

2024 NUCLEAR SCIENCE USER FACILITIES PROGRAM REVIEW MEETING

ACTIVATED MATERIALS LABORATORY AT THE UPGRADED APS



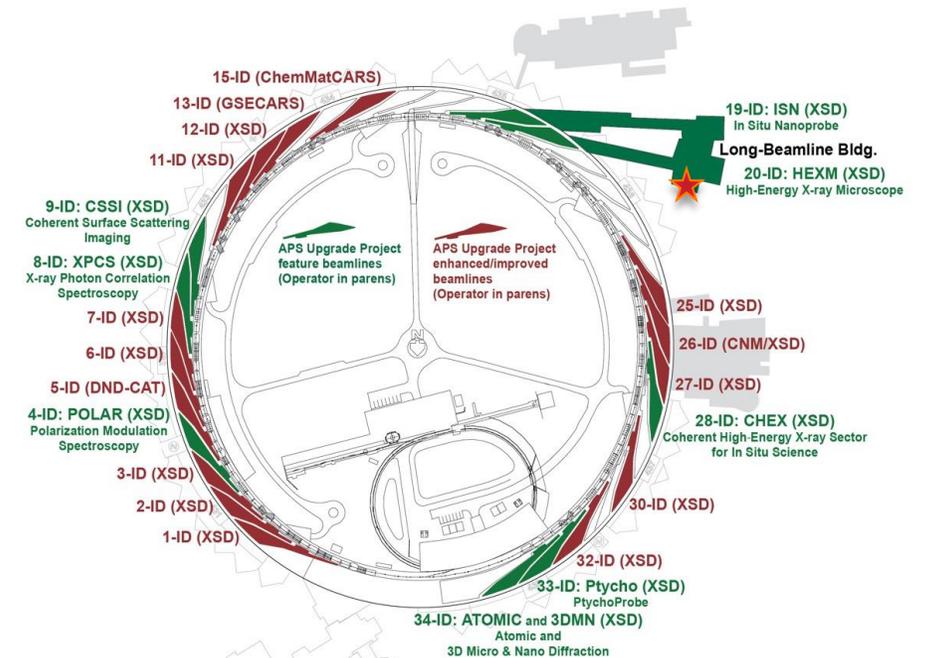
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ACTIVATED MATERIALS LABORATORY (AML)

- AML is a **Low-Activity Specimen Preparation Laboratory** next to the **High Energy X-ray Microscope (HEXM)** beamline in the **Long Beamline Building** being constructed at the APS, as part of the APS Upgrade Project.
- AML is an NSUF partner user facility.

Key dates:

- APS dark period: 2023-04 to 2024-04 (just ended)
- AML user access: 2024 fall



AML COMPONENTS

For more details, see:

<https://www.anl.gov/nse/activated-materials-laboratory>



- 2 sets of glove boxes and fume hoods
- 1 heavy duty hoist



- 2 lead-shielded storage cabinets
- 2 lead-shielded rolling barriers
- 1 lead-shielded mobile cart



- Mezzanine area: air duct with HEPA filters
- Basement area: 3 retention tanks (in case of water spill in AML)



- 1 Keyence VHX 7000 digital microscope with automated XYZ stage
- 20-200x magnification

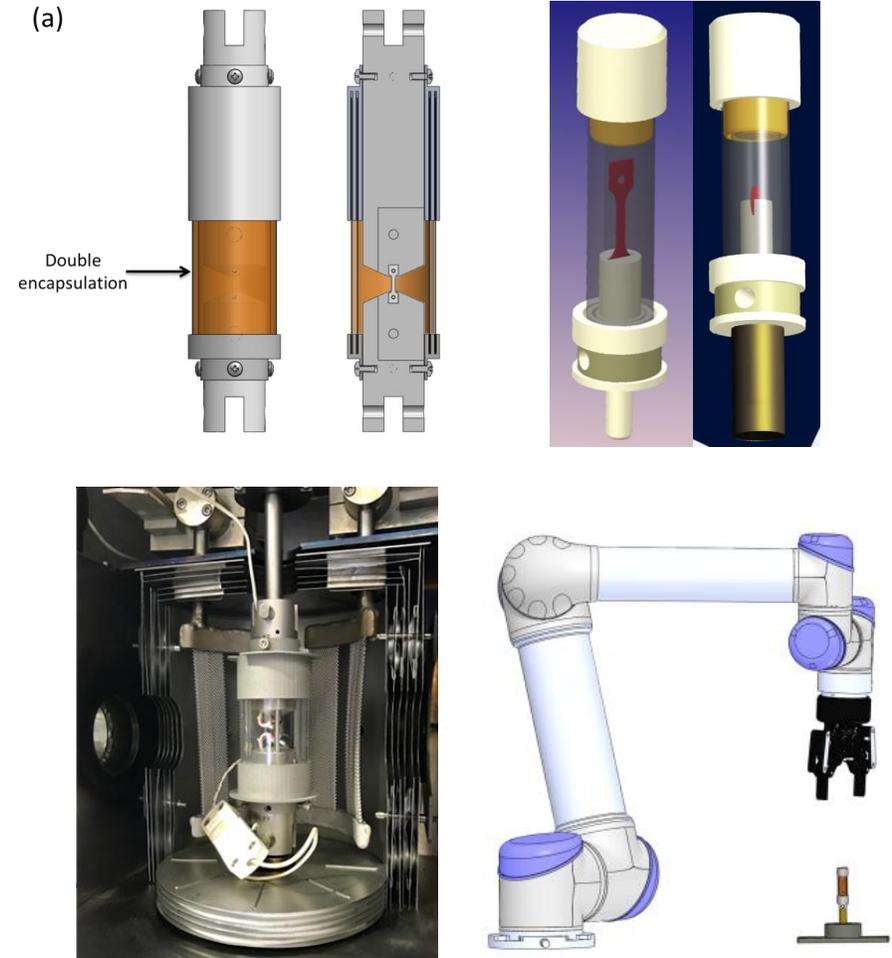
AML SCOPE AND FUNCTION

- An NSUF partner user facility
- A central lab providing encapsulated Rad samples for characterization at all APS beamlines
- A Radiological Facility
 - Radiation exposure limit: 100 mR/h at 30 cm.
 - Nuclear fuel samples will be handled inside glove boxes
 - ALARA engineered controls/w HEPA filtered exhaust
- A Radioactive Specimen Preparation Lab
 - Receiving/shipping samples
 - Handle open Rad samples in solid form
 - Support radioactive sample experiments at beamlines
 - Interface with APS's Radioactive Sample Safety Review Committee (RSSRC)
- AML will work with other onsite radiological facilities for more involved initial processing and sample preparation



RAD SAMPLE ENCAPSULATION

- All radioactive samples at the APS require containment. Containment requirements will depend on the sample form, size, activity, and other factors. The containment must be approved by the Radioactive Sample Safety Review Committee (RSSRC).
- AML and beamline staff will assist users in design and engineering of sample containment and shielding, communications with RSSRC members, and participate in RSSRC review.
- Effort required depends on specific experiments.



