research

Coherent *In-Situ* and Operando Studies of Materials (CSO)

<u>Headrick</u>, Nelson, Evans-Lutterodt, Dooryhee
+ co-proposers from NC, Yale, Johns Hopkins, Boston, San Diego

September 11, 2018

Rationale for the Proposed CSO Beamline

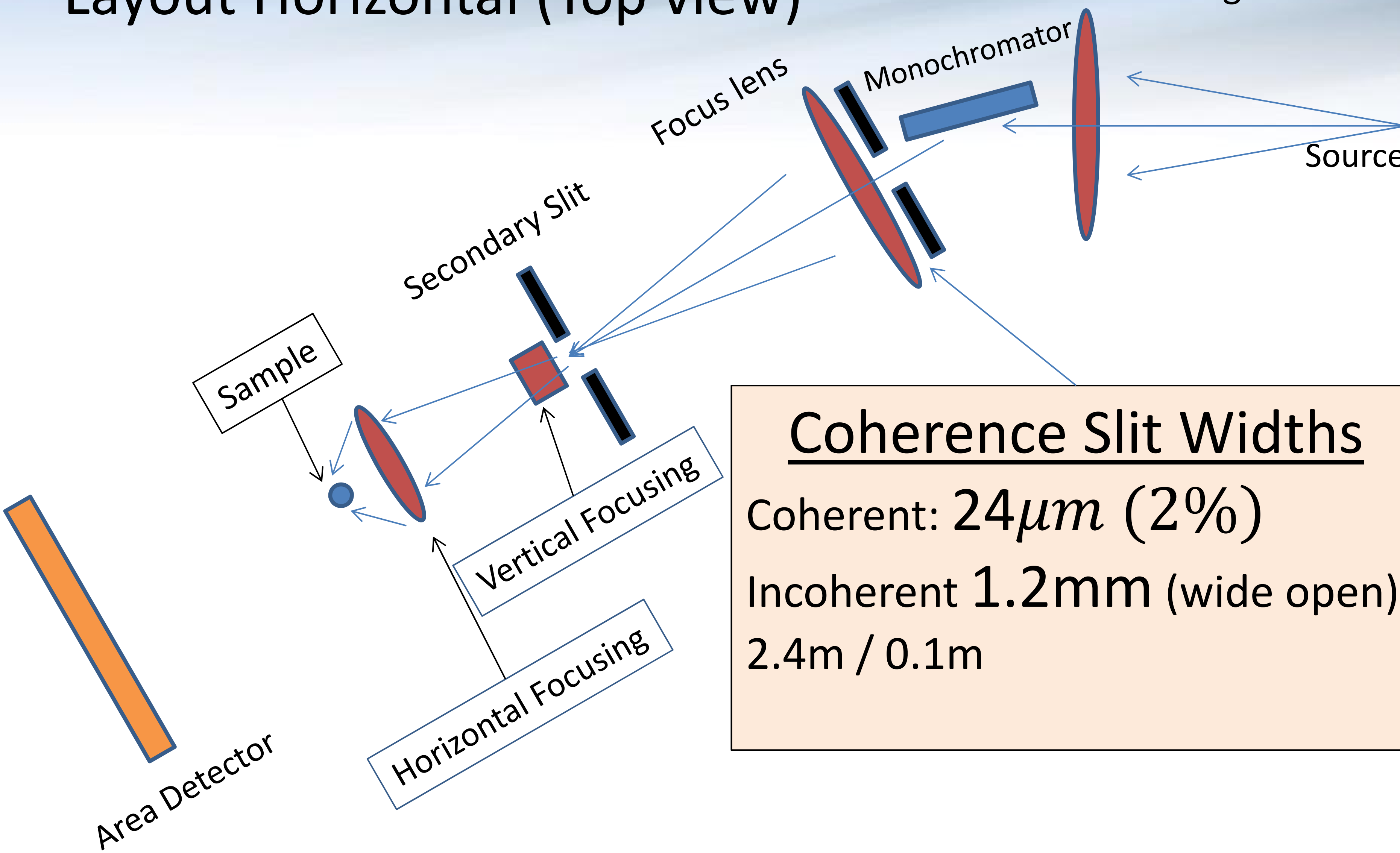
- **What is being proposed**: Coherent, in-situ studies of growing thin-films and nano-materials and material synthesis, enabling Co-GISAXS, XPCS, and imaging techniques (coherent phase contrast, coherent diffraction and ptychography).
- **Science case**: Many new functional materials exist as monolayers on substrates (Graphene, WSe2) or as nano-particle/substrate combinations that are best studied in situ, or operando.
- **User interests and demands**: In-situ studies of the dynamics of growing thin films with <u>incoherent</u> diffraction have been demonstrated at ISR (4-ID). The feasibility of <u>coherent</u> studies of growing thin films have also been shown at CHX (11-ID). Extending to coherent studies enables new perspectives on the growth processes. On both beamlines, the demand exceeds supply.

Rationale for the Proposed CSO Beamline

- **NSLS-II portfolio impact**: Currently existing coherent hard X-ray beamlines such as APS 8-ID and NSLS-II CHX (11-ID) are not well equipped to support relatively bulky in-situ chambers, and so the lack of such a facility currently limits scientific progress in the area.

- **Scope, performance, and feasibility**: Many accommodations during the design and construction of ISR were made to enable a side branch beamline with a canted second undulator. Fixed energy of 15 keV, 1 micron spot size.

Layout Horizontal (Top view)



Coherence Slit Widths

Coherent: $24\mu m$ (2%)

Incoherent 1.2mm (wide open)

2.4m / 0.1m

Fixed Energy MX

Lonny Berman, Sean McSweeney, Robert Sweet

September 11, 2018

Rationale for the Proposed New Beamlines

- **What is being proposed:** Two fixed-wavelength beamlines for MX viewing an undulator source, leaving space in the sector to accommodate a second (canted) undulator and tunable beamline that can be built later.
- **Science case, including national and/or global context:**
  - Several synchrotron sources are planning to shut down shortly in order to implement major upgrades: CHESS in 2018-2019 (already started), ESRF in 2019-2020, APS in 2022-2023, ALS in 2024-2025. This will severely impact their MX user communities.
- **User interests and demand:**
  - An opportunity exists to deliver substantially higher MX beamline capacity than currently exists at NSLS-II. With the proposed beamlines, NSLS-II could support ~500,000 samples/yr and attract new communities including those displaced elsewhere.
  - There is good reason to believe that the additional user communities to be accommodated through these beamlines would become permanent additions to the NSLS-II community.

Rationale for the Proposed New Beamlines

- **NSLS-II portfolio impact:** The beamlines will be ideal for MX experiments for which wavelength tunability isn't vital – reducing load on AMX, FMX, and NYX, allowing them to focus on their unique capabilities.
- **Scope, performance, and feasibility:**
  - We propose to employ instrumentation proven at both APS and ESRF. The cost is relatively inexpensive and will result in beamlines ~20 times more intense than existing examples of this design, because of the NSLS-II source. The two beamlines can be constructed in 3 years owing to their simplicity.
  - The beamlines will be fixed wavelength (~0.98 Å) viewing an undulator source, based on a diamond monochromator (Laue geometry) and CRL focusing system for each, with automated MX endstations. The anticipated flux is ~5×10^{12} ph/sec, with a ~25 µm beam focus.

INSPIRE (INelastic Scattering, Photoemission, and InfraRed Endstation): A New Endstation and Optics Upgrades for the IOS (23-ID-2) Beamline

I. Waluyo, I. Jarrige, A. Hunt, J. Dvorak

September 11, 2018

U.S. DEPARTMENT OF ENERGY | Office of Science

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Rationale for the Proposed New Beamline

