Argonne NATIONAL LABORATORY

FY 2016 NEET R&D Award -

Investigating Grain Dynamics in Irradiated Materials with High-Energy X-rays

PIs:

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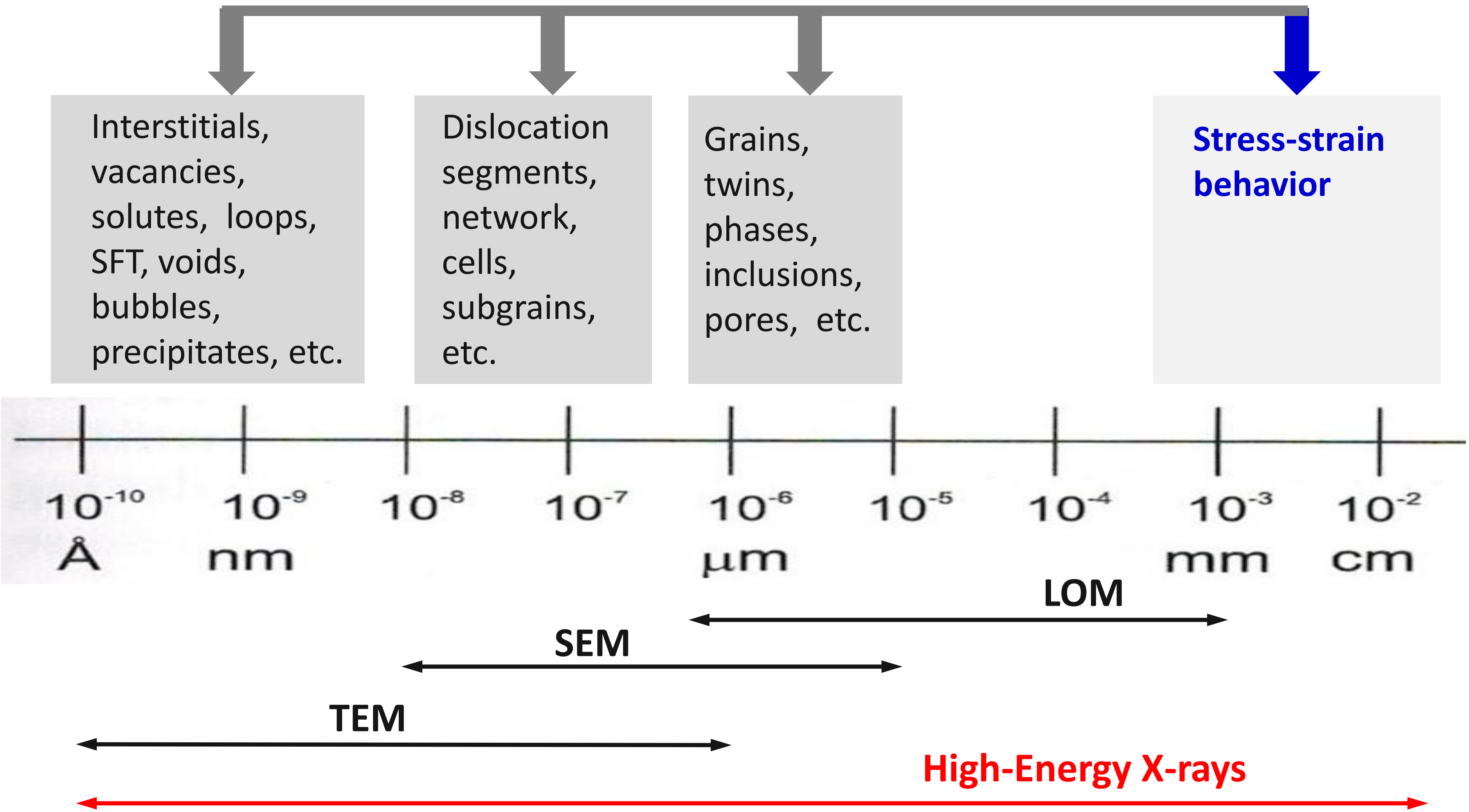
DOE-NE Cross-cut Coordination Meeting
August 16, 2016