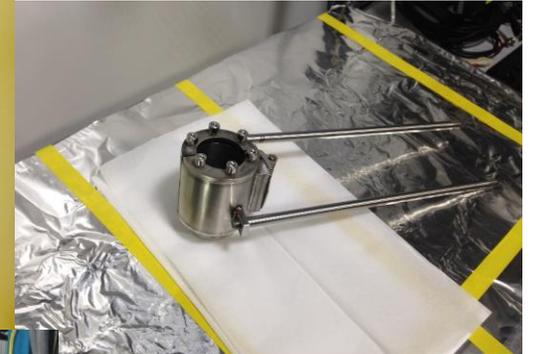
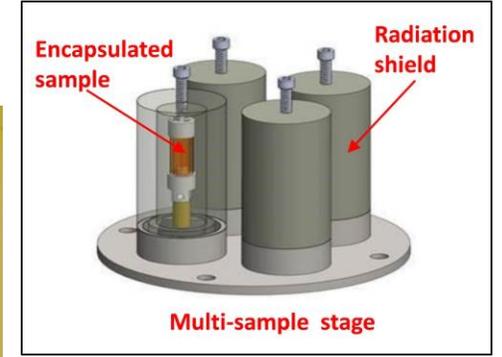
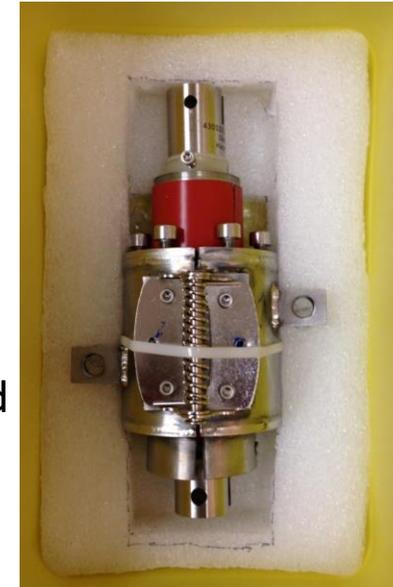
SAMPLE SHIELDING AND STORAGE

■ Sample shielding

- Set up local shielding
- Sample survey, inventory, cleaning, decontamination
- Sample mounting, holder assembling
- Sample transfer to beamlines
- Sample removal and post-experiment for temporary storage and return shipment

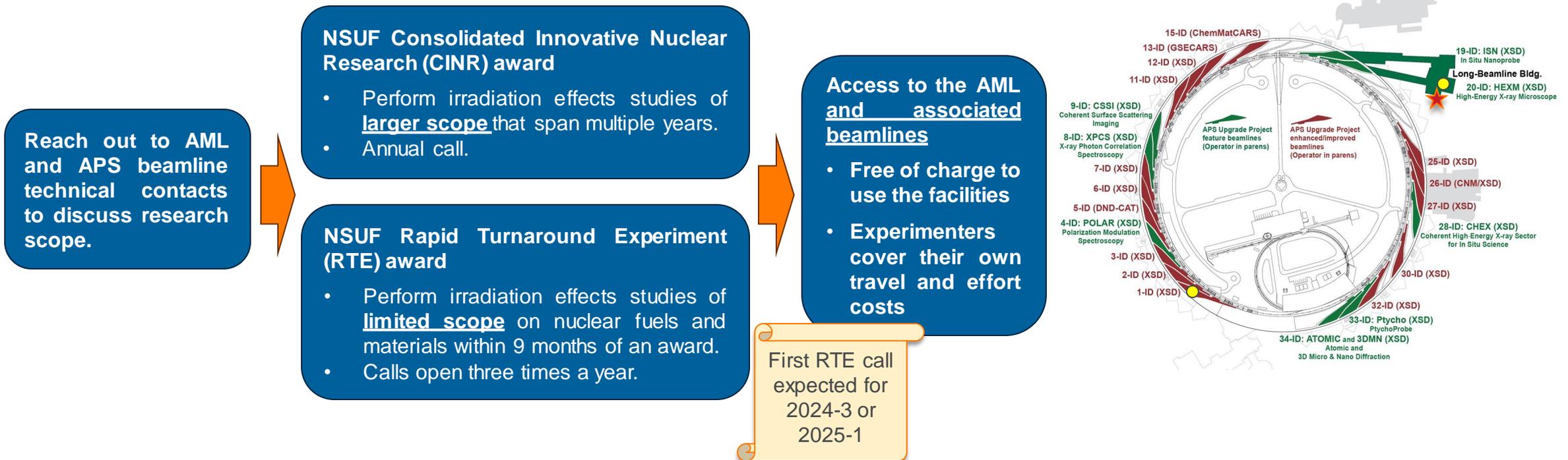
■ Sample storage

- AML will provide users short-term storage options before and after the experiment.
- Shielded, lockable storage cabinets are installed in the AML to provide adequate safety and security to prevent removal or use by unauthorized personnel.
- AML staff will be responsible for sample inventory and sample retrieval



AML ACCESS

NSUF research supports DOE-Office of Nuclear Energy’s missions. Most of the research looks at either **understanding the mechanisms of radiation on materials and fuels** to address the challenges of the current fleet of reactors, or looks at materials and fuels for the next generation. – NSUF website

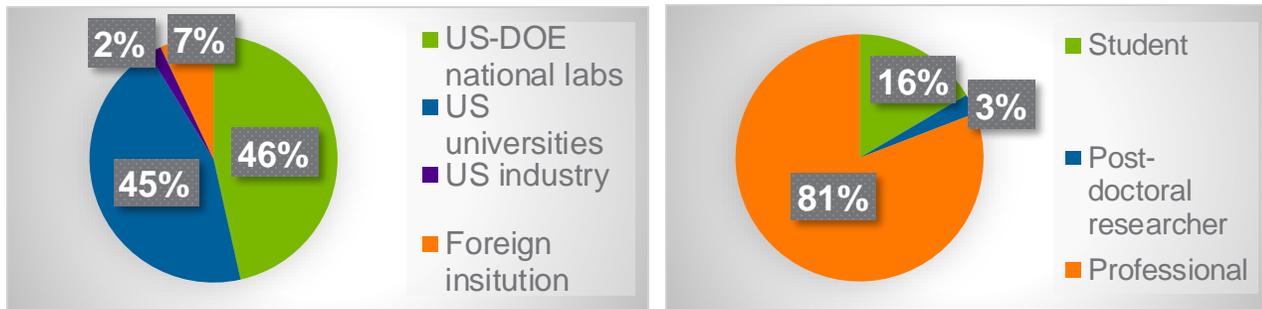


- This process bypasses the APS General User Proposal (GUP) process.
- Users with activated materials can still go through the APS GUP process and the necessity of utilizing the AML can be evaluated on a case-by-case basis.