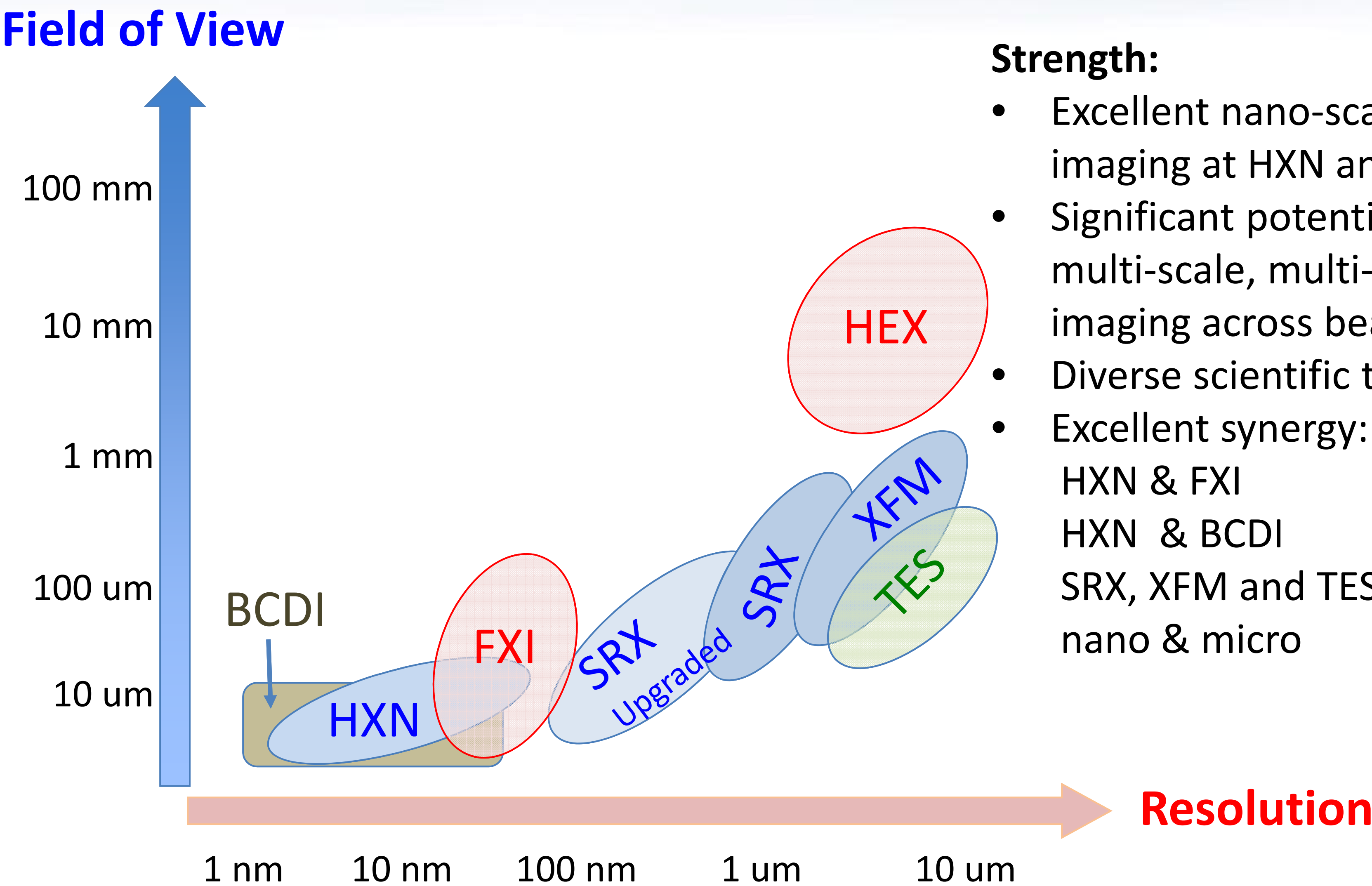
- **What is being proposed:** INSPIRE is a new endstation and optics upgrades to the IOS (23-ID-2) beamline that will enable ambient pressure XPS, XAS, IR, and XES/RIXS experiments on the same system under the same close-to-realistic operating conditions, at chemically relevant time scales.
- **Science case:** INSPIRE will provide a much-needed operando capability for real-time, multi-modal measurements of the electronic structure with a unique combination of complementary probes to serve the fields of:
  - **Catalysis:** Reveal the kinetics of catalytic reactions, derive a cohesive understanding of the structure-function relationship of the catalyst.
  - **Materials Science**: Track changes in the electronic structure and the surface composition of fuel cells and photovoltaics under operating conditions.
  - **Batteries:** Unravel multiscale processes and dynamics under working conditions.
- **User interests and demands:** IOS already has a broad user base from the heterogeneous catalysis (~65%), materials science (~25%) and battery (~10%) communities. The existing techniques at the beamline, AP-XPS and XAS, are already in high demand with an over-subscription rate close to 3. XES/RIXS is in high demand at the new operando facility at the ALS.

Rationale for the Proposed New Beamline

- **NSLS-II portfolio impact:** Real-time measurements of the chemical state and electronic structure of energy systems in realistic conditions using a combination of soft X-ray and IR spectroscopy will be an indispensable addition to the NSLS-II portfolio. This unique capability will provide sensitivity to surface chemical states, chemical bonds, occupied valence states, and to the ligands, which is not currently available at NSLS-II.

- **Scope, performance, and feasibility:**
- INSPIRE evolved from QIX (Quick Inelastic X-ray Scattering) from the 2016 beamline development proposal process, which was proposed as a new endstation at the IOS beamline.
- Scope includes a refocusing KB mirror pair and an endstation with three experimental probes
- It is advantageous to combine QIX with AP-XPS in the same endstation because the tightly focused and high flux beam required for QIX is also necessary for increasing the operating pressure and time resolution of AP-XPS experiments.
- INSPIRE will also benefit from the future neighboring INF beamline to use synchrotron IR to perform simultaneous AP-XPS/IR and RIXS/IR experiments.
- Preliminary conceptual engineering design was done to show that it is feasible to have three probes in the same endstation at IOS.

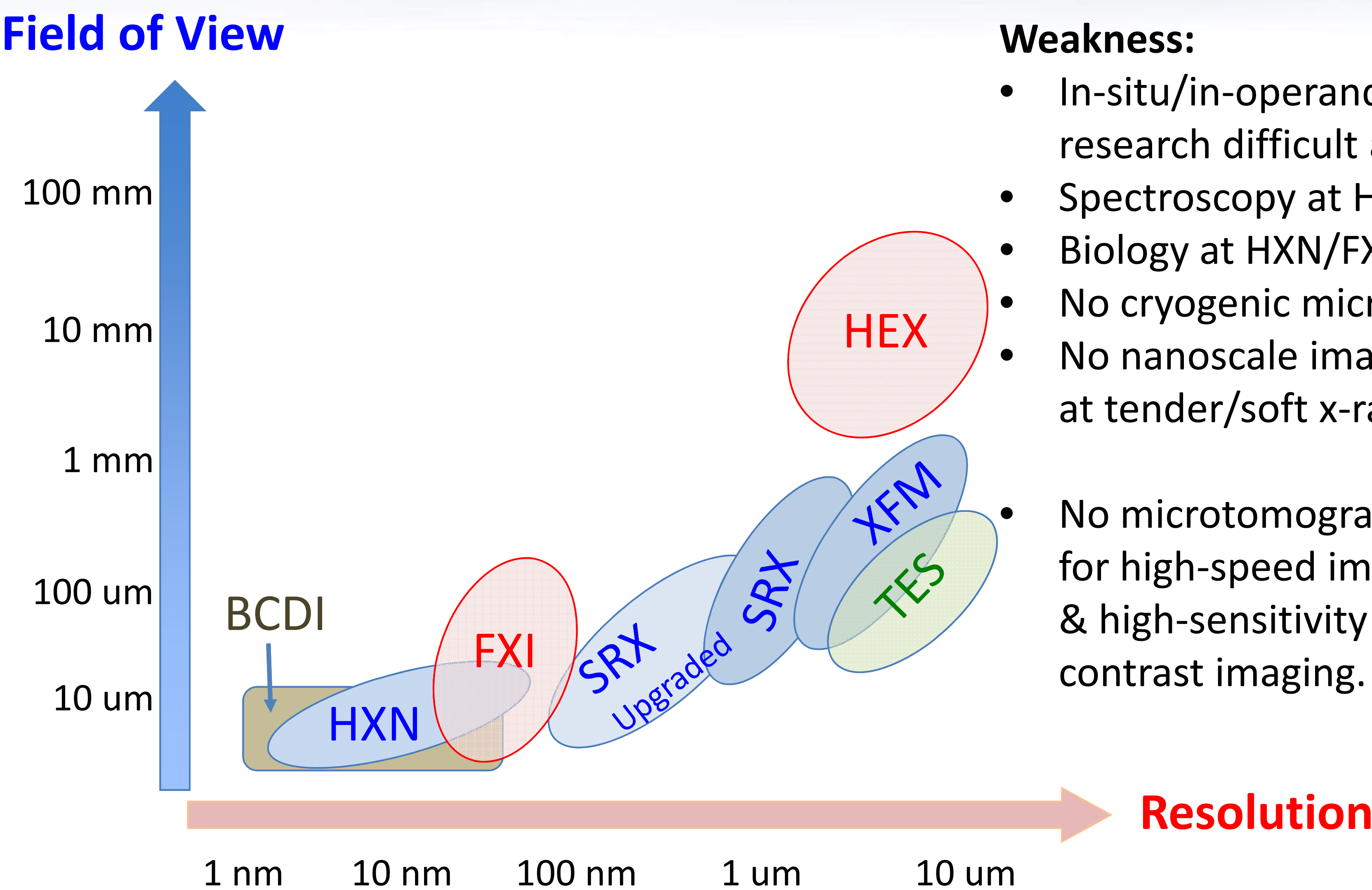
Current "Hard-X-ray" Imaging Capabilities at NSLS-II



Strength:

- Excellent nano-scale imaging at HXN and FXI
- Significant potentials for multi-scale, multi-modal imaging across beamlines
- Diverse scientific tools
- Excellent synergy:
  HXN & FXI
  HXN & BCDI
  SRX, XFM and TES
  nano & micro