U.S. DEPARTMENT OF ENERGY

Motivation - *In situ* Multiscale Experiment of Nuclear Reactor Materials with High-Energy X-rays



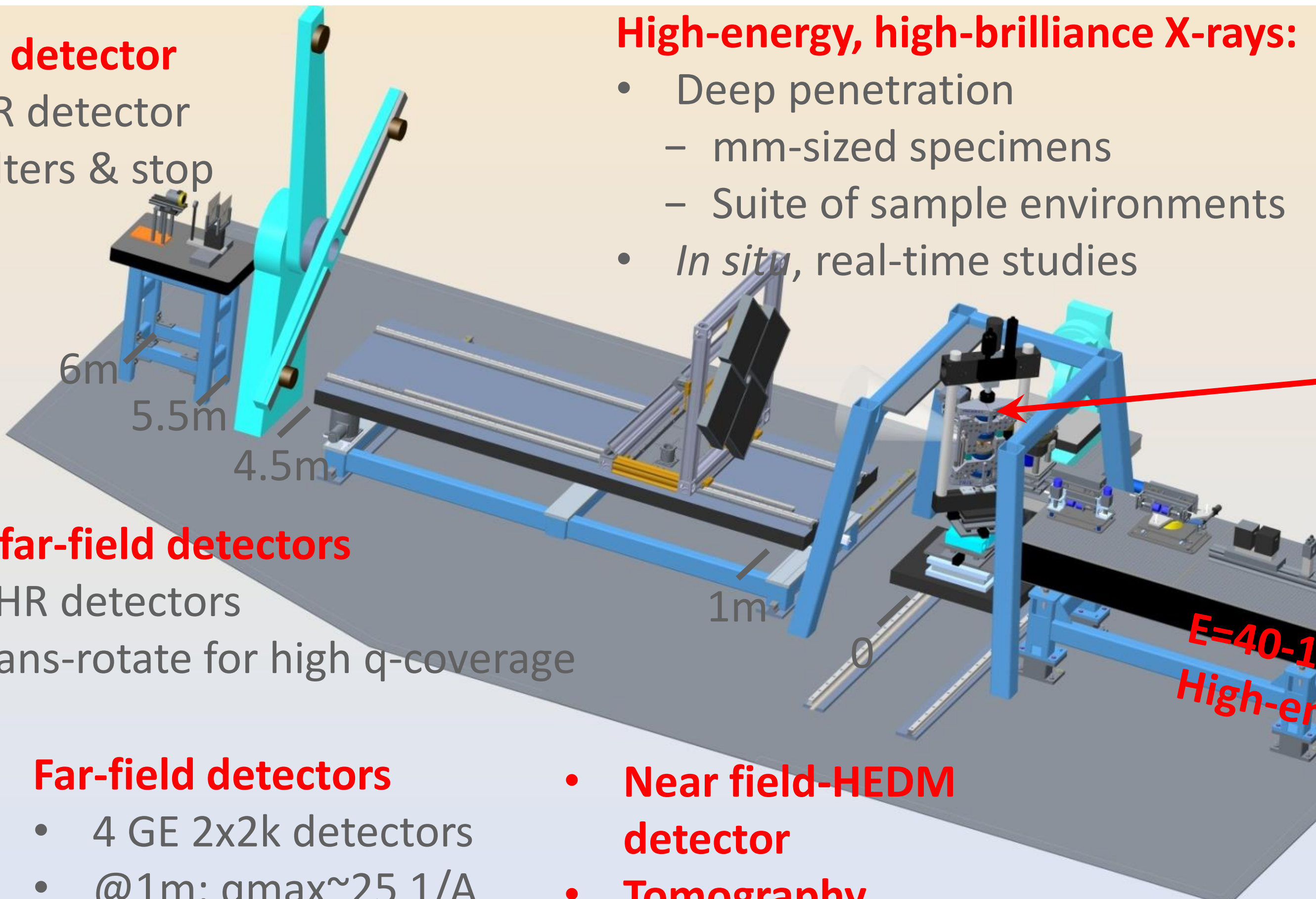
Combined *In situ* High-Energy X-ray Techniques (APS Beamline 1-ID)