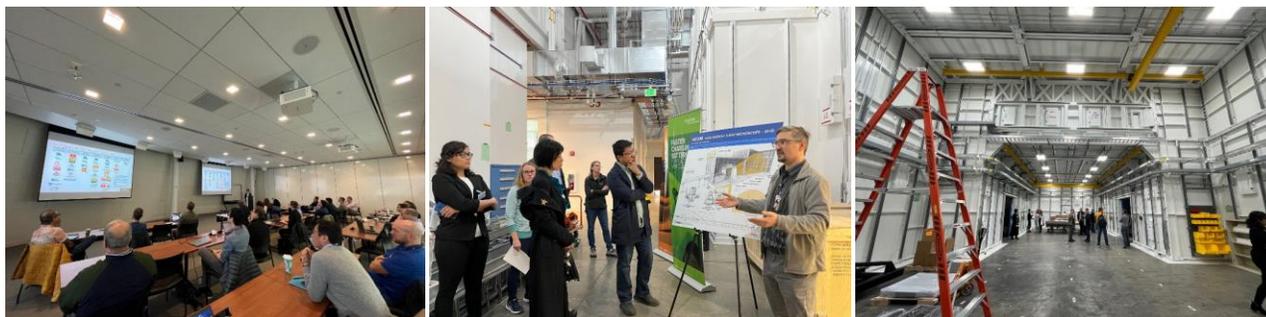
ACTIVITIES – FY24

- **AML-APS hybrid workshop, Nov. 7 & 8, 2023**

- 1.5-day technical talks, 0.5-day facility tour
- 115 participants:



- A complete report is available to share.
- Gathered valuable community inputs
- Highlights in photos

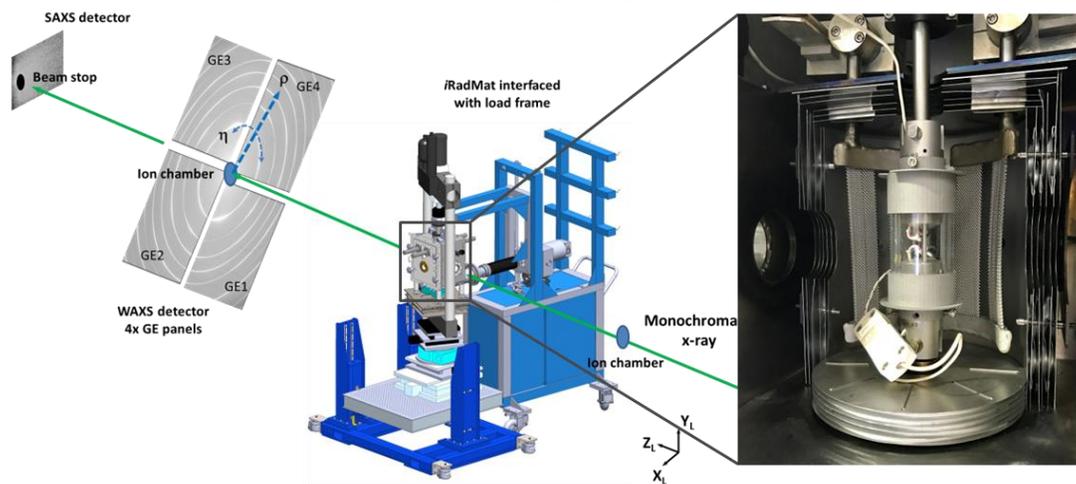


- **Awards (various sponsors)**

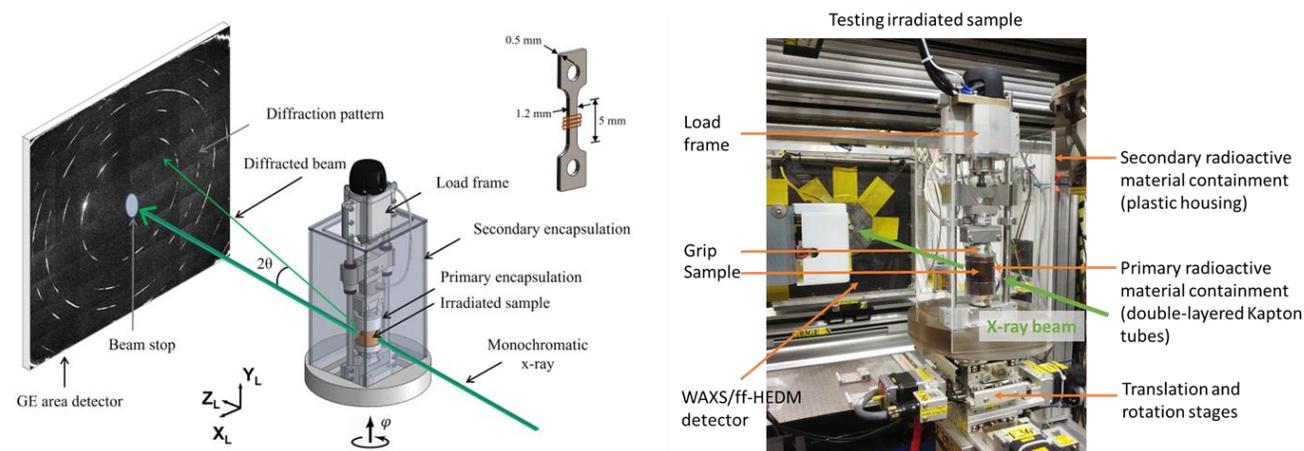
Sponsor	Institution	Title	Scope
ANL LDRD	ANL	Simulation-guided high-energy x-ray diffraction experiment framework for understanding irradiation-induced slip anisotropy in structural metals	Establish a technical framework to integrate High-Energy BCDI as a zoom-in companion to HEDM, and enable near-real-time steering of <i>in-situ</i> experiments to study deformation in n-irradiated stainless steels (3-year project)
DOE-NE, NSUF (FY24 CINR)	UIUC; U Alabama; ORNL; ANL	Characterization of Irradiation-Assisted Stress Corrosion Cracking in 316 Stainless Steel Baffle-Former Bolts Harvested from a Commercial Pressurized Water Reactor	PIE on PWR baffle-former bolts in studying IASCC with synchrotron x-ray techniques and electron microscopy (3-year project)

In situ X-ray diffraction on n-irr samples with mechanical loading

iRadMat apparatus
(local shielding, high T, vacuum)



Compact load frame
(room-temperature, on rotation stage)



- Lists of previous experiments on neutron-irradiated tensile specimens tested *in-situ* at the APS:

Material	Sample geometry	Neutron irradiation condition	Experiment	Publication
Fe-9%Cr	SS-J2	300°C, 0.01 dpa; 450°C, 0.01 dpa; Advanced Test Reactor, INL	In-situ tensile at room temperature, WAXS, SAXS	[1]
HT-UPS stainless steel	SS-J3	400°C, 3 dpa; Advanced Test Reactor, INL	In-situ tensile at room temperature and 400°C, WAXS, SAXS	[2]
316 stainless steel	SS-3	320°C, 5.5 dpa & 11.8 dpa; BOR-60 Reactor, Russia	In-situ tensile at room temperature, WAXS, SAXS	[3]
Fe-9%Cr	SS-J2	300°C, 0.1 dpa; 450°C, 0.1 dpa; Advanced Test Reactor, INL	In-situ tensile at room temperature, WAXS, HEDM	[4]

[1] X. Zhang, M. Li, J.-S. Park, P. Kenesei, J. Almer, C. Xu, J.F. Stubbins, Acta Materialia 126 (2017) 67-76.