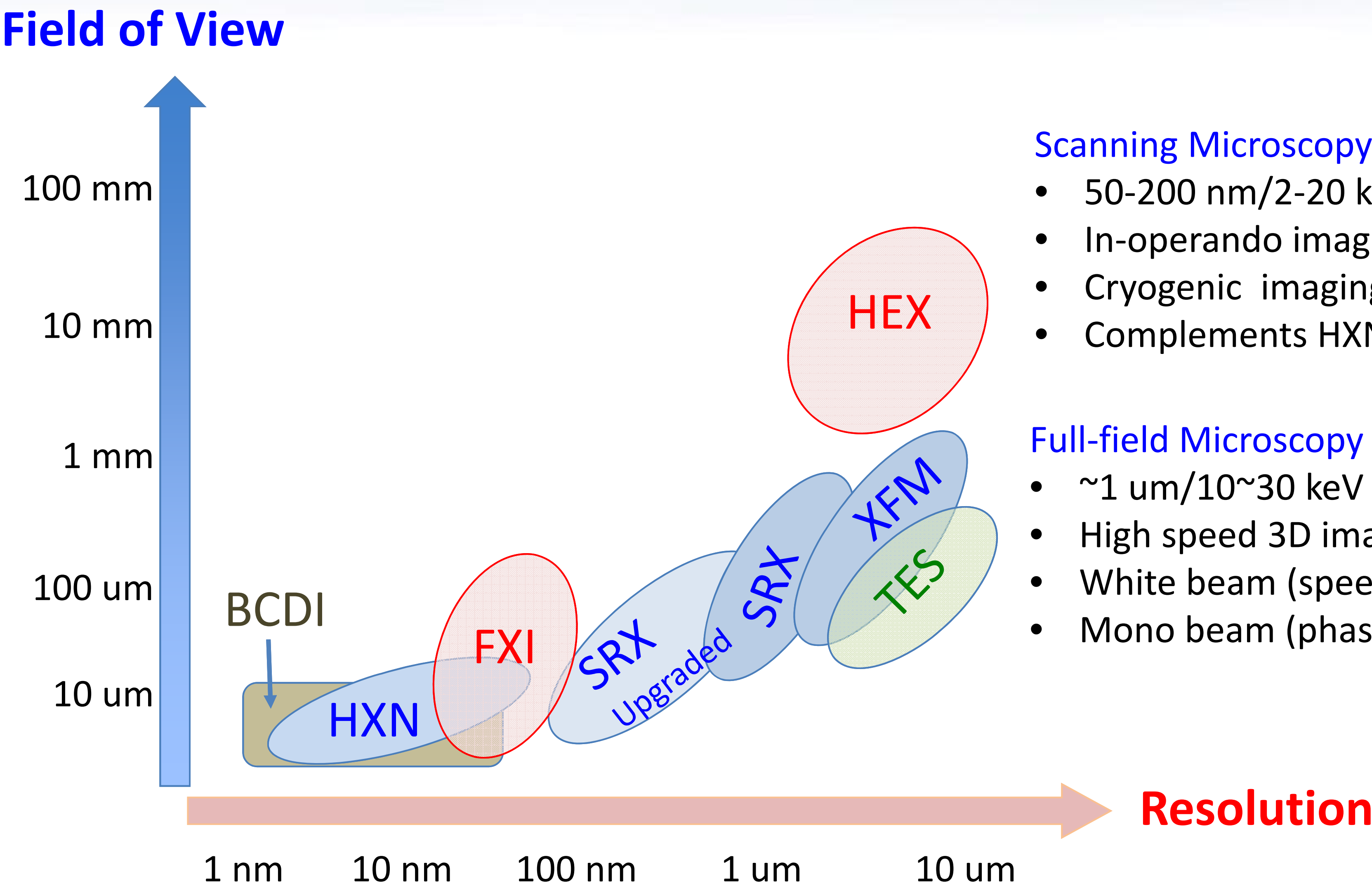
Current "Hard-X-ray" Imaging Capabilities at NSLS-II



Weakness:

- In-situ/in-operando research difficult at HXN
- Spectroscopy at HXN
- Biology at HXN/FXI
- No cryogenic microscope
- No nanoscale imaging at tender/soft x-ray regime
- No microtomography BL for high-speed imaging & high-sensitivity phase contrast imaging.

Gaps in the NSLS-II Imaging Capabilities



Scanning Microscopy

- 50-200 nm/2-20 keV
- In-operando imaging
- Cryogenic imaging
- Complements HXN/SRX

Full-field Microscopy

- ~1 um/10~30 keV
- High speed 3D imaging
- White beam (speed)
- Mono beam (phase-contrast)

In-situ and Operando Nanoprobe (ION) / Cryo X-ray Nanoprobe (CXN):

A story of fire and ice at one beamline

Hanfei Yan & RyanTappero

September 11, 2018

U.S. DEPARTMENT OF ENERGY | Office of Science

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Rationale for the Proposed New Beamline

- Concise one-sentence on what is being proposed

Station I: A nanoprobe with sub-100 nm resolution optimized for in-situ/operando microscopy imaging and bridging the gap between SRX and HXN

Station II: A tender-ish X-ray nanoprobe with full cryogenic capabilities and low background fluorescence detection is proposed for the bio-geo-enviro sciences user community.

- Science case, including national and/or global context

Imaging elemental and structural variations in battery materials in 3D to understand electrochemical reaction; Imaging evolution of the corrosion process in materials; Understand the phase change of functional materials as the temperature changes

Interrogations of the abundance, localization and/or chemical state of trace elements and nutrients (e.g., P and S) in delicate samples (e.g., tissues, cells) and natural specimen must be made in the frozen-hydrated state. Few nanoprobe beamlines in the world can do it.

- User interests and demands

Strong demand for in-situ/operando microscopy research with sub-100 nm resolution in a broad range of scientific areas

Biological & Environmental Sciences; Plant & Soil Sciences; Ecosystem Science; Diverse applications needing 50-200 nm.

Rationale for the Proposed New Beamline

- NSLS-II portfolio impact

*Fill the resolution gap
*Increase synergy for in-situ/operando research across diffraction, spectroscopy and imaging programs at NSLS-II

*Adds nano-scale tender energy capabilities to compliment TES
*Adds Cryo-XRF microscopy and ptychography capability to facility
*Fills gap in resolution/ field-of-view between HXN and SRX

- Scope, performance, and feasibility, including implementation as a new endstation or as a new branchline on an existing beamline

*Build a bonded-MLL based scanning microscope that delivers 30-100 nm resolution in the energy range of 10 – 20 keV.
*In-house developed MLL endstation reduces cost

*Build a ZP-based scanning microscope that delivers 50-200 nm resolution (2.1 – 12 keV)
*Turn-key endstation reduces risk and engineering costs
*DOE-BER likely to fund endstation

Shared resources:
ION & CXN will share IVU source and Photon Delivery System (parasitic, reduces OP cost!)
ION & CXN will utilize existing infrastructure at sector 5 (reduces project cost and risk!)

Microtomography Beamline

Xianghui Xiao & Wah-Keat Lee

September 11, 2018

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Rationale for the Proposed New Beamline

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A hard x-ray full-field micro-imaging beamline focusing on real time characterizations of real-size specimens under real conditions.

- Science case, including national and/or global context

While components of many functional systems are at the nanometer scales, the systems' performances/behaviors could be very complex at macro scales. Besides, it is necessary to require sample in the 1-20 mm scale to be statistically meaningful in the investigations of heterogeneous materials, e.g., rocks and cement.

Huge number of functional systems are in the 1-20 mm scale – e.g. seeds, insects, batteries, shales, soils and rocks, etc.

Micro-imaging can be used to characterize materials' fatigue and mechanical failure; particle distribution, porosity, tortuosity and connectivity; rock-fluid interaction under high T/P conditions; small animal physiology and growth, etc.

All major synchrotrons have similar beamlines that are highly productive.

Rationale for the Proposed New Beamline

- User interests and demands

Most users of nano-resolution techniques have a need for this at system level.

Demand is well documented by success at all such beamlines worldwide.

Broad user community: Material Engineering/Science; Energy Storage Materials; Geosciences; Biomedical, Paleontology; Industrial Applications, etc.

- NSLS-II portfolio impact

Fills a crucial capability gap in NSLS-II imaging portfolio.

Complements our high resolution techniques (FXI, HXN, SRX, FXM). Same user community as high resolution techniques.

- Scope, performance, and feasibility, including implementation as a new endstation or as a new branchline on an existing beamline

An optimized 3PW will provide a world-class source. Instrumentation is well-known and easy to implement. Shares identical analysis software as FXI.

Experiment Development Program: Source, Optics & Detectors – beamline needs

- ‘Test’ beamlines have played a huge role in the development of synchrotron science
  - undulators, high heat load optics: early work at CHESS and NSLS
  - phase contrast imaging and refractive lenses: BM05 at ESRF
  - UHRES mono for Mossbauer, tunable x-ray polarizers, x-ray waveguides: NSLS

- Beamlines have evolved from ‘simple/open/flexible’ concepts at $1^{st}/2^{nd}$ generation synchrotrons to more ‘complicated/specialized/fixed’ concepts at $3^{rd}/4^{th}$ generation sources. White beam access is rare, as are places to test IDs and optical schemes.