High-energy, high-brilliance X-rays:

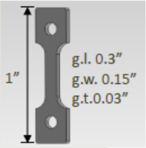
- Deep penetration
  - mm-sized specimens
  - Suite of sample environments
- *In situ*, real-time studies

SAXS detector

- HR detector
- Filters & stop



Lab-scale mechanical test



Very far-field detectors

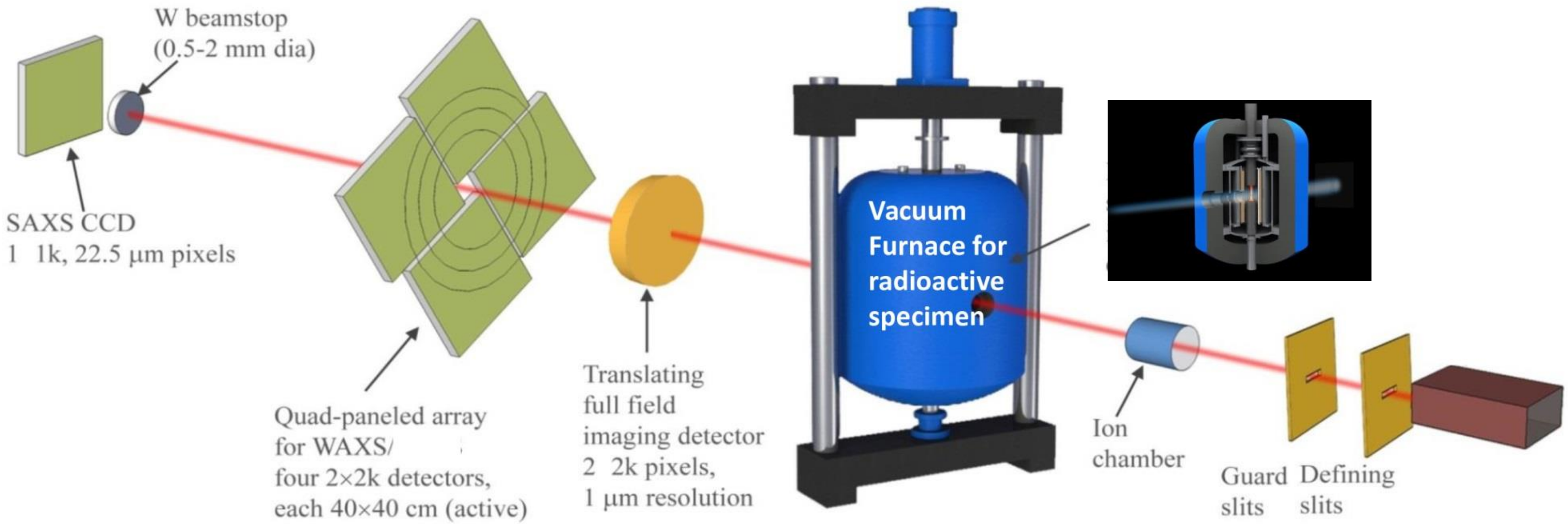
- 3 HR detectors
- Trans-rotate for high q-coverage