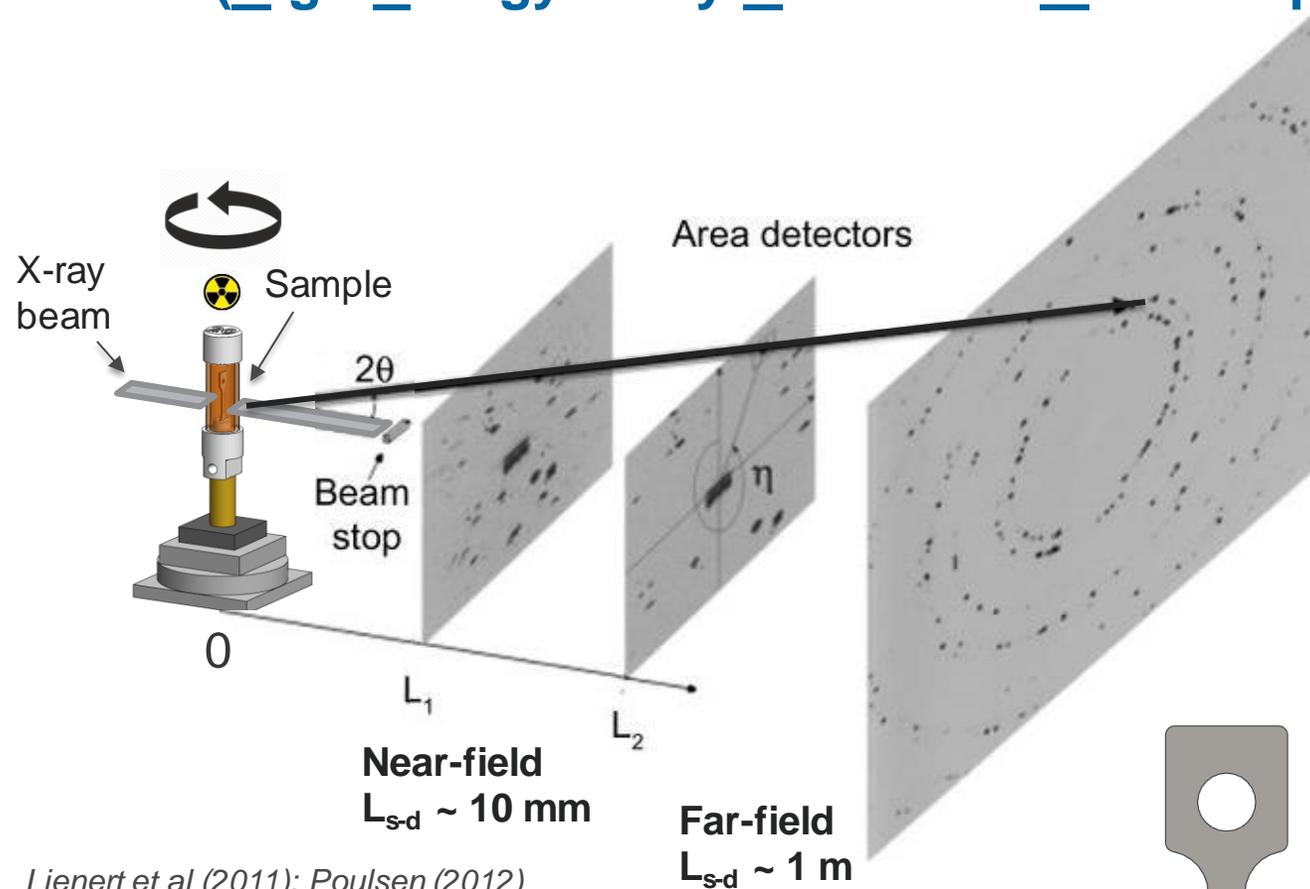
[2] C. Xu, X. Zhang, Y. Chen, M. Li, J.-S. Park, P. Kenesei, J. Almer, Y. Yang, Acta Materialia 156 (2018) 330-341.

[3] X. Zhang, C. Xu, Y. Chen, W.-Y. Chen, J.-S. Park, P. Kenesei, J. Almer, J. Burns, Y. Wu, M. Li, Acta Materialia 200 (2020) 315-327.

[4] E. Mengiste, D. Piedmont, M. Messner, M. Li, J. Stubbins, J.-S. Park, X. Zhang, M. Kasemer, Acta Materialia, 263 (2024) 119503

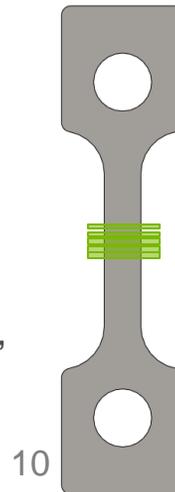
3D/4D Characterization of Radioactive Samples

HEDM (High-Energy X-ray Diffraction Microscopy)



Lienert et al (2011); Poulsen (2012)

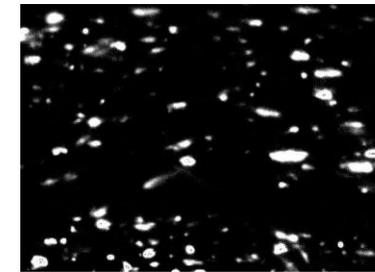
Both HEDM techniques measure the sample in “layers” to cover large volume.



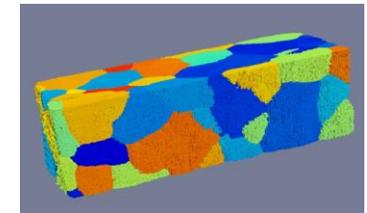
Near-field HEDM (nf-HEDM):

- Reconstructs grain morphology and crystallographic orientation **voxel by voxel** (1-2 μm resolution).