- New and novel insertion devices will play a big role for future NSLS-II upgrade. Where will we test them?

- At wavelength metrology are essential for optics development for increasingly bright sources. Where will we do this?

- Timely detector development requires frequent access to beam – possibly for extended periods of testing. Where/how do we facilitate this?

- Where can we prototype new optical schemes?

- To lead in synchrotron science, we need a dedicated place to do the required testing.

Instrumentation Test Beamline

<u>Jeff Keister</u>, Mourad Idir, Wah-Keat Lee, D. Peter Siddons, Oleg Chubar, Toshi Tanabe, Yong Chu, Xianghui Xiao, Konstantine Kaznatcheev, Zhong Zhong, Lonny Berman, Joseph Dvorak

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Rationale for the Proposed New Beamline

Concise one-sentence on what is being proposed:

- A flexible beamline located on a straight section to serve as a test platform for IDs, optics, detectors, instrumentation and techniques.

Science case, including national and/or global context:

- Test beamlines have seeded many significant developments: refractive optics, phase contrast imaging, wavefront sensing, inelastic scattering instrumentation, UHRES monochromators, x-ray polarizers and mini-gap in-vacuum undulators
- This proposal provides “in-beam/at-wavelength” test capabilities to support development of novel state-of-the-art instrumentation for synchrotron science, uniquely positioned at NSLS-II straight section to support R&D (including IDs) for upgrade paths for NSLS-II and other light sources in the DOE complex
  - Enables R&D for optics, detectors, and insertion devices to exploit high brightness and coherence of NSLS-II
  - Open space concept with white beam delivery to the end enables prototyping of novel optical schemes and instrumentation

User interests and demands

- Optics: MLLs and refractive lenses, aspheric x-ray mirrors and associated mechanics, wavefront-preserving optics and sensors, heat load and adaptive cooling investigations
- Detectors: microbeam scanning to test uniformity & spatial resolution, rate dependence of detector parameters
- New beamline and experimental schemes: white beam allows testing of novel arrangements of optics and detectors, such as Guinier geometry for powder diffraction
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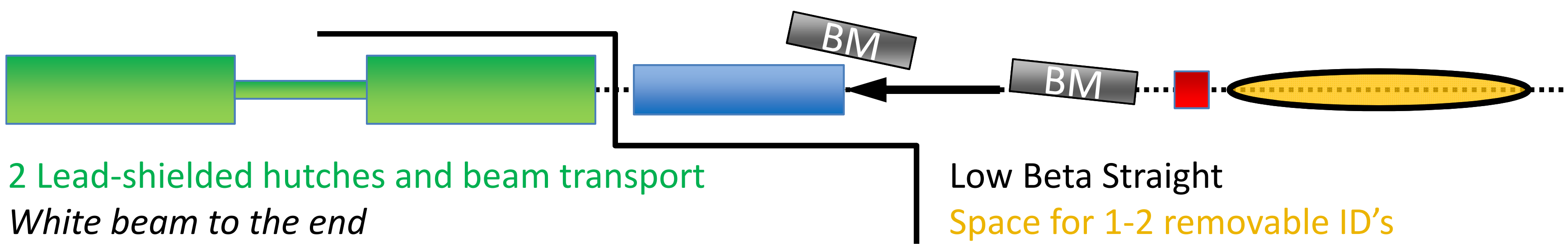
- Supports long-term collaborations on detector and other instrumentation development

Scope, performance, and feasibility, including implementation as a new endstation or as a new branchline on an existing beamline



2 Lead-shielded hutches and beam transport

White beam to the end

Movable components:

- *Monochromator*
- *Detector test station*
- *Focusing mirror*
- *4-circle goniometer*
- *Metrology test station*

Low Beta Straight

Space for 1-2 removable ID's

3-pole wiggler at downstream end

3PW and ID-compatible front end

Transfocator in the front end

Rationale to *consider* MicroCT-TEST marriage

- MicroCT will have high user demand and high number of publications. Balances TEST, whose metrics of success may be somewhat different.
- MicroCT gains from this tie-up because by itself, it is difficult to justify on a straight section. A regular 3PW spectrum is not hard enough and not good for phase contrast (due to source size). The tie-up enables a 3PW to be placed in a straight low beta section thus allowing for a lower gap and higher magnetic field – resulting in an optimum spectrum for MicroCT (absorption and phase).
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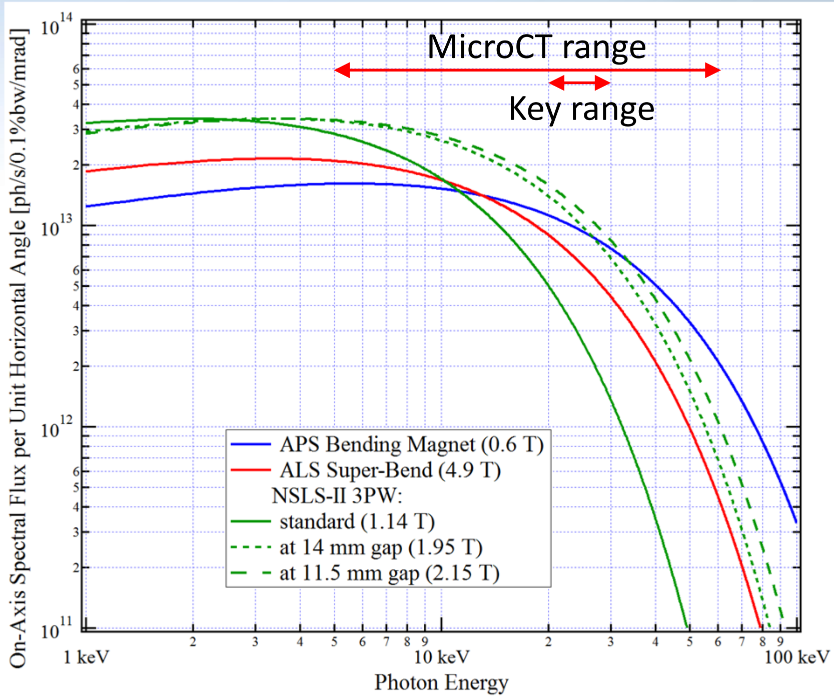

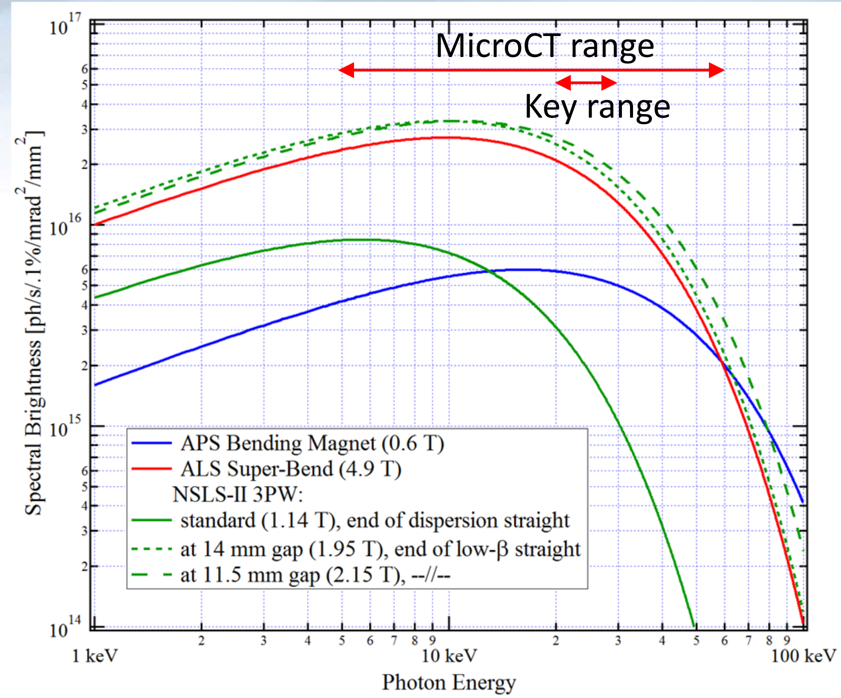

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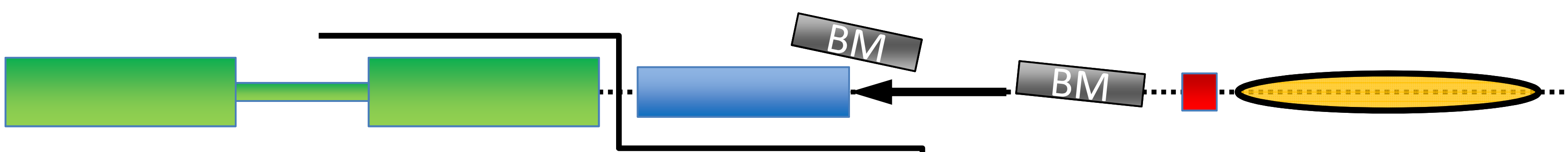

MicroCT set-up can be portable (sample & detector stages on a single tabletop)

Only other beamline components are filter box and a DMM (upstream)

