2024 NUCLEAR SCIENCE USER FACILITIES USERS ORGANIZATION ANNUAL MEETING

ACTIVATED MATERIALS LABORATORY AT THE UPGRADED APS

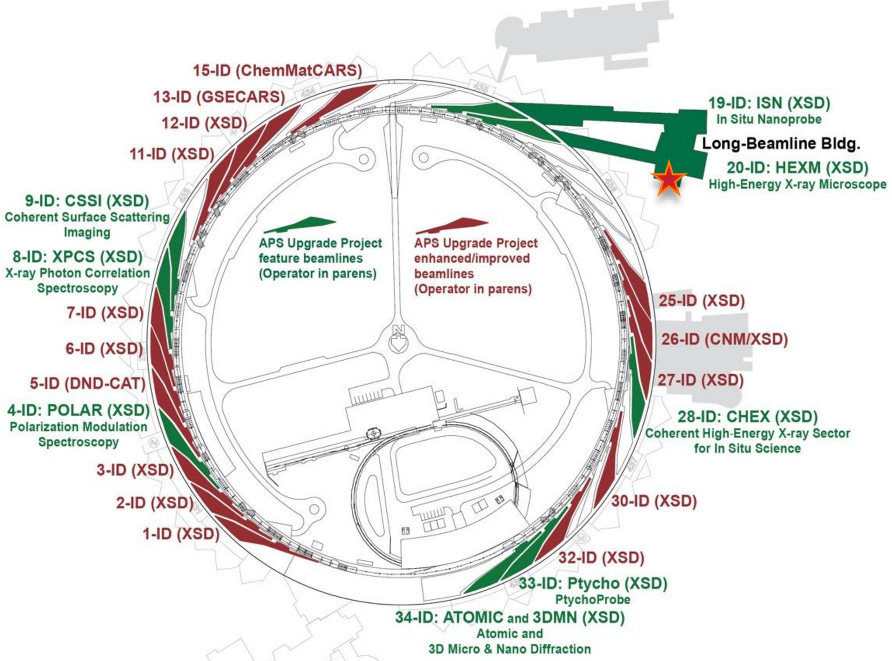
XUAN ZHANG, JONATHAN ALMER, MEIMEI LI
Argonne National Laboratory

Argonne NATIONAL LABORATORY

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
- AML user access: 2024 fall



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

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

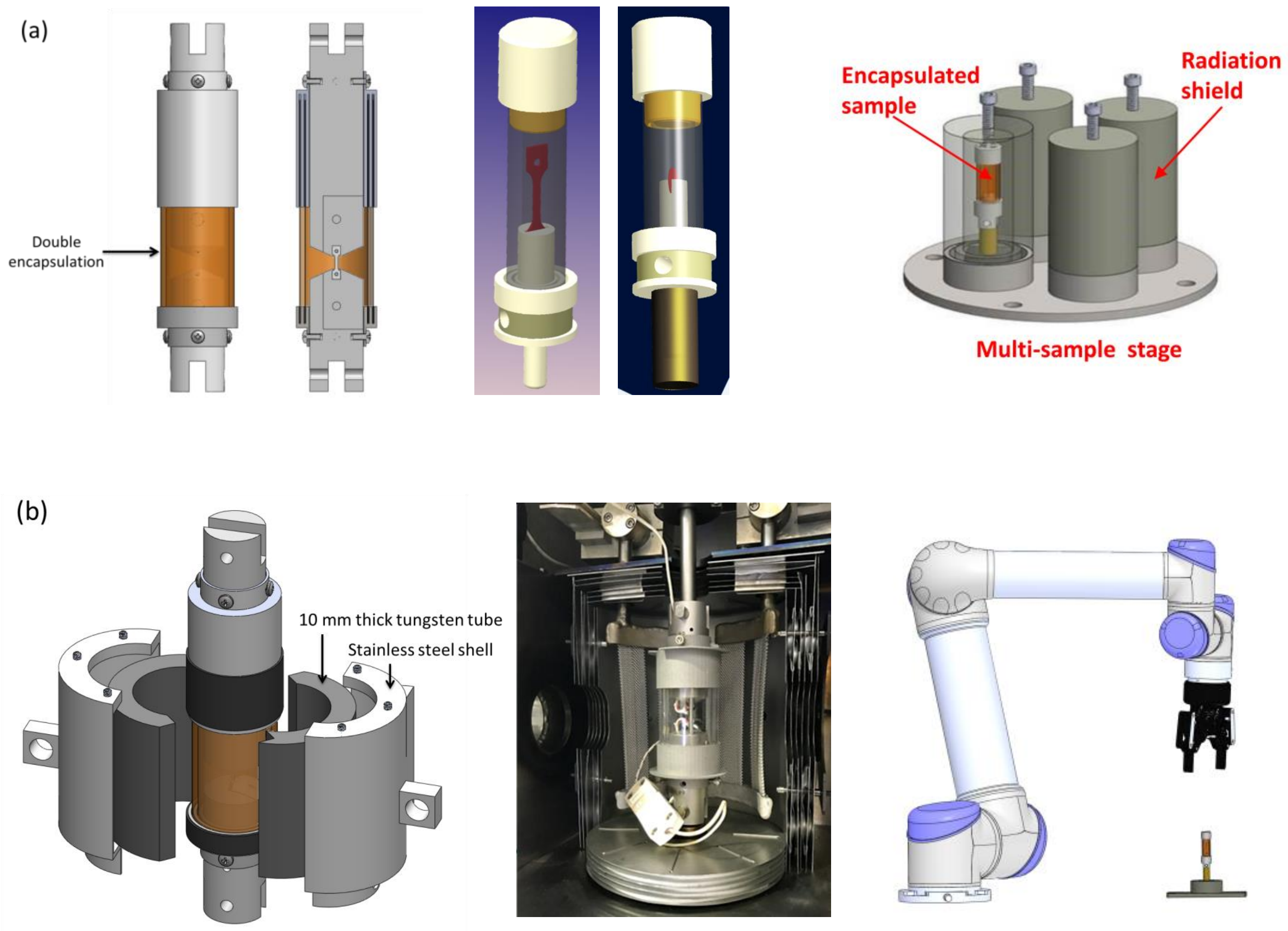
- 2 lead-shielded storage cabinets
- 2 lead-shielded rolling barriers
- 1 lead-shielded mobile cart

