Scientific Opportunities for High Energy X-ray Diffraction with an MBA Lattice

S. Shastri[1], B. Suter,[2] J. Schuren,[3] and J. Almer[1]

Additional Contributors

- Hanna Leemerize and J. Birkedal (U. Aarhus)
- M. Li (ANL)

[1] ANL/APS
[2]Carnegie Mellon University
[3]AFRL

HEXD Science Enabled by MBA with SCU

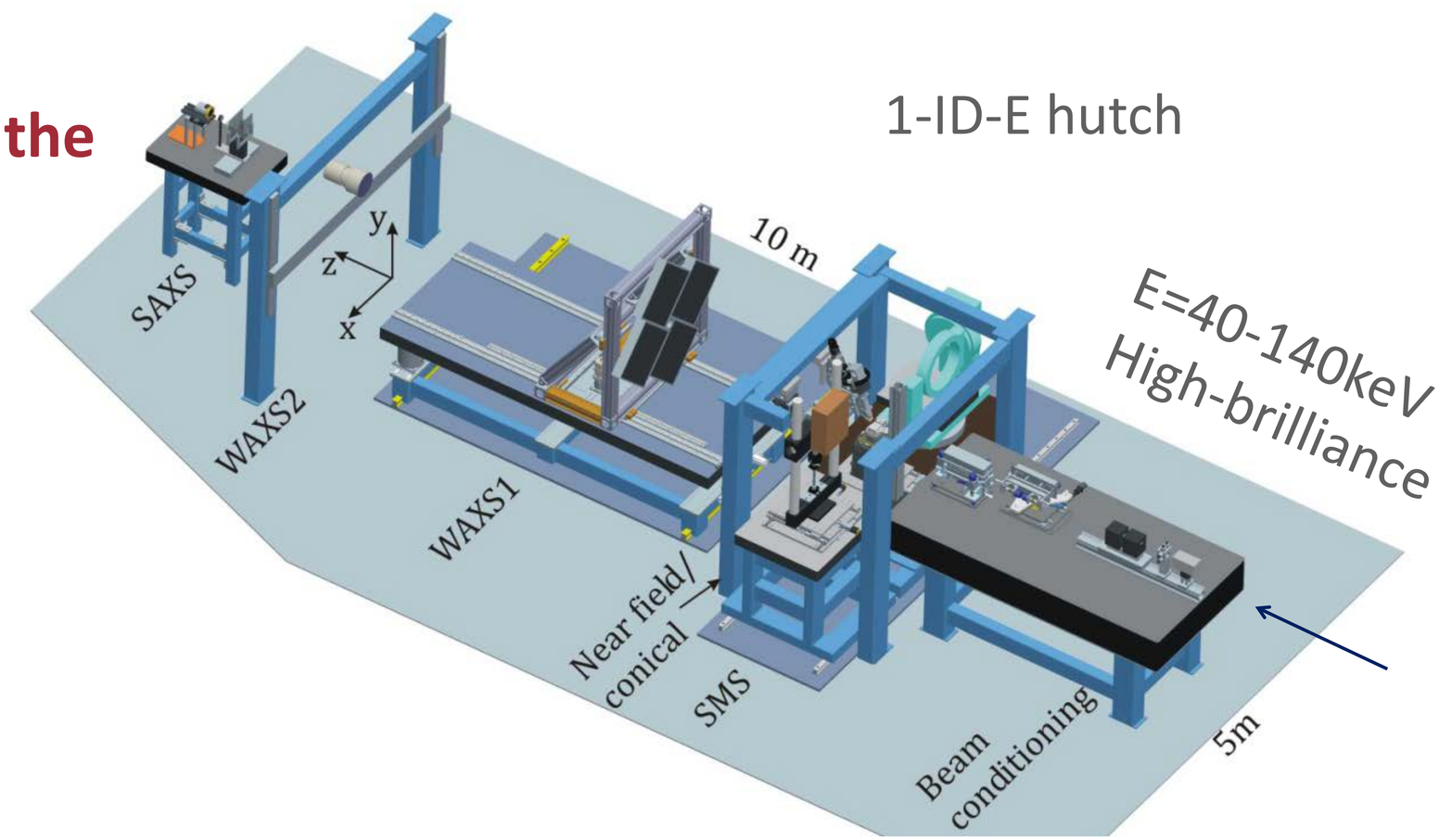
Integrated Computational Material Science and Engineering for Structural and Power Generation Material Systems

- **Structural Design Status Quo**
  - Links between materials processing, microstructure, and properties/performance have been built empirically for certain material systems
  - Current processing strives to produce homogeneous properties over a component such that designers can treat the constituent materials as a continuum
- **HEXD Science Enabled with MBA and SCU – Using inhomogeneous microstructure properties**
  - Brilliance limited techniques for mapping the structure and stresses in 3D in situ at the mesoscale enables **the development/validation of computational modeling tools** which allow designers to produce components with optimized, location specific properties
- **Fatigue Limited Design Status Quo**
  - Addressed statistically employing costly large scale mechanical testing programs to determine properties
  - Fundamentally limits incorporation of new materials with potential improvements
  - Countless theories for fatigue crack initiation exist, but no experimental validation at the relevant length scale exists
- **HEXD Science Enabled with MBA and SCU – Feasible to track evolution during fatigue**
  - With the flux increases we can track materials over hundreds of thousands of cycles during typical beamtime allocations to provide the key characteristics governing fatigue crack initiation