Detector images as sample rotates:



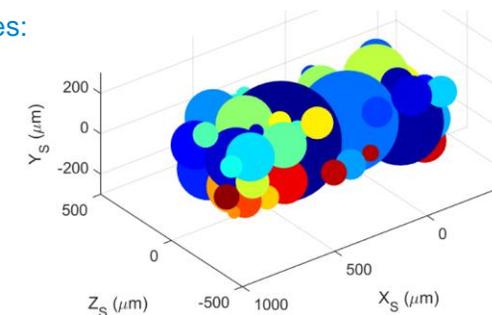
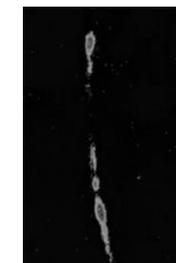
EBSD-like reconstruction



Far-field HEDM (ff-HEDM):

- Reconstructs centroids, crystallographic orientations, and **elastic strain tensors domain by domain** (10 μm resolution).

Detector images as sample rotates:



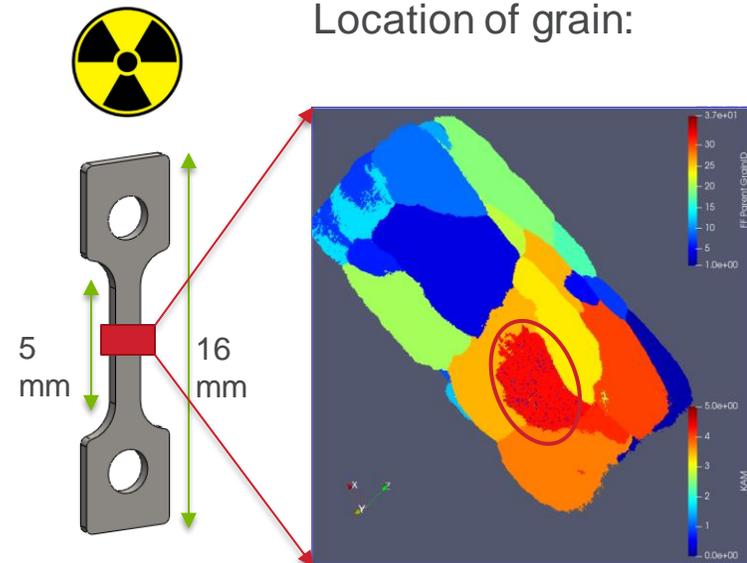
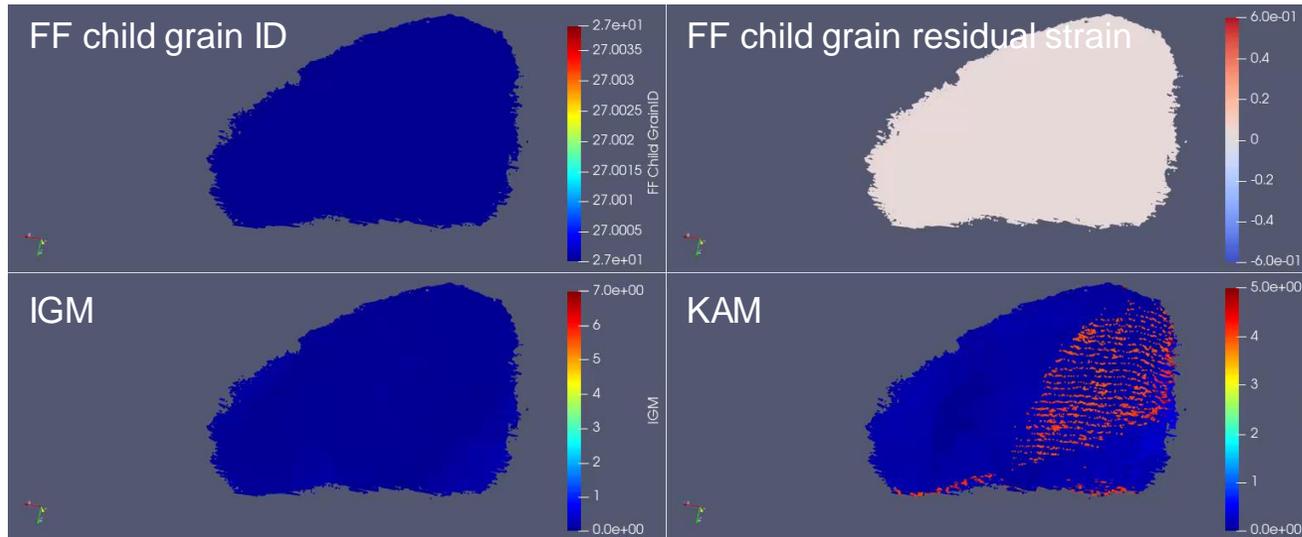
INDIVIDUAL GRAIN EVOLUTION IS VISUALIZED IN 3D

0.1 DPA AT 450°C

Publication in preparation

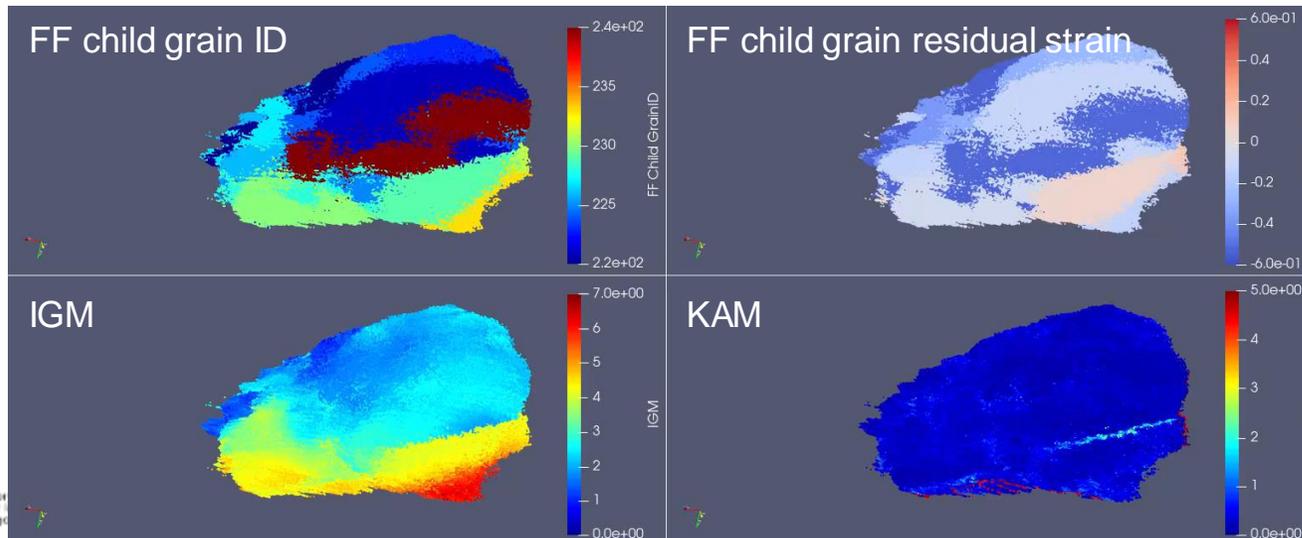
Undeformed

- Radius = 57 μm
- 1 child grain

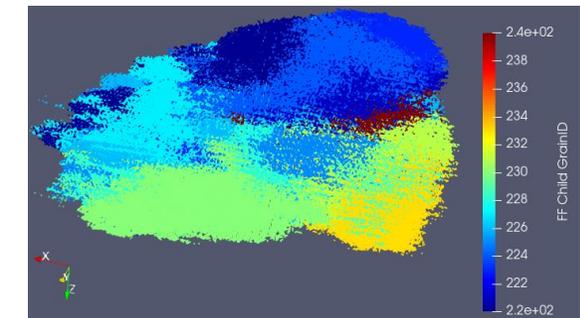


Deformed

- Radius = 44 μm
- 13 child grains



Cross-section view of grain:



APS Upgrade (APS-U)

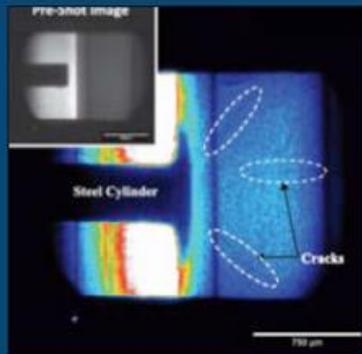
- APS dark period: 2023-04-17 to 2024-04 (just ended!)

The upgraded APS will be the world's brightest storage ring light source, exceeding today's capabilities by 2-3 orders of magnitude in brightness, coherence and nano-focused flux.

High Energy

Penetrating bulk materials and operating systems

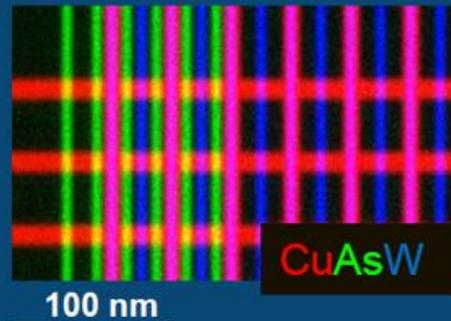
- World-class brightness for hard x-rays
- 3D mapping deep inside samples
- X-ray cinematography in previously inaccessible regimes



Brightness

Providing macroscopic fields of view with nm-scale resolution

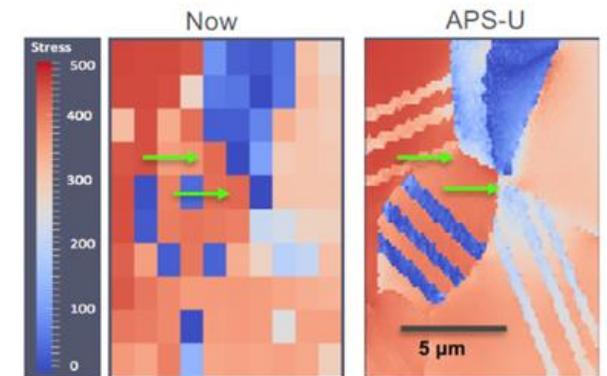
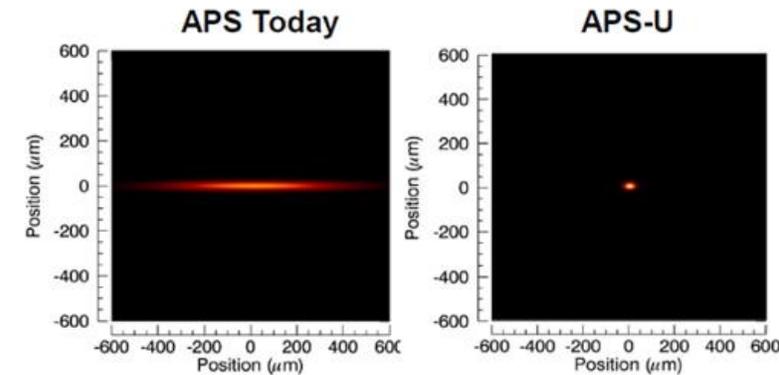
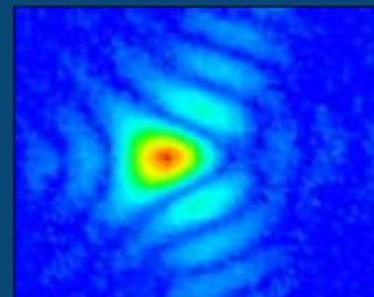
- Multi-scale imaging connecting nanometer features across macroscopic dimensions
- Fast sampling with chemical, magnetic, electronic sensitivity



Coherence

Enabling highest spatial resolution even in non-periodic materials

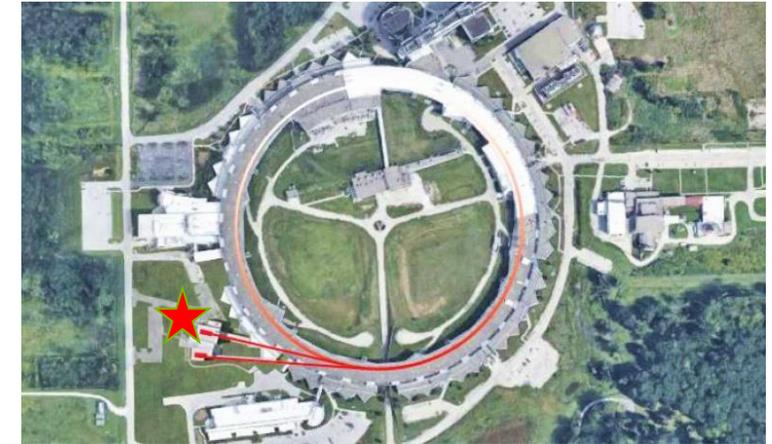
- Extends lens-less imaging to hard x-ray domain, with resolution down to <1 nm, localizing atoms
- Increases phase contrast for fast full-field imaging
- Correlation methods improve by 10,000x-1,000,000x