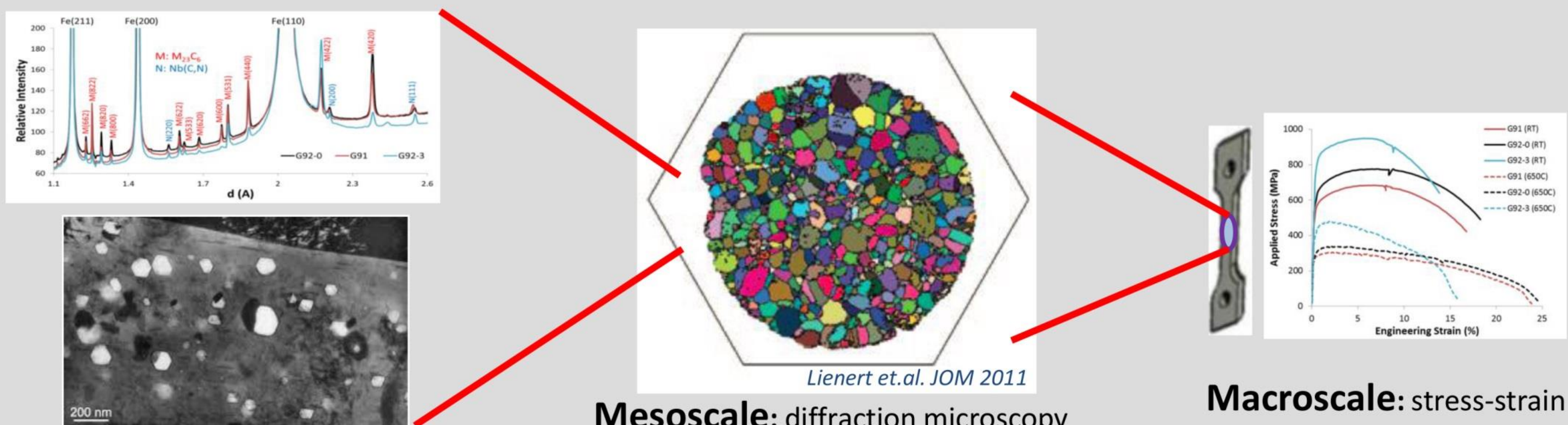
Far-field detectors

- 4 GE 2x2k detectors
- @1m: qmax~25 1/A
- Center-hole (SAXS)

- **Near field-HEDM detector**
- **Tomography**
- Conical slit
- Lasers

In situ Thermal-Mechanical Experiment of Neutron-Irradiated Materials



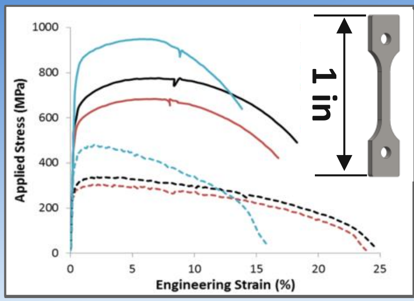


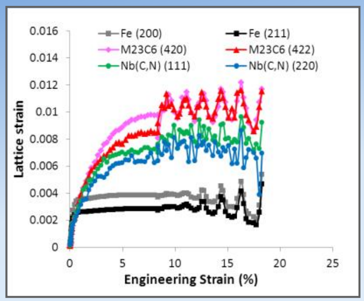
(Sub)nanoscale: WAXS and SAXS

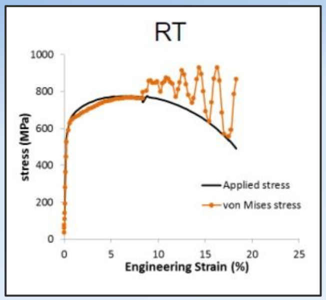
Mesoscale: diffraction microscopy & tomography

Macroscale: stress-strain behavior

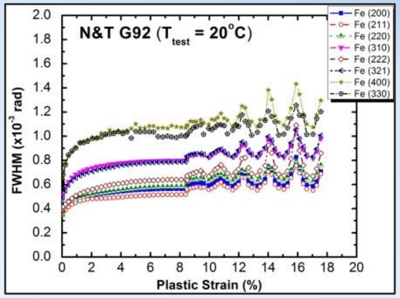
In situ Characterization using 2D X-ray Techniques: Grain-Average Behavior