HEXD and Combined Modalities

- **Need for High Energy x-rays**
  - Critical for studying engineering materials employed in the transportation, power generation, and battery communities
  - HEXD enables 3D in situ mesoscale science which bridges basic research and industrial/societal impact
- **National Access**
  - APS 1-ID is the only HE beamline in the country where HEDM can be conducted
  - Many key technologies employ materials that cannot leave the country (i.e. materials employed in the nuclear power, defense, and aircraft/power generation communities)

APS-U will enable user access to combined modalities, *in situ*, on the same sample



Variety of detection modes:

- Tomography
- Near-field diffraction
- Far-field diffraction (WAXS1)
- Very far-field diffraction (WAXS2)
- Small-angle scattering

HEXD Gains with MBA

- APS now:
  - σ_x , σ_y , $\sigma_{x'}$, $\sigma_{y'}$ = 270 µm, 11 µm, 11.4 µrad, 3.5 µrad
  - 1-ID source: PMU, 2.4m long, 2.3 - 2.5 cm period
  - Full beam size at 70 m: 2.0 x 0.7 mm FWHM (H x V)
- MBA lattice:
  - σ_x , σ_y , $\sigma_{x'}$, $\sigma_{y'}$ = 9 µm, 4 µm, 7 µrad, 1.5 µrad (216 bunch-mode, 68 pm emittance, 11 % coupling)
  - 1-ID source: SCU, 2 m long, 1.7 cm period
  - Full beam size at 70 m: 1.2 x 0.4 mm FWHM (H x V)

Key Changes – full beam size decreases while flux increases

Technique(s)	Beam type used	Current: Beam size at sample (HxV), µm*	MBA: Beam size at sample (HxV), µm*	Flux(-density) gain on sample at 70 keV: MBA/now	Comments
Near-field diffraction (nf-HEDM)	1d v-focused@ short-f	2000 x 0.8	1200 x 0.3	3.6 (9.7)	Desire mm's -wide beam
Far-field diffraction, PDF, SAXS	2d focused @ long-f	635 x 26	21 x 9	6.0 (473)	Fully brilliance driven
Far-field diffraction, PDF, SAXS	2d focused @ short-f	19 x 0.8	0.6 x 0.3	6.0 (473)	Fully brilliance driven
Far-field diffraction, PDF, flourescence	Slit, unfocused	Variable (100x100 typ)	Variable (100x100 typ)	6.0 (6.0)	Flux density driven
Direct beam tomography	Full, unfocused	2000 x 700	1200 x 400	2.3 (6.0)	Total flux driven

- Focused #s are source-size limited values (assume perfect optic)

Short-f: 68m source-optic, 2m optic-sample
Long-f: 35m source-optic, 35m optic-sample

HEDM Enabling Predictive Mesoscale Science

Opportunity

- Polycrystals serve in all sectors of the economy, frequently under extreme conditions of temperature and loading
- HEDM maps crystal orientations at sub-grain scale (nf) and strains at single grain scale (ff) in-situ in 3D
- Application critical materials responses tracked
- Computational models validated against real data required for accelerated materials development

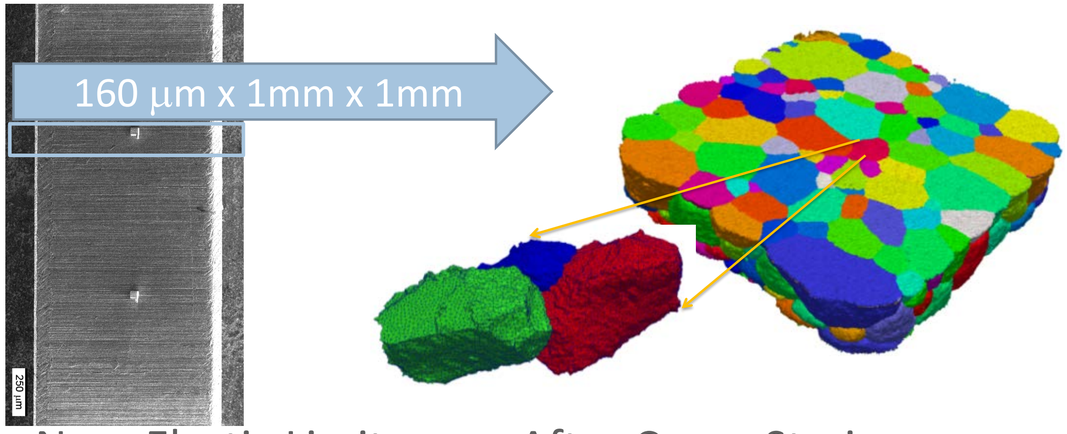
Challenges

- Many critical processes depend on “rare events” making large, high resolution datasets required: position, orientation, strain (12 dimensions + time)
- Develop predictive capability for myriad applications including turbine blades, metal processing and battery operation – *Central to materials genome initiative/predictive mesoscale science*
- High user demand: National labs, industry, academia