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Microtomography Beamline

Xianghui Xiao & Wah-Keat Lee

September 11, 2018

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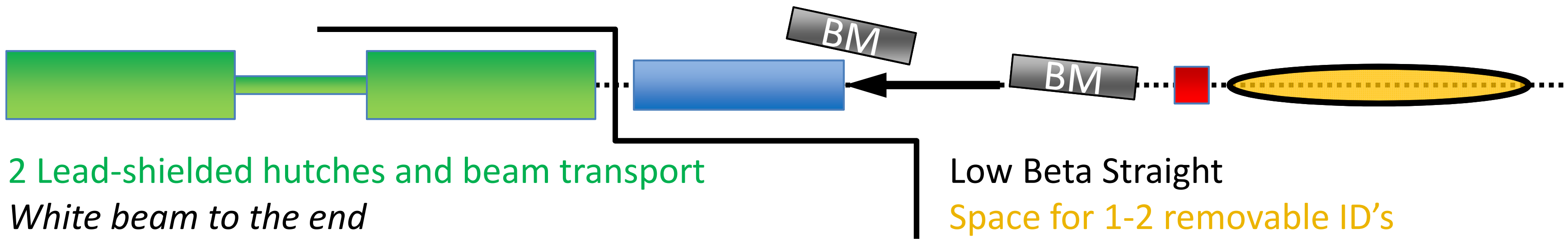

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3PW and ID-compatible front end

Transfocator in the front end

Merging MCT with MTB

Jeff Keister, Mourad Idir, Wah-Keat Lee, D. Peter Siddons, Oleg Chubar, Toshi Tanabe, Yong Chu, Xianghui Xiao, Konstantine Kaznatcheev, Zhong Zhong, Lonny Berman, Joseph Dvorak

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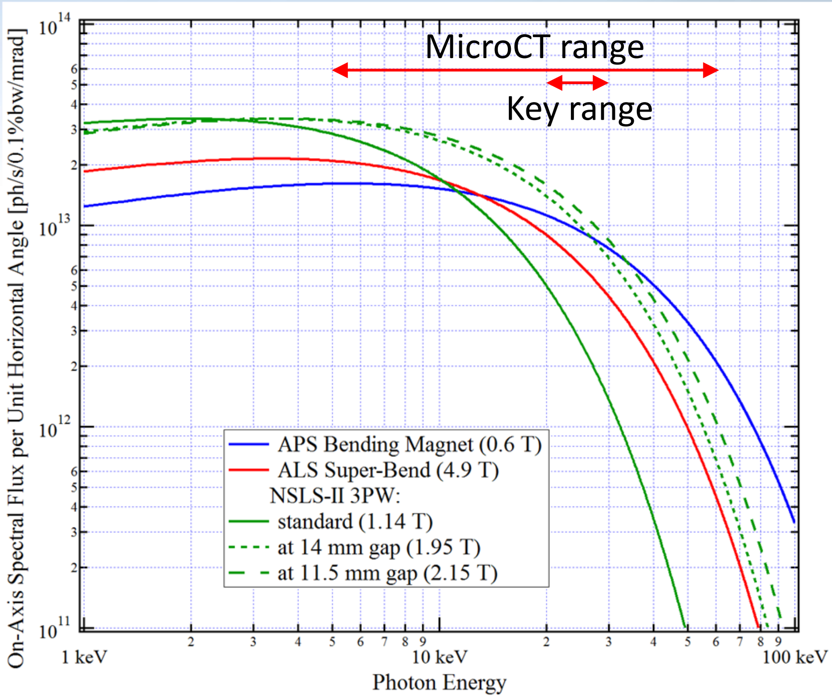

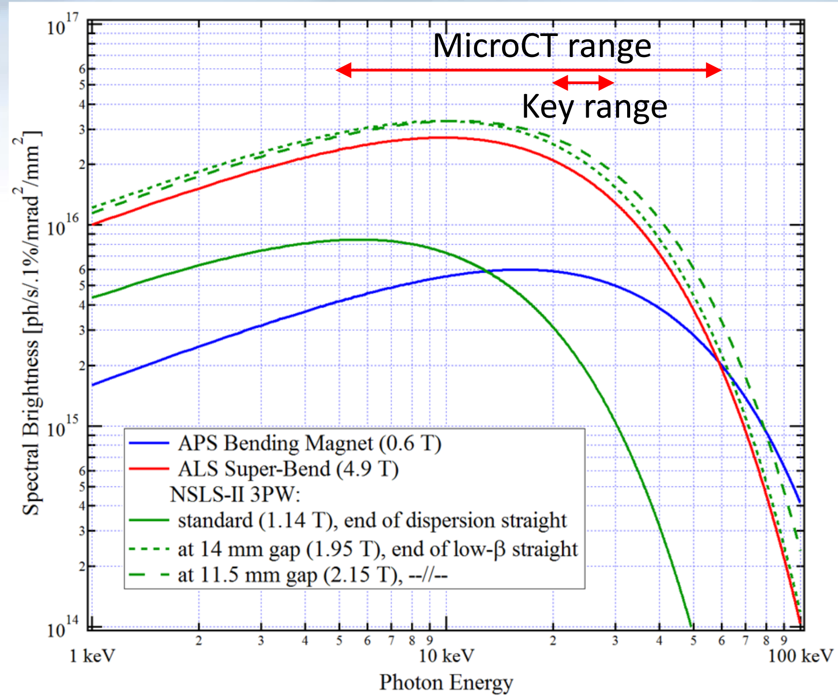

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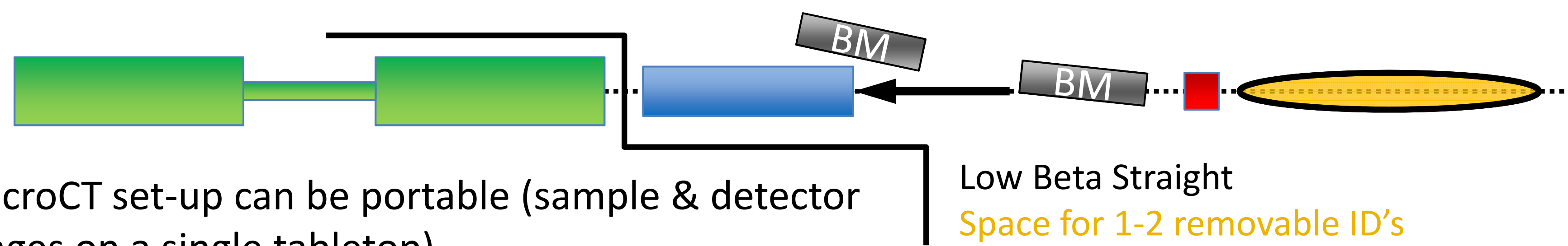

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3PW and ID-compatible front end
Transfocator in the front end

Multi-Dimensional Multi-Modal Diffraction (MMD)

K. Evans-Lutterodt, P. Stephens, E. Dooryhee
+ co-proposers Carnegie, Stanford, Wisconsin, Oak Ridge, IBM, Stony Brook

September 11, 2018

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Rationale for the Proposed MMD Beamline

- **What is being proposed:** strengthen NSLS-II diffraction capabilities to fill beam size gap and sample gap, enabling extended diffraction mapping and imaging that can sensitively probe particle-level atomic structures, defects and inhomogeneities that may dynamically change during synthesis reactions, quantum phase transitions, or device operation.
- **Science case:**
  - Sensitive resolution from meso to micron scales: domain patterning in ferroelectrics, films, interfaces, magnetic materials, shape memory alloys, ...
  - Phase inhomogeneities, strain/composition gradients during materials synthesis or processing (F, E, B, P, T, ...) *e.g*. batteries, catalysts, interconnects, ...
  - Electronically driven structural phase transitions / order parameters in quantum materials (charge density waves, orbital order, superconductivity, ...)
  - High Pressure, high/low temperature science with DACs (APS 11-ID-C)
- **User interests and demands:** (1) diffraction mapping with excellent spatial and temporal resolution and (2) high-rate & high-resolution in situ studies to sensitively probe changes in a sample

Rationale for the Proposed MMD Beamline

- **NSLS-II portfolio impact:**
  - Fill beam size gap (100-500nm range) between nanoprobe and bulk diffractometers (optimized micro-diffraction)
  - Enable practical working distance for in-situ diffraction and preserve resolution
  - First NSLS-II capability for space-resolved diffraction mapping with high flux for time-dependent (depth profiling, lateral mapping and tomography) studies
- **Scope, performance, and feasibility:**
  - undulator-based, canted (preferred) or 2-endstation NEW beamline.
  - Dedicated, small-beam diffraction / imaging probe at energies up to 25 keV with stable spot from 30 nm to 3 microns with large working distances with limited gonio capabilities. In-line focusing optics to change spot sizes without moving sample and choice of coherent and incoherent sample illumination.
  - Large beam (0.1 – 1 mm) without demagnification (with transfocator option) and full goniometry for high-angle resolution (detector/analyzer), high-rate (kHz) and energy-discriminating (Si/Ge strip) powder diffraction and for single crystal diffraction (*incl*. diffuse scattering and resonance)