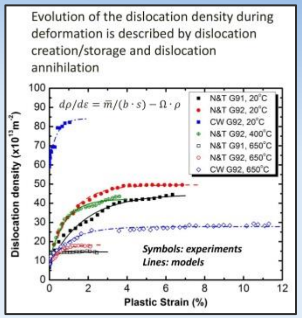


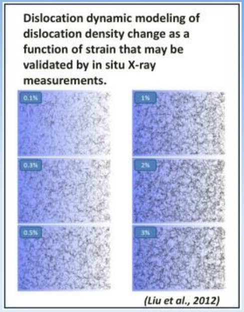




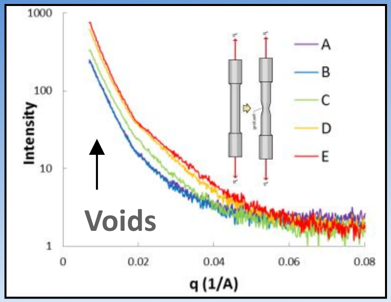
Diffraction peak shifts revealed load partitioning among phases during deformation

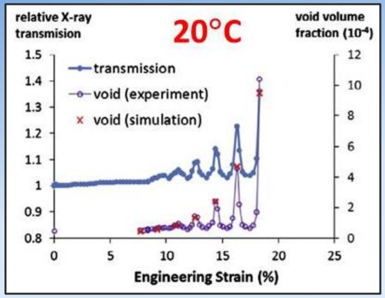


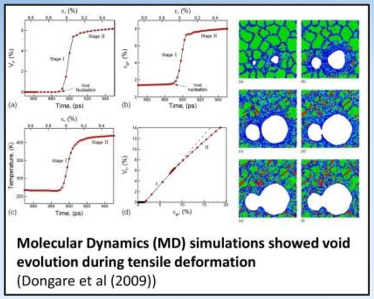




Diffraction peak broadening revealed dislocation evolution during deformation



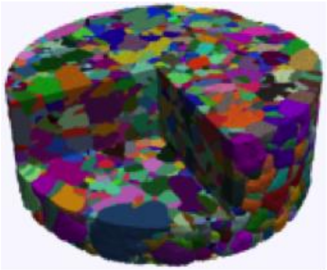




SAXS captured void formation and evolution during necking

High-Energy Diffraction Microscopy (3D-XRD): Individual Grain Characteristics

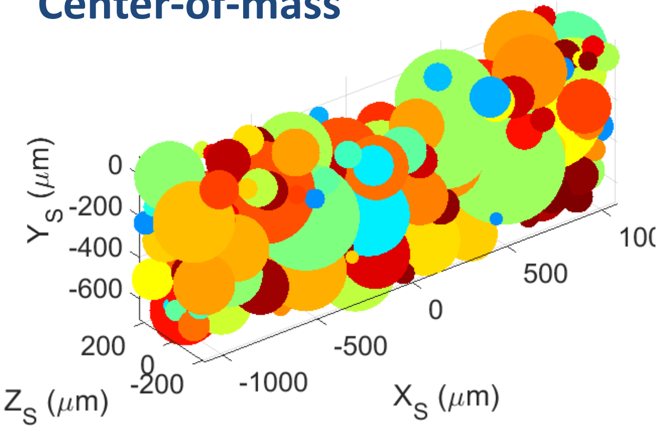
- **Three-dimensional, grain-scale characterization**:
  - Characterization of microstructural and micromechanical response of individual grains within the bulk of a polycrystalline specimen.
- **Near-field (nf-) HEDM** (similar to 3D-EBSD):
  - Sample-detector distance: mm – cm
  - Not suitable for in situ study with complex environments



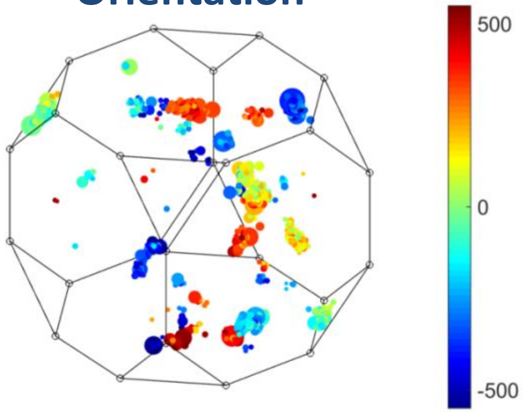
- Grain shape and orientation map
- Detailed GB geometry