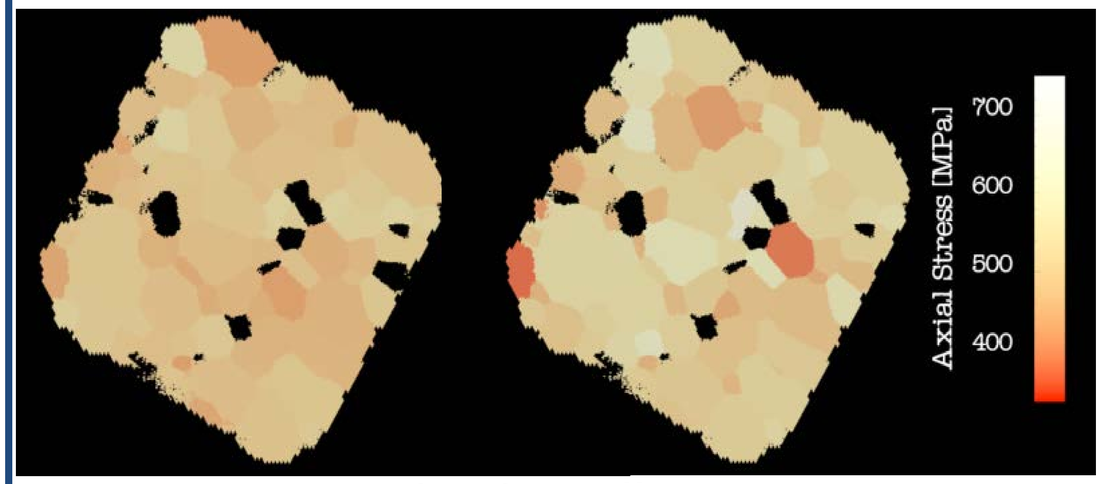
4GSR Strength

- MBA/SCU high flux leads to rapid data collection and opportunity to study fatigue
- Focused beam: improved spatial resolution
- Beam stability: accuracy of large datasets
- Higher flux shifts technique from physics novelty to engineering tool

AFRL Led PUP at Sector 1:
Enabling Predictive Mesoscale Science



Near Elastic Limit After Creep Strain



Titanium alloy

nf-HEDM orientation map
+
ff-HEDM strains superimposed
+
HE absorption tomography

Multi-$M AF investment

Images from PUP Team: AFRL, APS,LLNL, CMU

MBA and SCU Enables New HEDM Capabilities

Opportunity

- Fatigue cracks have been seen to start both within and between grains. Models exist but essentially no data inside real materials
- Upgrade makes tracking of mechanical conditions in situ in 3D possible : intragranular fatigue crack initiation observation

Challenges

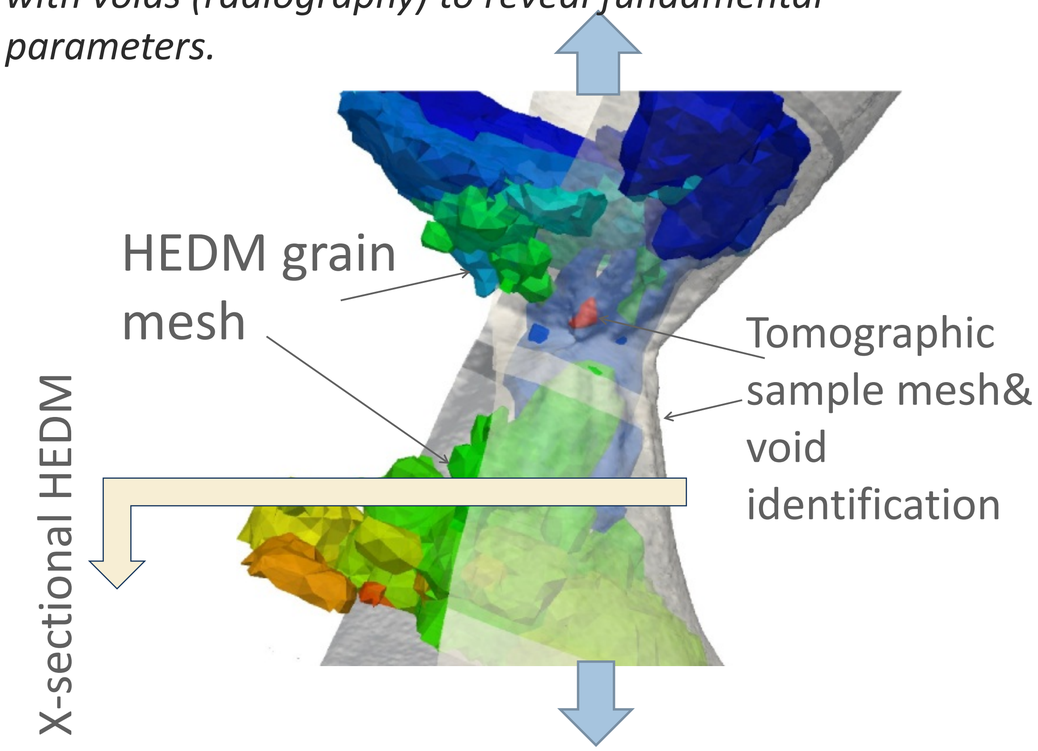
- Ability to map strains on sub-grain scale in ~1000 grains does not exist
- Require reduced focal size < 1μm AND small divergence AND high stability
- Increased flux for high-energy resolution: $\Delta E/E \sim 10^{-4}$ cuts flux by ~20X from HEDM standard beam
- Ex: find origins of cracks in fatigued materials: intra-grain stress concentrations

4GSR Strength

- Gain of ~4X intensity at 70keV in focused beam
- Submicron focusing < 1μm spatial resolution reveals earliest stages of deformation, higher deformation states and more complex materials (e.g. composites)
- High brilliance will allow practical data speeds with high energy resolution for strain sensitivity
-

In-situ deformation/annealing studies

Individual grain characteristics (HEDM) can be correlated with voids (radiography) to reveal fundamental parameters.