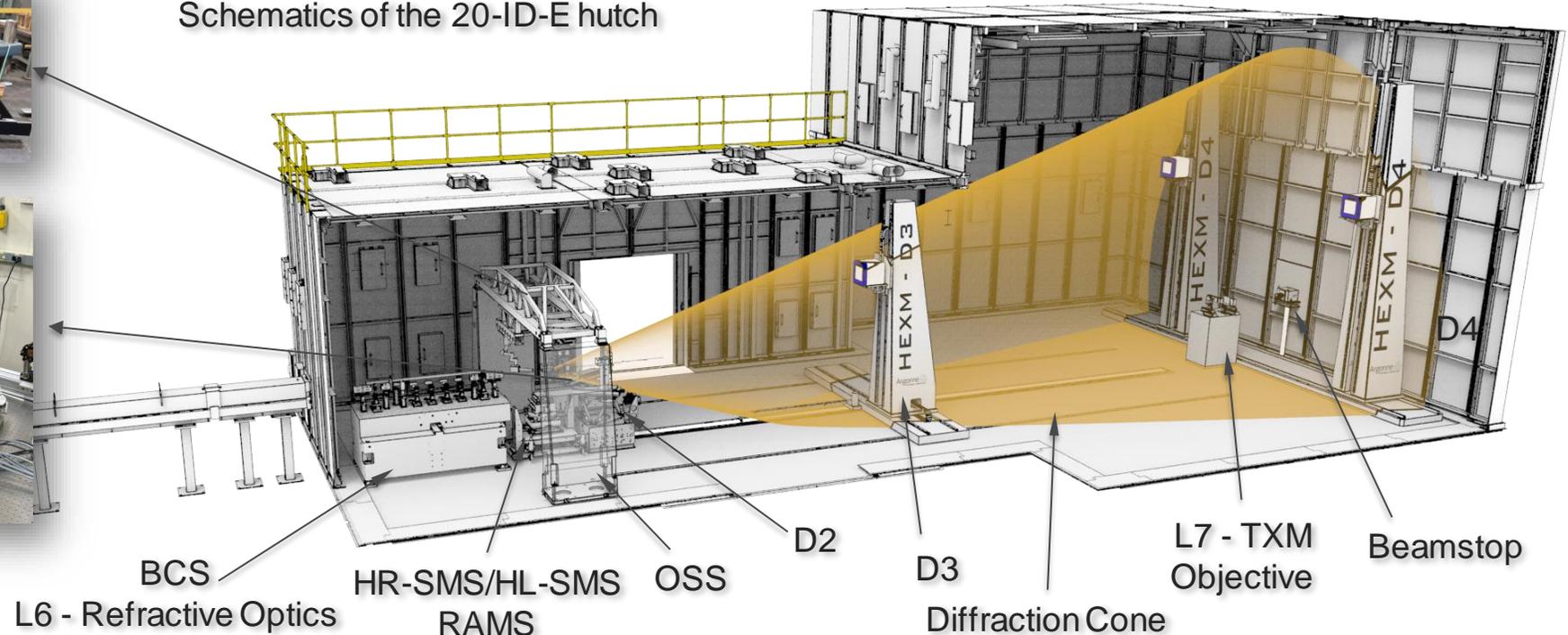
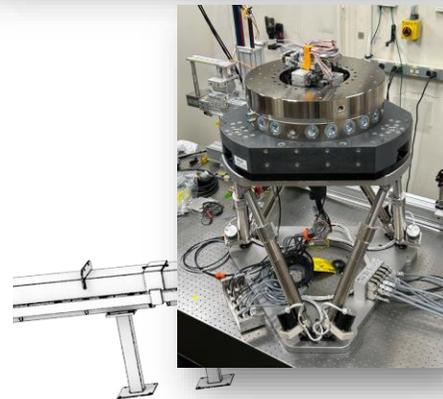
Simulation of Ni-superalloy grains under fatigue deformation; arrows show crack initiation at current (left) APS-U (right) resolutions. Courtesy M. Sangid (Purdue U).

HIGH-ENERGY X-RAY MICROSCOPE (HEXM) @ 20-ID

- One of two 'long beamlines' under APS-U
- A world-leading, high-energy (40-120 keV), multiscale X-ray imaging beamline.
- Two new white-beam hutches, at nominal distances **D@70 m** and **E@180m**
- Activated Materials Lab next to 20-ID-E (NSUF/DOE-NE)