- **Far-field (ff-) HEDM:** sample-detector distance: meter (suitable for *in situ*)

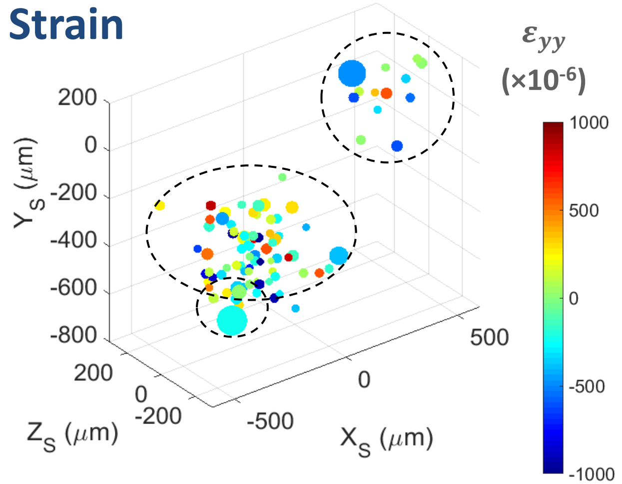
Center-of-mass



Orientation

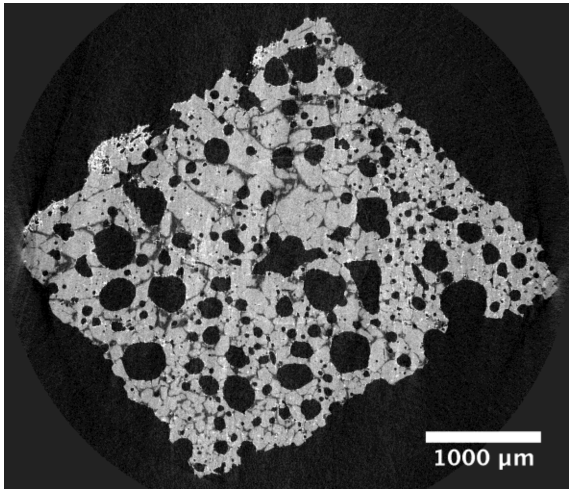


Strain

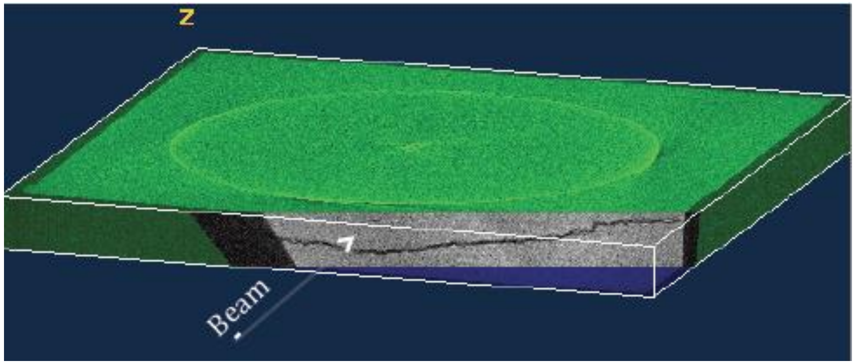


X-ray Tomography

- 3D visualization of the internal structure (pores, voids, cracks, etc.) in a material