Stresses within polycrystals are anisotropic and inhomogeneous

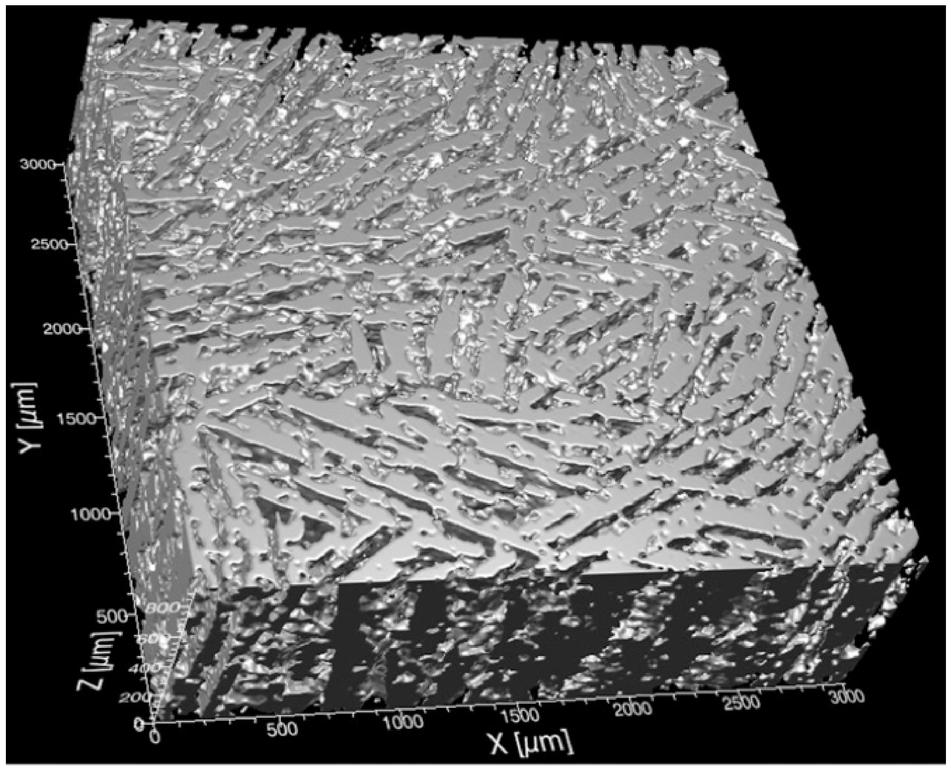
nf-HEDM extension to strain mapping

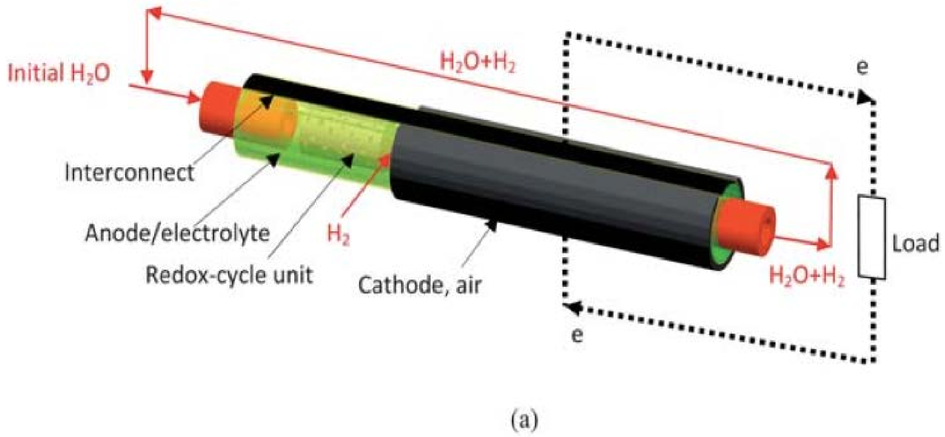
- Resolve in ω rather than 2θ
- Split strain states between images
- Proof-of-principle, July 2013
- 4 hours for 1 layer; analysis on-going
- Becomes practical tool for MGI, ICME with MBA's 10X brilliance at high E

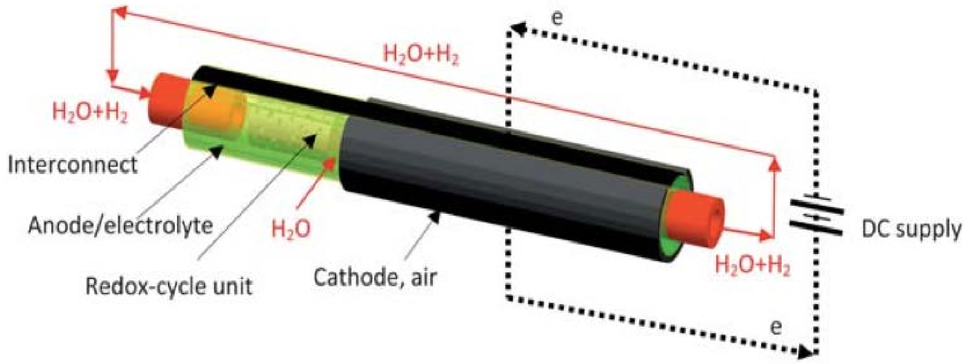
Images courtesy of B Suter et al.