Multimodal High Pressure Tools (MPT)

Donald Weidner, Pamela Burnley (Nevada), William Durham (MIT), Lars Ehm, Shun Karato (Yale), Andreas Kronenberg (Texas), Li Li, Matthew Whitaker and Wenlu Zhu (Maryland)

September 11, 2018

Rationale for the Proposed MPT Beamline

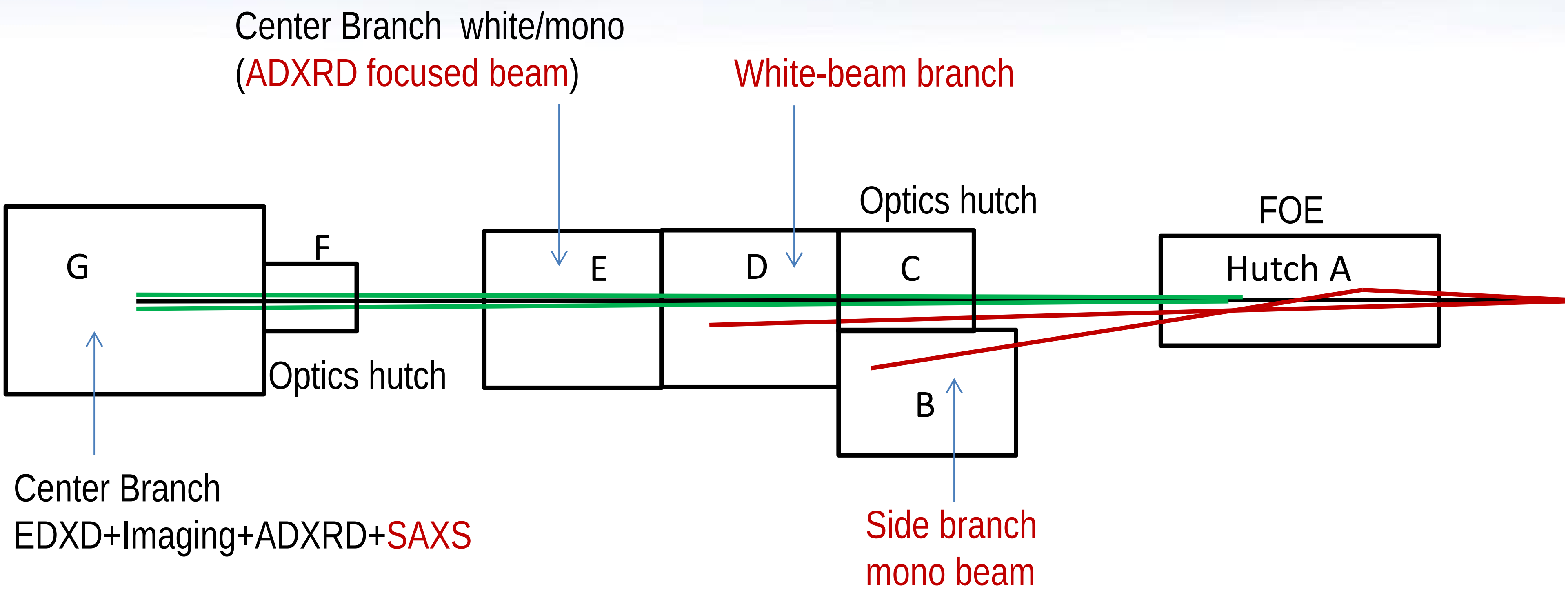
- **What is being proposed:** determine the 3D distribution of stress, and structure as a function of pressure, temperature, and time in order to model mechanical, thermodynamic, and kinetic properties. Samples are: multigrained, multiphased materials w/ and w/t the presence of fluids and melts.
- **Science case:** These properties are fundamental to
  - energy (*e.g*. fracking and carbon sequestration)
  - water in the subsurface (*e.g*. earthquake related to water injection/removal)
  - natural hazards (*e.g*. earthquakes, land failure)
  - Earth evolution (*e.g*. plate tectonics, deep earthquakes)
  - Materials synthesis (e.g. functionalized diamond, boron nitride or oxynitride photocatalysts), mechanical properties in crystalline and nano-crystalline materials, transitions even in the most basic materials, such as H_2, H_2O, and glasses.
- **User interests and demands:** Users from (1) mineral physics, (2) rock physics, and (3) engineering. Workshop: '*Envisioning the Next Generation of In-situ Synchrotron X-ray Techniques in Large-Volume HP Apparatus for Mineral and Rock Physics'* Sep. 28-30 to discuss such a beamline (>60 participants).

Rationale for the Proposed MPT Beamline

- **NSLS-II portfolio impact:** unique in the world: deformation studies on Earth minerals, ceramic, novel, and functional materials at higher pressures than anywhere else. Hardware will encompass a pressure range from 50MPa to 35GPa and temperatures up to 2,000K with deviatoric stress as an important state variable with multimodal (imaging and diffraction) x-ray analysis, bridging length scales (atoms to grains) and time scales seconds to days.

- **Scope, performance, and feasibility:**
  - The energy range: 20 – 100 keV with access to both mono beam and white beams
  - Spot size of 5 – 15 mm for full field imaging and diffraction, focusable to 0.1 mm in the vertical direction.
  - The (provisional) white beam endstation at HEX would serve most of the needs.

HEX Beamline Layout



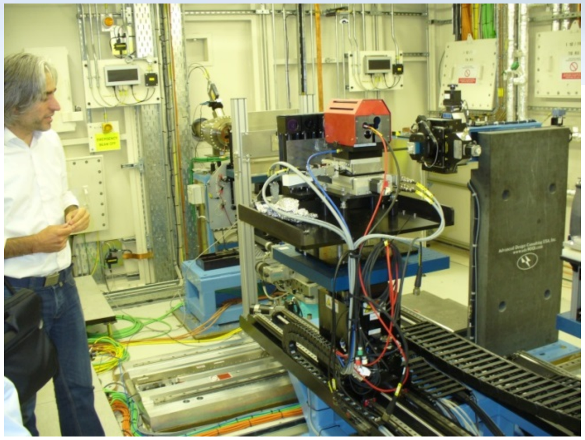
Center branch is in the scope

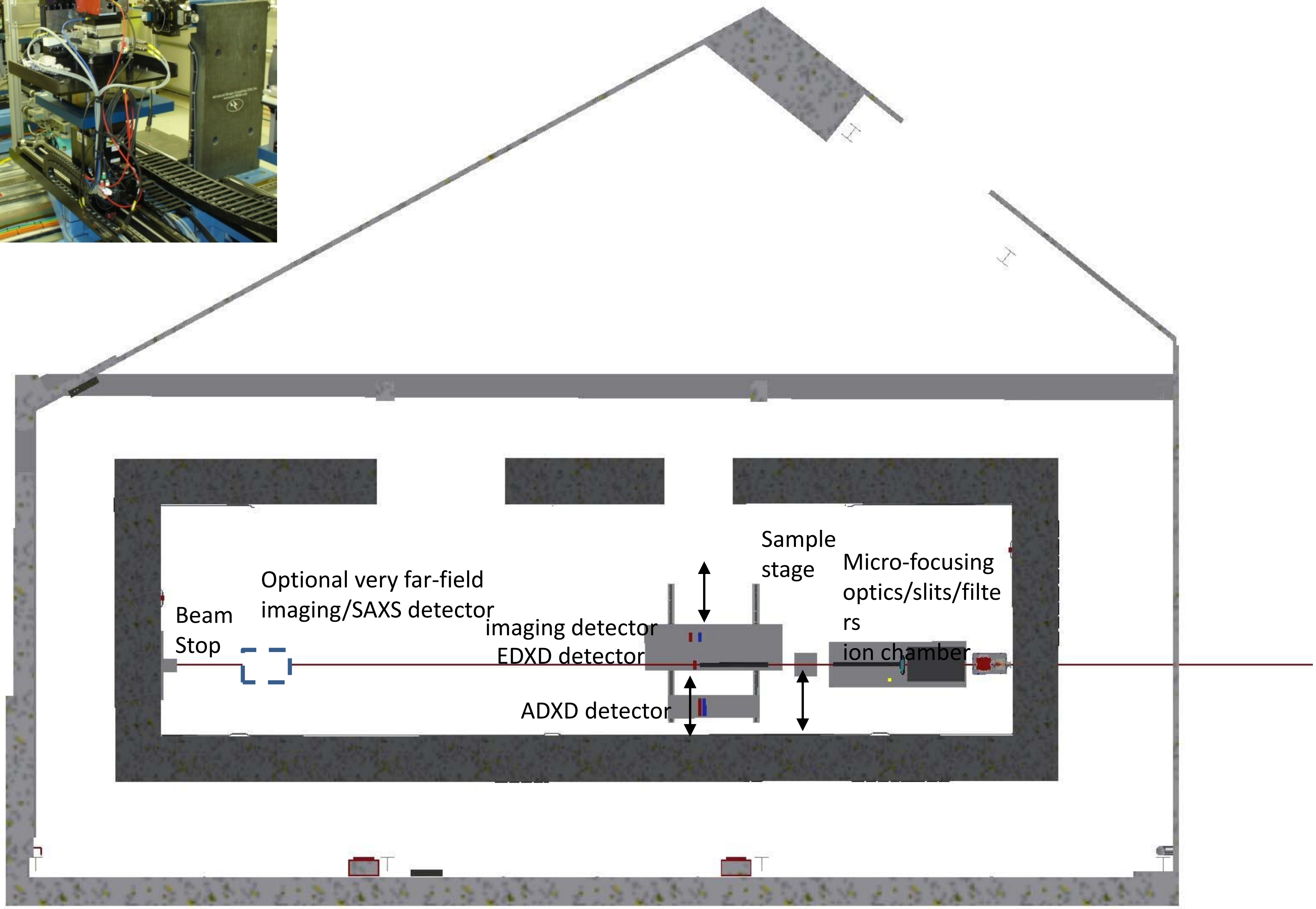
Optics and endstations for white beam (D) and side (B) branches are not in current scope