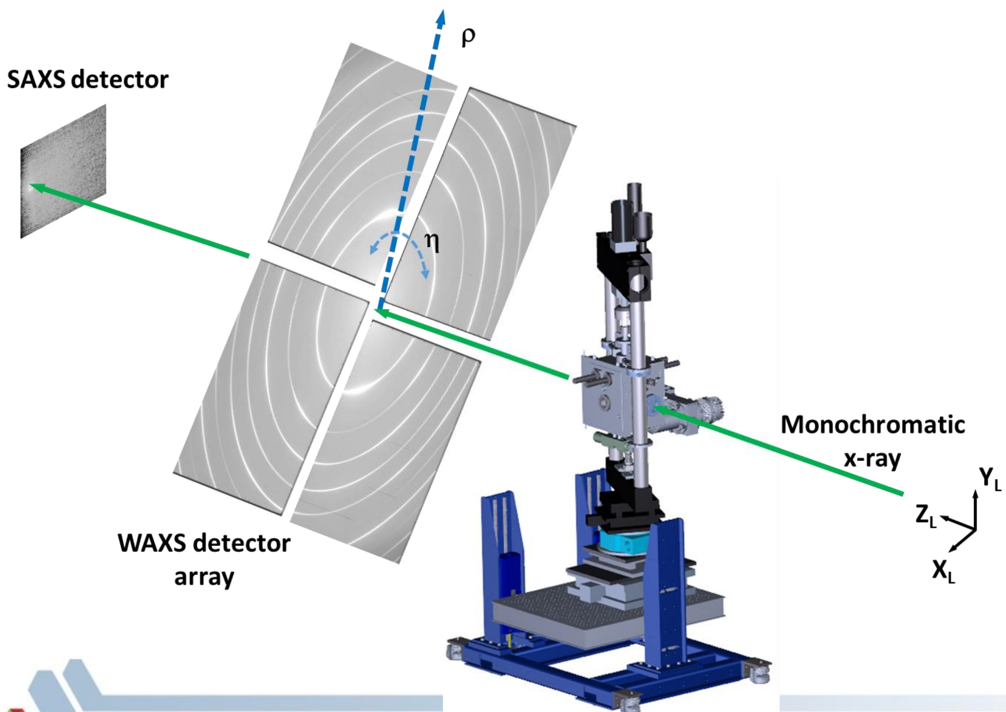
Absorption Tomography provides information due to electron density, revealing presence of voids, cracks, etc.
(by AFRL, unpulished)

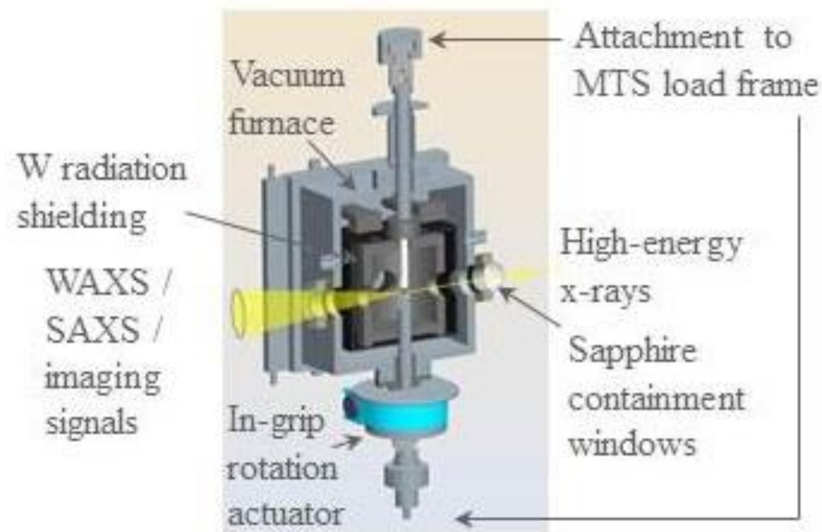


X-ray tomography of thermally-fatigue GlidCop specimen measured at APS beamline 1-ID.
(A. Khounsary et al. J. Phys 425 (2013) 212015)

In situ 4D Characterization under Thermal-Mechanical Loading

- Integrate *in situ* thermal-mechanical loading capability with 3D X-ray techniques to enable time- and spatial-resolved (4D) characterization of neutron-irradiated polycrystalline materials
  - Single-grain diffraction measurements in a polycrystalline sample
  - Monitor grain dynamics under thermal-mechanical loading
  - Link local events with average properties