HEDM and Iron-Air Foam Batteries

- Is phase change during cycling homogeneous, or are pore walls more or less favorable sites than struts.
- How does strain associated with Iron redox evolve in the matrix with cycling.
- How do the size, shape and tortuosity of the pores affect phase change, strain and cycling.
- How do additives in the foams affect phase changes, strain and cycling?



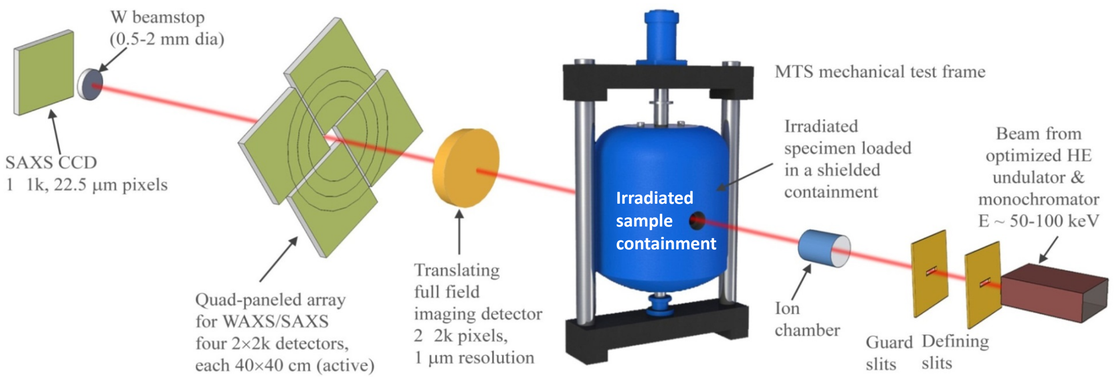




N. Xu *et al.*, *Energy Environ. Sci.*, vol. 4, no. 12, pp. 4942–4946, Nov. 2011

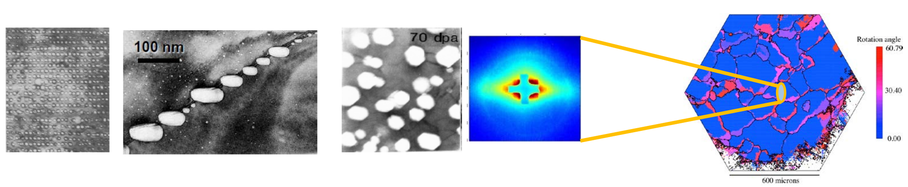
In situ Studies of Nuclear Materials in Extreme Environments

In situ Characterization of Irradiated Materials under Thermo-mechanical Loading



Combined techniques and penetrating hard x-rays visualize material evolution on relevant length scales:

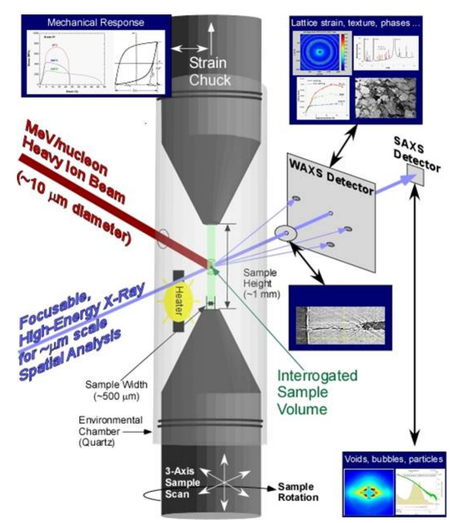
- Tomography = meso-scale structure
- **S**mall **A**ngle **X**-ray **S**cattering = nano-scale structure
- **W**ide **A**ngle **X**-ray **S**cattering = atomic scale structure (phase and lattice strain)

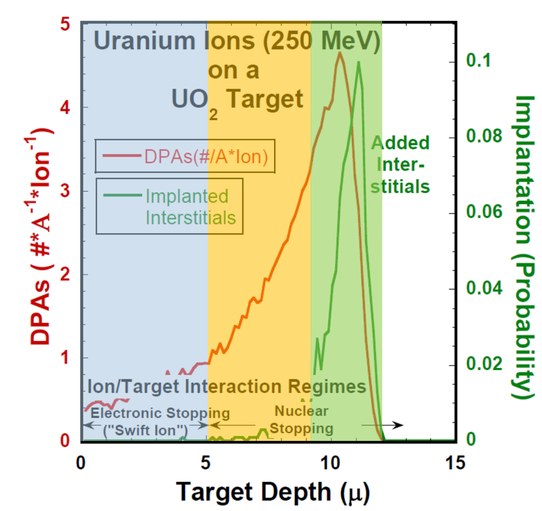


Information of spatial distributions of irradiation defects, e.g. voids and bubbles, and defect annihilation at microstructural sinks measured by combined WAXS/SAXS/tomography is critical to the design of high-performance materials and development of predictive models.

XMAT*:* eXtreme MATertials beamline for *in situ* studies of materials under irradiation

The proposed XMAT beamline will provide x-ray probes for *in-situ* study of materials in simulated nuclear reactor environments, enabling rapid evaluation of new materials performance under extreme service conditions including structural materials and for the first time nuclear fuels.





Focused X-ray beams allow studies of spatially-separated effects with well-controlled parameters in extreme radiation environments.