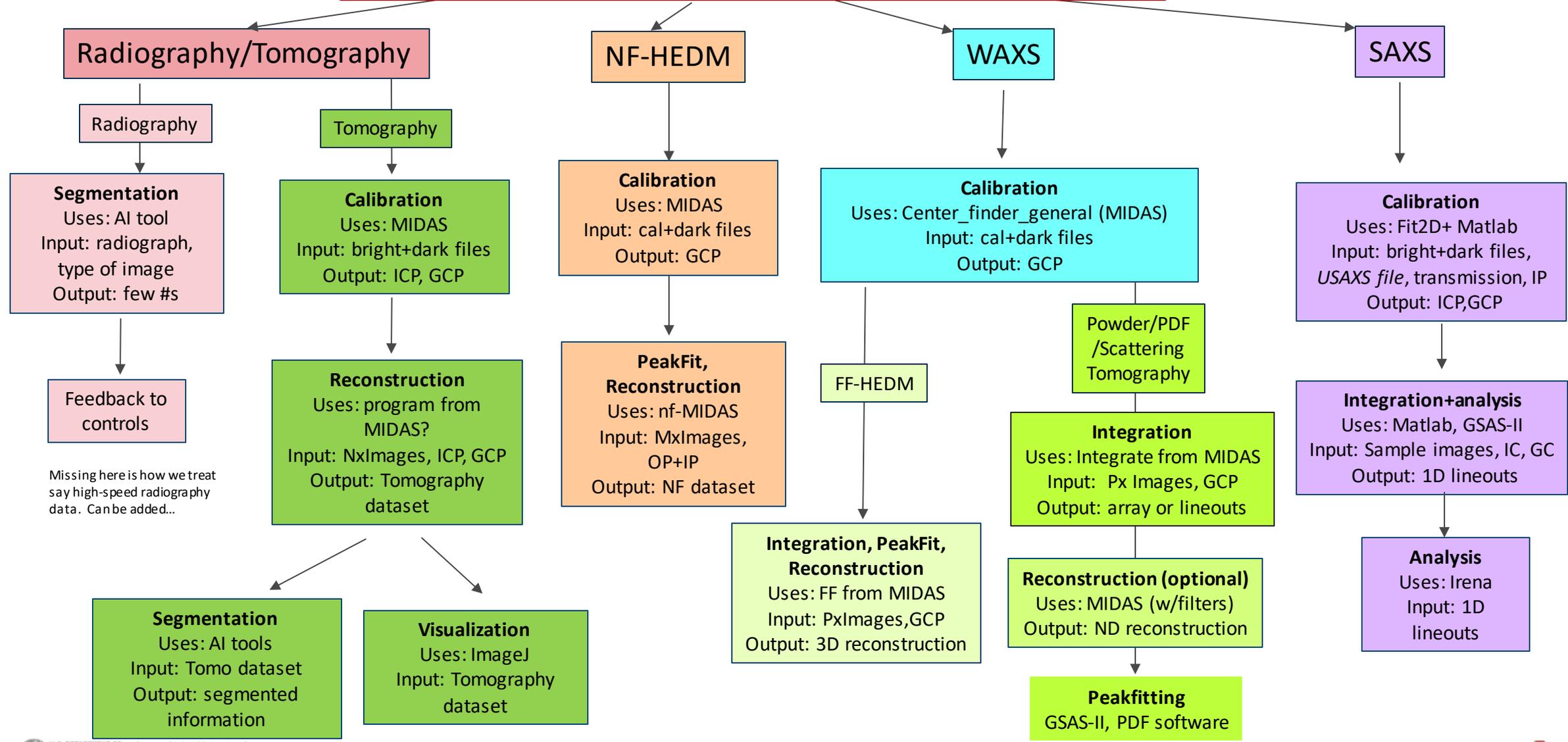


Schematics of the 20-ID-E hutch

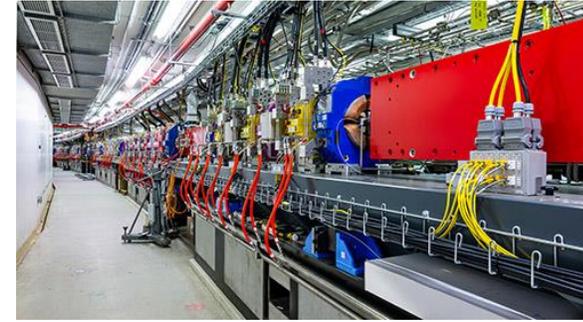


DATA FLOW for HEXM & 1-ID USERS

2D Images from AreaDetector saved as HDF5 plus metadata*

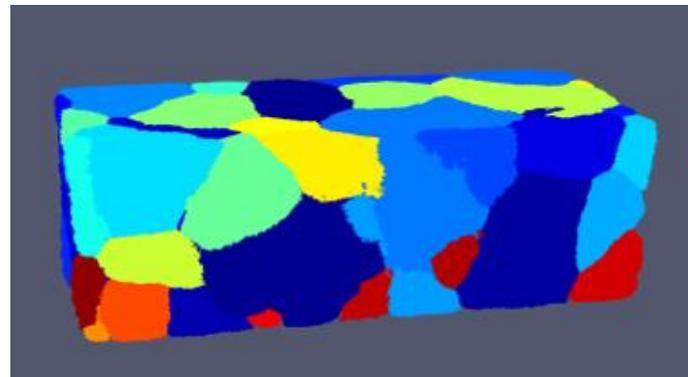
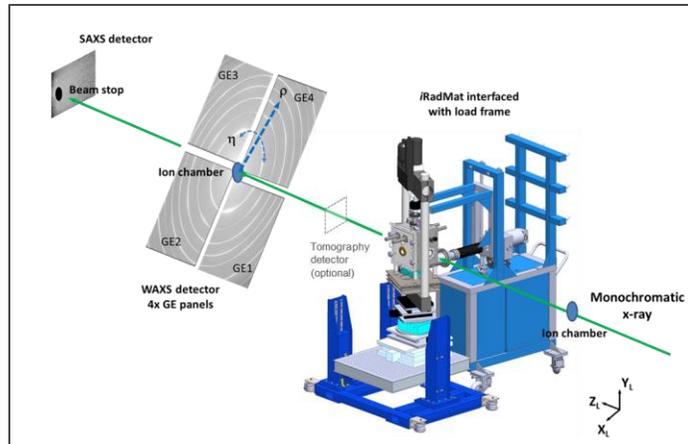


APS-U & BEAMLINe STATUS



- New storage ring being commissioned with stored electrons on April 10; the commissioning phase takes ~ 2 months.
- Assuming no issues, radiation shielding verification will take place shortly thereafter for all ready beamlines (will include 1-ID; 20-ID/HEXM will take longer).
- Beamlines will undergo commissioning throughout summer 2024, availability for user operations will vary from summer 2024 to winter 2025.
- Ring current will increase from 25mA to 200mA in steps over course of next 1+ year.

SYNCHROTRON X-RAY TECHNIQUES FOR NUCLEAR MATERIALS



Capabilities:

1. *In situ* dynamic measurements

- *In-situ* observation of materials response to external stimuli
- Complex sample environments (loading, heating, corrosion)

2. Non-destructive 3D characterization

- Mapping microstructural and micro-mechanical heterogeneity at grain level

3. Multiple length scales in one experiment

- From macro to meso to micro, with different modalities, all in one experiment

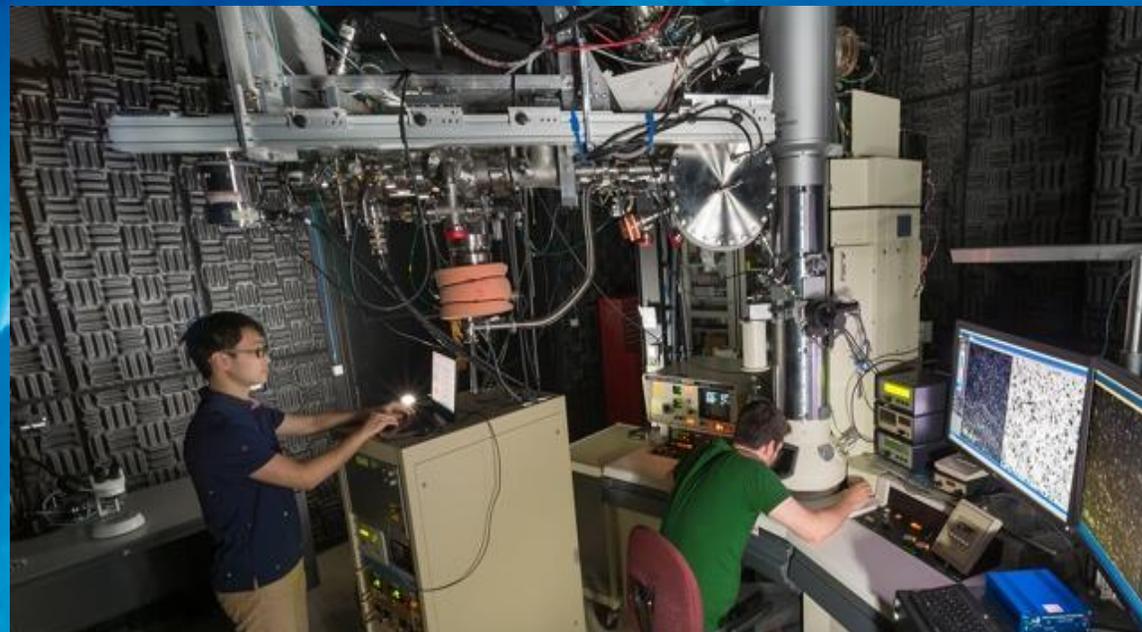
Advantages:

- Minimum sample preparation (friendly to radioactive samples)
- Sampling mm^3 volume (great statistics)
- Comprehensive sample environment
- Direct structure-property correlation
- **Real materials, real environments, non-destructive, *in-situ*, 3D**

Argonne's NSUF Partner Facilities



Activated Materials Laboratory (AML)



Intermediate Voltage Electron Microscope (IVEM)

U.S. DEPARTMENT OF
ENERGY

Office of
NUCLEAR ENERGY