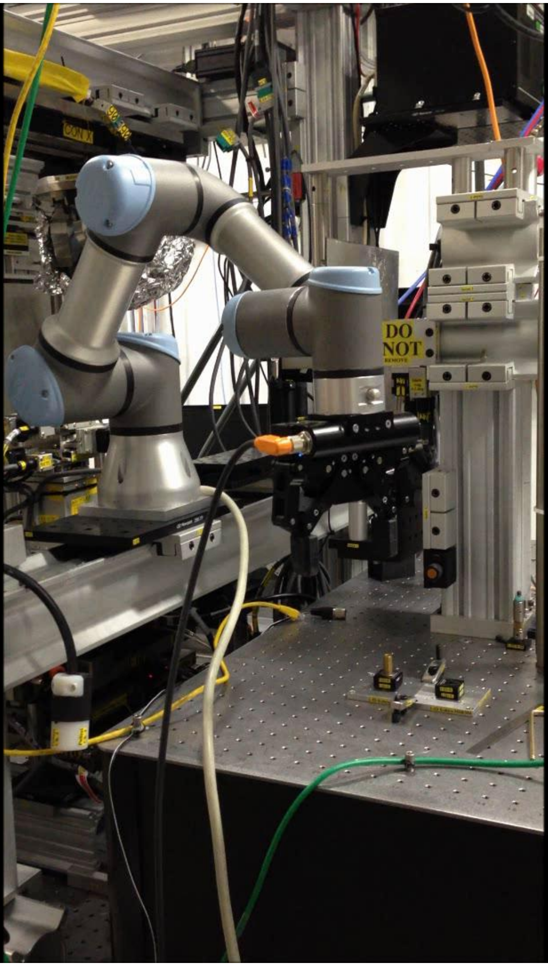




Proposed Work

- **4D characterization of neutron-irradiated materials with high-energy X-rays**
  - Demonstration of high-precision rotation & axial loading mechanism to enable 4D X-ray characterization
  - Develop HEDM data analysis methods to specifically address irradiated specimens, including effects of peak overlap and broadening which result from radiation damage
  - Develop a swappable specimen stage with a location tracking mechanism for synchronized measurements of the same specimen volume by *ex situ* nf-HEDM and *in situ* ff-HEDM.
- *In situ* mechanical testing of neutron-irradiated materials at high temperature with high-energy X-rays
  - Demonstrate the capability for *in situ* mechanical testing of an activated specimen at temperatures up to 600°C.
- Establish activated specimen holder library
  - A collection of well-established and approved holder designs for irradiated specimens will be of great value to the nuclear material community.
  - The specimen holder library will be closely coordinated with the NSUF sample library to accommodate different sample types, geometry and dimensions.

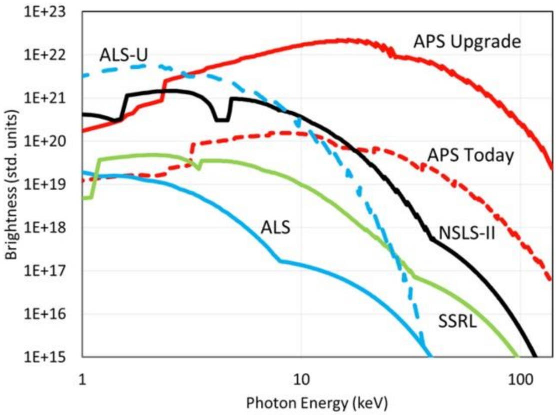
Automated Handling of Radioactive Specimens using Robot



What's Next?

APS Upgrade – Transform today's APS into the ultimate 3D X-ray microscope

Orders-of-magnitude increase in brightness and coherent X-ray flux.



Early Science at the Upgraded APS, Oct. 2015.

High energy, high brilliance X-rays → ***High energy, high brilliance, high coherence X-rays***

New Beamline Concept – "<u>High-Energy X-ray Microscope (HEXM)</u>"

- New imaging techniques
  - Direction-beam imaging: full-field transmission X-ray microscopy (TXM)
  - Diffraction-beam imaging: Bragg coherent diffraction imaging (BCDI)
- A long beam extending beyond APS experimental hall allows separate building
  - Include *in situ* ion irradiation beamline (XMAT)
  - Include Activated Material Laboratory (AML)