Capturing kinetics during materials synthesis & processing

Opportunity

- Synthesized and engineered materials offer new opportunities in energy production and storage.
- Creating materials requires *in situ*, multi-modal structural characterization techniques to understand and improve processing and performance.

Challenge

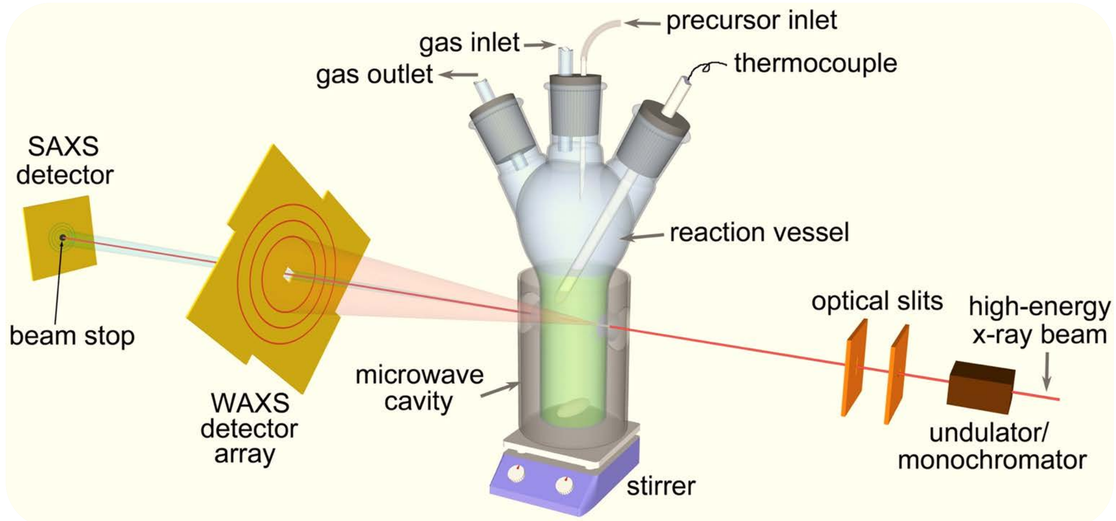
- Combine processing environments with high-energy x-rays to enable high-throughput screening of engineered and synthesized (nano)materials.
- Provide a generic platform to study structural transitions and defect evolution of (nano)materials in real time and under relevant conditions.

4GSR Strength

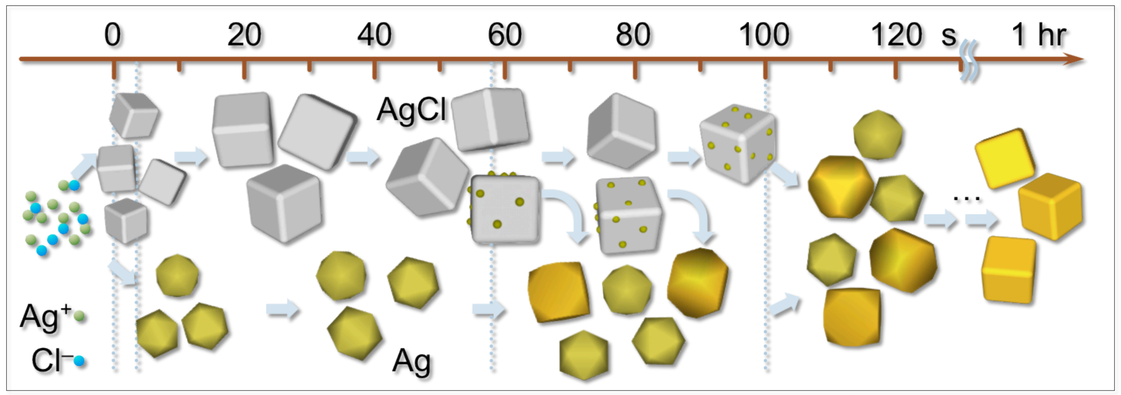
- HEXD with MBA will enhance brilliance-driven SAXS/WAXS, increase temporal resolution, enable lower Z-materials and sensitivity for dilute systems
- 10X improved q_{min} for SAXS -> probe larger particles
- Impact a range of studies including:
  - nano-particle synthesis
  - dynamic deformation of structural materials
  - dynamics in welding metal structures

In situ nano particle synthesis

High-energy x-rays penetrate through complex chemical reactor vessels. Atomic- and nanoscale structures determined from multiple detector signals.



Wide **A**ngle **X**-ray **S**cattering yields atomic arrangement and chemical phases present. **S**mall **A**ngle **X**-ray **S**cattering produces information on particle size and morphology.



Nanophase evolution involved in the synthesis of Ag nanocubes

Peng, Okasinski, Almer, Ren, Wang, Yang, and Sun, *J. Phys. Chem. C* **2012**, *116*, 11842.