HEX Branches

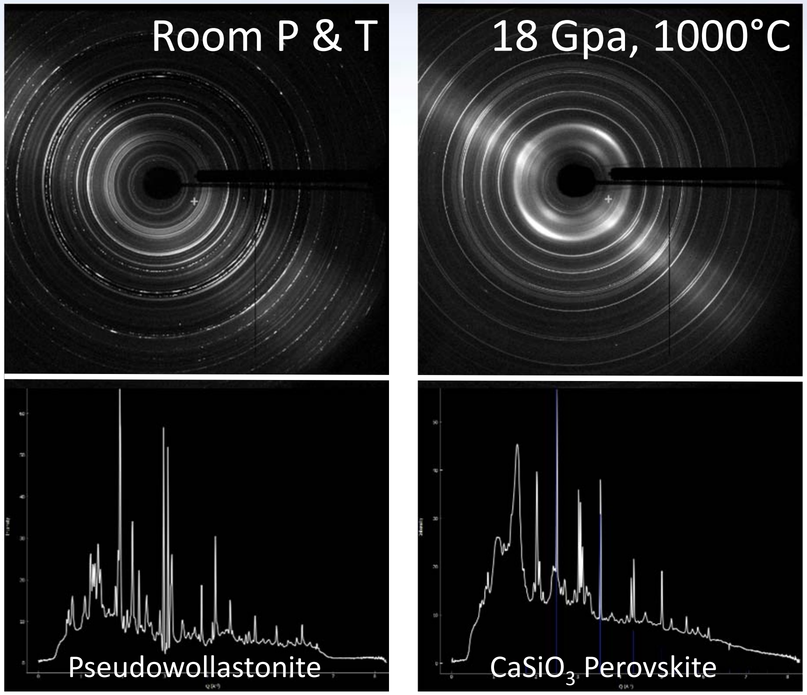
	Side branch	Center branch	White-beam branch
Technique	ADXD, PDF	EDRD, ADXRD, imaging, propagation-based phase contrast, small angle scattering, CT	EXRD
Beam size	0.5 mm x 0.5 mm	0.5 mm x 0.5 mm for diffraction 15 mm x 100 mm for imaging 10 microns x 10 microns for micro-focusing	5 mm x 5 mm
Flux/flux density (at 50 keV)	10^{12} ph/s for focused beam	10^{11} ph/s/mm^2 for imaging beam 10^{15}/ph/s/0.1%bw/mrad^2 for white beam 10^{13} ph/s for focused beam	10^{15}/ph/s/0.1%bw /mrad^2 for white beam
Resolution	0.5 mm	10 microns for EDXD 1 microns for Imaging 0.5 mm for ADXRD with unfocused beam 10 microns for ADXD with micro-focused beam	10 microns EDXD
Energy range (keV)	40-120	20-200 keV for white beam 30 – 150 keV for imaging and ADXD	20 – 200 keV white beam
Mode	Monochromatic	White/Monochromatic	White

Endstation F in External Building





Deformation of $CaSiO_3$ Perovskite Under Extreme Conditions



X-ray diffraction patterns of (left) starting sample material at ambient conditions, (right) sample at high pressure and temperature on the right. $CaSiO_3$ perovskite requires high P & T to form, and is an unquenchable phase. Once phase transformation was complete, the differential hydraulic system was engaged and sample was uniaxially deformed to ~15% strain in the DT-25 apparatus; the first of its kind in the world. These are the first measurements on the rheological properties of $CaSiO_3$ perovskite. -- (08/20/2018)

Scientific Achievement

Recently commissioned high pressure set-up at NSLS-II (XPD beamline) delivers first results.

Significance and Impact.

- The XPD beamline pressure rig is the first of its kind in the world capable of performing deformation studies on Earth, ceramic, novel, and functional materials at higher pressures than anywhere else.
- First study of the rheological properties of CaSiO3, an important phase found in the Earth's mantle.

Research Details

- COMPRES Partner User researchers from Stony Brook University developed, installed, and commissioned the high pressure multi-anvil endstation at beamline 28-ID-2-D at NSLS-II.
- this unique equipment is capable of achieving pressures >20 GPa and temperatures >2000°C. DT-25 can then be used to deform samples uniaxially at high T over ~2x pressure available in other systems.

Work was performed at Brookhaven National Laboratory by R.S. Triplett, D.J. Weidner, M.L. Whitaker, K.J. Baldwin, W.B. Huebsch, and M.T. Vaughan

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BROOKHAVEN NATIONAL LABORATORY

Processing and Liquid Scattering (PLS)

Ben Ocko, BNL

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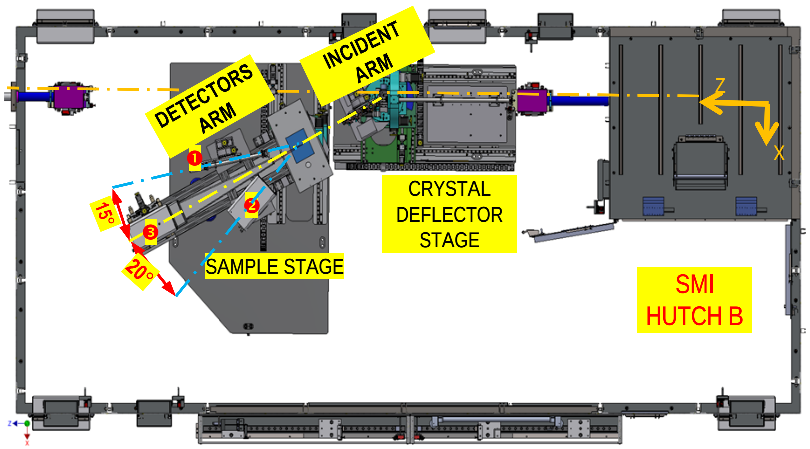
Rationale for the Processing and Liquids Beamline

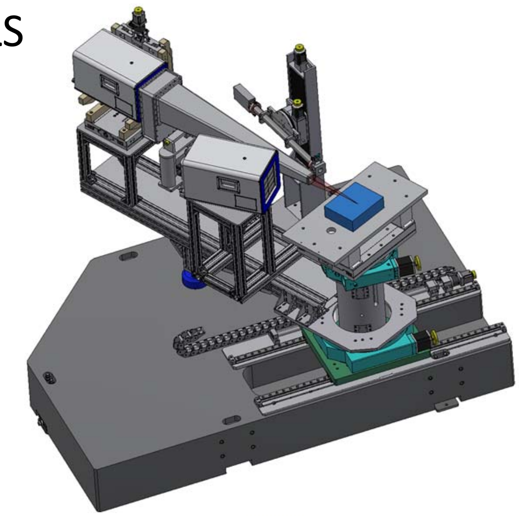
- **What is being proposed:** World-class beamline for studies of in-situ processing & liquid surfaces
- **Science case**:
  - The molecular and atomic structure of **liquid surfaces** (vapor and liquid/liquid) is important to chemistry, biology, physics, & materials sciences where specific systems of interest include Langmuir monolayers, molten salts, liquid metals, ionic liquids and electrochemical interfaces. Functional properties are dictated by surface structure.
  - During materials **processing**, functional properties of thin soft-matter and polymer films are determined by the intermediate phases that develop. Concurrent structural & optical measurements (ms time scales) are required to develop a comprehensive relationship between processing conditions & function.
- **User interests and demands:** High for both liquid surface and processing x-ray scattering facilities. There is only one operating liquid instrument in the US. European and US facilities much oversubscribed. Increasing demand for in-situ processing requires an open scattering environment with adequate space for complementary probes.
- **NSLS-II portfolio impact:** No current NSLS II facilities for liquid surfaces and only limited complementary capabilities for in-situ processing at CMS, a 3-Pole Wiggler source**.** PLS end station construction underway as a time-shared facility with SMI. Insufficient available beamtime for an extensive science portfolio for SMI and PLS in time shared mode.