High Energy X-ray Diffraction Science

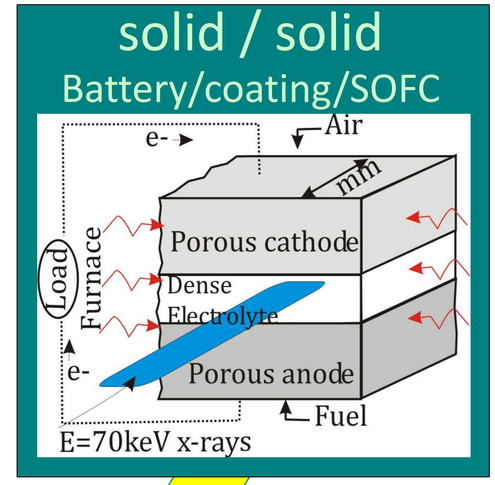
Studies of Buried Interfaces

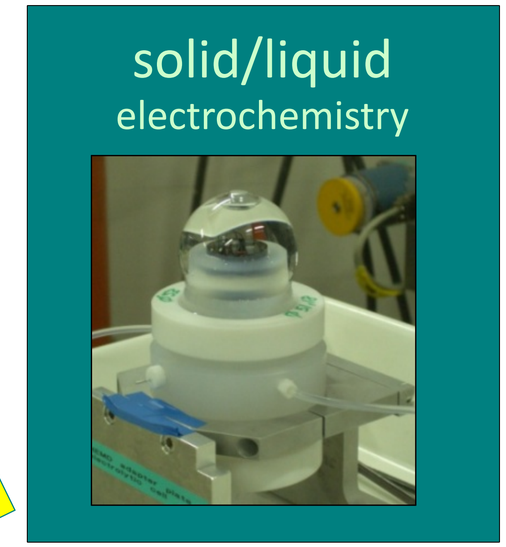
Microfocused high-energies enable complementary techniques to probe buried interfaces

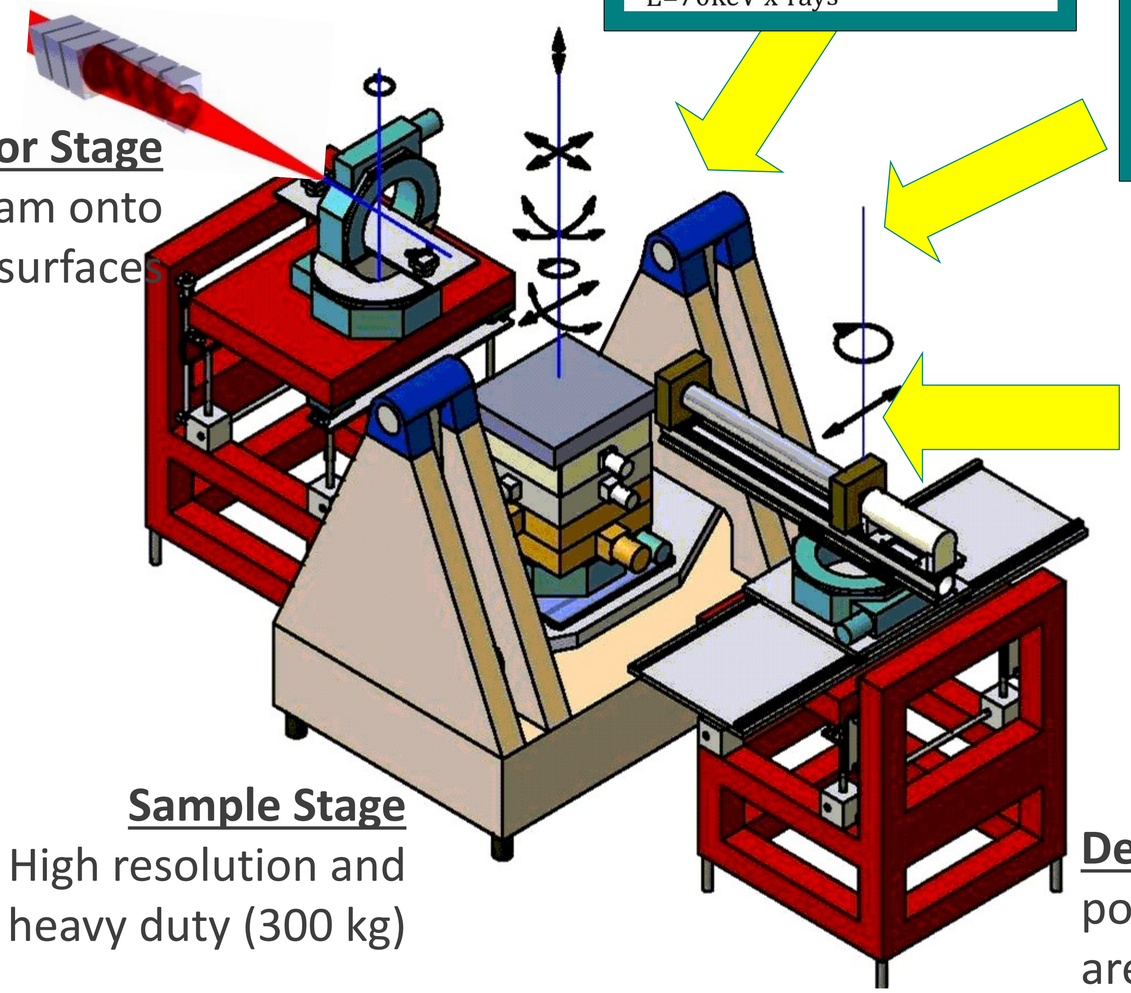
- Transmission SAXS/WAXS
- Glancing Incidence SAXS/ WAXS: enhanced surface sensitivity
- X-Ray Reflectivity: e^- density depth profile
- Crystal Truncation Rod measurements

2D focusing enabled by MBA lattice is critical to extend this technique to technological (non-perfect) interfaces:

- Vertical: reduce footprint for rougher surfaces, higher resolution
- Horizontal: scan in-plane homogeneities







This new probe is complementary to the surface-science beamlines and will enable studies under a variety of sample environments, particularly focused on energy-materials.