Proposed Technical Scope

- **Optics:** Canted, nitrogen cooled undulator, 8-24keV, double crystal Si monochromator, focusing mirror optics (tanks already exist) for focusing at sample or secondary source, 5 µm vertical beam with CRL optics.
- **End Station**: Allows beam to be tilted downward with sample remaining horizontal. Existing hutch. Spectrometer (below) currently being developed (Jump-start J-PLS) as time shared facility with SMI, FY19 completion. PLS end station upgrades include (1) improved detectors. (2) double crystal deflector and (3) CRLs for vertical focusing.
- **PLS project:** benefits from SMI infrastructure (~$1M) and JPLS end station (~$1M)

J-PLS end station (under construction), will be enhanced for PLS





soft x-ray STXM for soft materials

Konstantine Kaznatcheev, Eliot Gann, Ron Pindak

September 11, 2018

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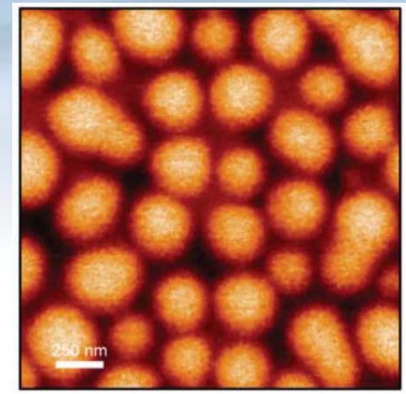
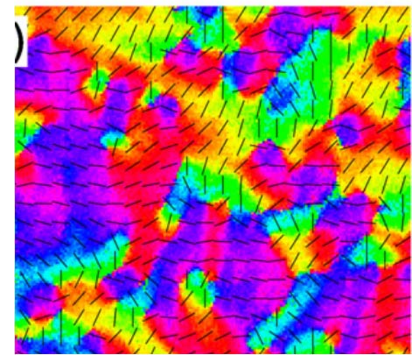
Rationale for the Proposed New Beamline

- **What is being proposed:** Leverages existing X1A NSLS components to build a soft x-ray (250-750eV) beamline and equip it with a versatile STXM dedicated to high resolution imaging of (radiation sensitive) soft matter and biological materials.
- **Science case:** Addresses key scientific directions of the Complex Scattering Program including: in-situ measurements of organic devices providing chemical mapping and orientational domain morphology, imaging of soft-tissue biological cells (supporting the regenerative medicine initiative at SBU), and ionic distributions in water filtration membranes.
- **User interests and demands:** Competitive beamlines include PolLux bend magnet beamline at the SLS (>20 pubs/yr) and the 5.3.2 bend magnet beamline at the ALS (>30 pubs/yr)
- **NSLS-II portfolio impact:** Complements the RSoXS and NEXAFS capabilities on SST. Will accommodate the NIST EM holders and include a cryo-cooling capability



Chemical compositional map of two OPV polymer components


Molecular orientaional domain morphology from linear dichroism

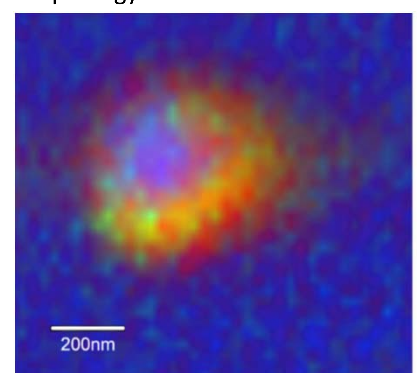
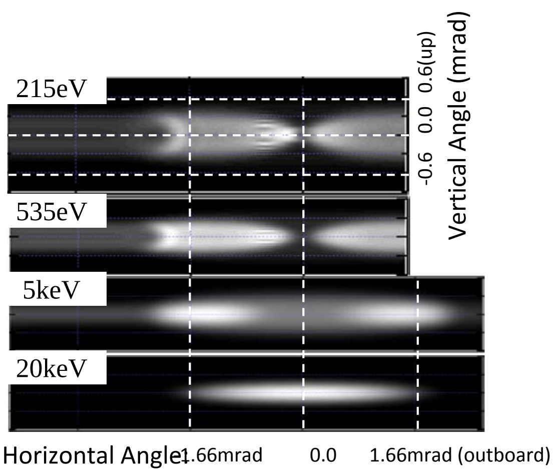
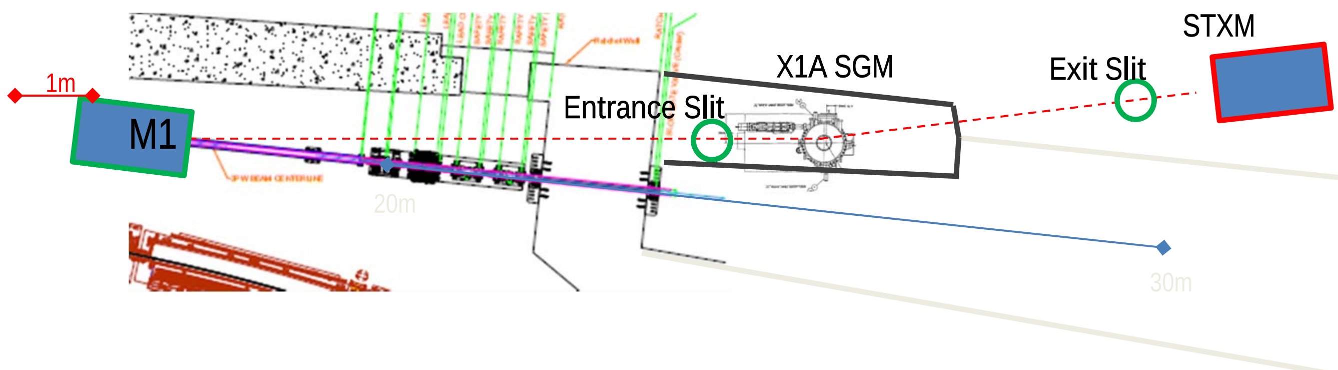


Image of nanoparticle encapsulated chemo-drug delivery to cancer cell

Proposed Technical Scope and Funding

- Both STXM limited angular acceptance and 3PW emitting properties permits STXM beamline development as a side-branch to a hard X-ray 3PW main-branch, while maintaining fully independent operation.
- TPW NSLS-II source brightness is similar to NSLS X1 ID and exceeds (x5) of ALS BM.
- Optical design follows NSLS X1 concept (also adopted by ALS): M1 (X13 ADC) focus the light on Entrance (horizontal direction) and Exit (vertical) Slits (X1A); horizontally dispersed SGM (X1A) provides good monochromatization (10^3) and adequate phase match ~2λ in each directions. STXM expects to reach an on-sample flux of 10^7 ph/s.





TIN: Tender and Hard X-ray Inelastic Scattering Nanoprobe

A Nanoprobe of Exotic Electronic Excitations in Complex Materials

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September 11, 2018

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Rationale for the Proposed New Beamline

- **What is being proposed:** World's first X-ray nanoprobe of electronic excitations using resonant/non-resonant IXS in the tender (2.5 – 4 keV) and hard (5 – 15 KeV) ranges with 50-nm spatial resolution and tunable energy resolution up to sub-10 meV.
- **Science case:**
  - Nanoscale ordering phenomena in QIS-relevant materials such as superconducting Weyl semimetals, spin liquid systems, and topological superconductors.
  - Imaging of emergent nanoscale spin textures
  - Imaging of charge dynamics in catalysis and energy storage materials under operando conditions to analyze microscopic mechanisms affecting functions and the cycle life.
  - Novel materials dynamics and quantum phase transitions under extreme pressure (>Mbar, up to the current static limit achievable by diamond anvil cells)
- **User interests and demands:**
  - Nano RIXS: Merges two techniques which are both in high demand. Promises to garner a lot of interest, as hinted by the high score received by ARI during the 2015 review.
  - Tender RIXS: Strong recent interest in the L edges of 4d TM and M edges of actinides, but limited availability of user instruments worldwide. No instrument in the US.
  - Hard RIXS: 27-ID at the APS the only hard x-ray RIXS instrument in the US and is heavily over-subscribed.

Proposed Technical Scope and Funding

- **NSLS-II portfolio impact:**
  - Together with IXS and SIX, TIN closes a critical capability gap for IXS studies at NSLS-II
  - TIN opens up a new dimension to NSLS-II's imaging capabilities in the tender and hard X-ray energy ranges with fine energy and momentum transfer resolutions
  - TIN will benefit from and contribute to the multi-modal effort in tender and hard X-ray imaging beamlines
- **Technical scope, performance, and feasibility:**
  - Will employ the Osaka double KB mirror design (Matsuyama et al, Opt. Ex. 20, 10310) to achieve a beam focus down to ~50 nm.
  - Two endstations, one all-in-vacuum designed for tender x-rays (2.5 – 4 keV) with 50-meV resolution, and the other for hard x-rays (5-15 keV) with tunable energy resolution from sub-10 to 200 meV.
  - Flexible spectrometer design to accommodate various types of analyzer optics (spherical analyzers and/or collimating + flat crystal optics).
  - Incorporate novel imaging/collimating optics for the analyzer to enhance signal rate.
  - TIN will build on the expertise of the IXS team in high-resolution crystal optics and of the Imaging Program in nano-focusing.