Diffraction Tomography

Opportunity

- Provides rich set of imaging information for any scattered signal; used in lieu of HEDM when spot overlap cannot be avoided:
- Nano-grains, high deformation, weakly ordered systems
- Energy materials and biological materials

Challenges

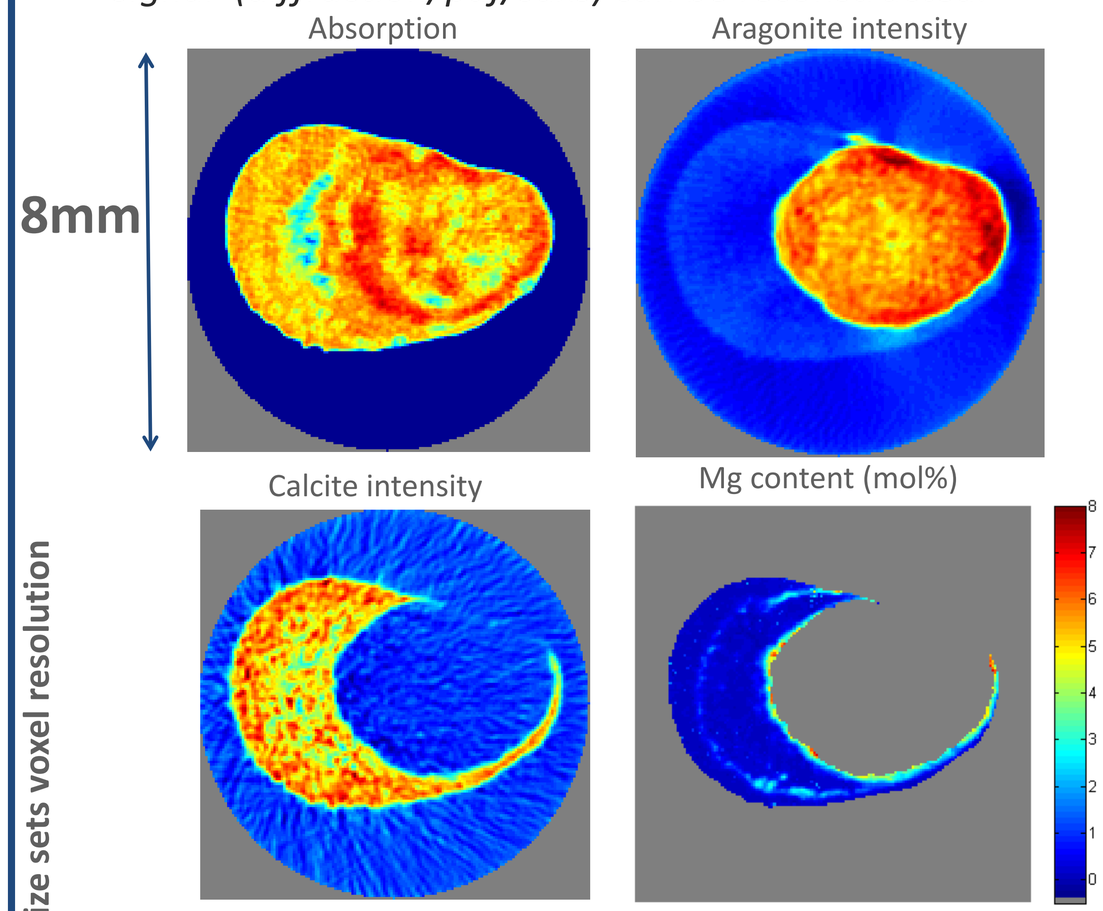
- Require reduced horizontal focal size ~1 um AND small divergence
- Data intense: Need for N^2 measurements for a single layer

4GSR Strength

- Ability to focus horizontally to ~1um will DIRECTLY benefit achievable resolution, currently set at ~10um
- Increased flux will permit rapid data collection
- This combination will allow voxel resolutions to micron level – enabling new methods to image material heterogeneity

Imaging with rich detail

Diffraction spectra from byssus sample are voxellized to produce detailed maps of phase distribution including intra-phase composition. Correlated data for any scattering signal (diffraction/pdf/saxs) can be reconstructed.



Leemreize, Almer, Stock & Birkedal. J. R. Soc. Interface 10 (2013)

DT technique: scan + rotate

Scientific Opportunities for High Energy X-ray Diffraction with an MBA Lattice

- MBA/SCU will enable critical advances in understanding of complex nano- to macro-scale responses of materials
  - High brightness, small source size, improved beam stability
  - Coupled to national priorities: Materials Genome Initiative, Integrated Computational Materials Engineering, energy, defense, civilian applications / manufacturing

- Concerns / cautions
  - HEDM relies on millimeter horizontal dimension of beam at sample
  - Throughput already an issue – accommodate fixed E side beamline?

- Needs
  - Aberration-corrected sawtooth lenses for sub-micron resolution
  - Possible defocusing/expanding optics for larger samples
  - Large area detectors: faster, better resolution, direct detection
  - High resolution detectors: improved (higher efficiency, spatial resolution) scintillators -- current limit on data rate for nf-HEDM
  - Computational infrastructure: direct connection to ANL systems
  - New types of data – new types of analysis will continue to be developed