- 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

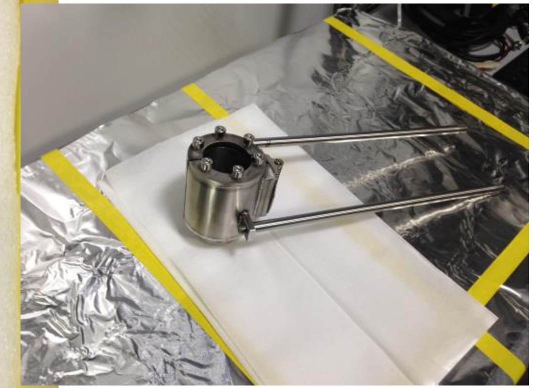
RAD SAMPLE ENCAPSULATION & SHIELDING

- 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 PREPARATION AND STORAGE

- **Sample preparation**
  - 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*

Reach out to AML and APS beamline technical contacts to discuss research scope.

NSUF Consolidated Innovative Nuclear Research (CINR) award

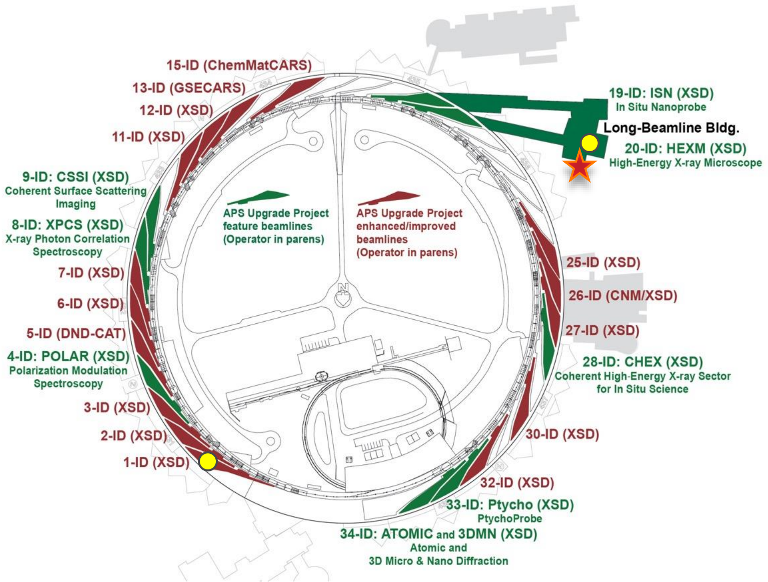
- Perform irradiation effects studies of **larger scope** that span multiple years.
- Annual call.

NSUF Rapid Turnaround Experiment (RTE) award

- Perform irradiation effects studies of **limited scope** on nuclear fuels and materials within 9 months of an award.
- Calls open three times a year.

Access to the AML and associated beamlines

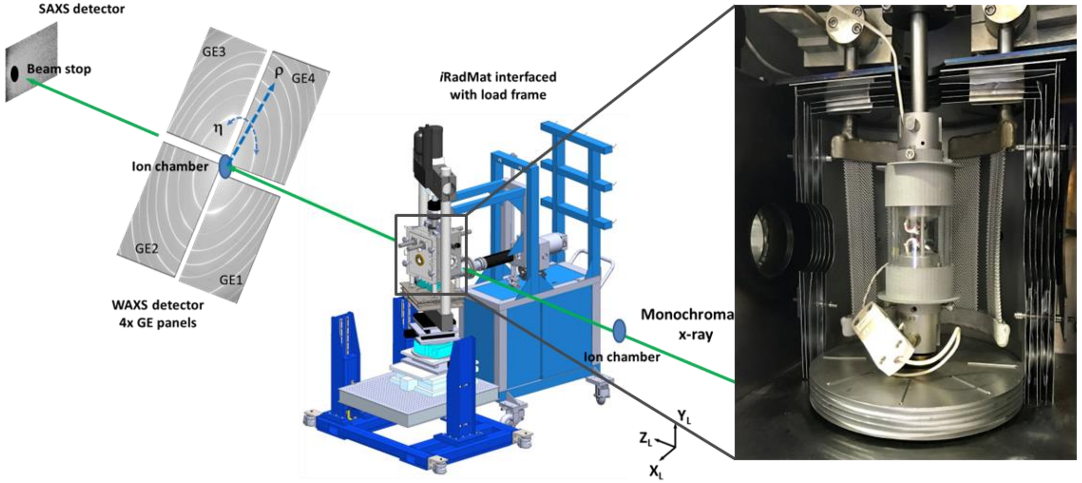
- **Free of charge to use the facilities**
- **Experimenters cover their own travel and effort costs**



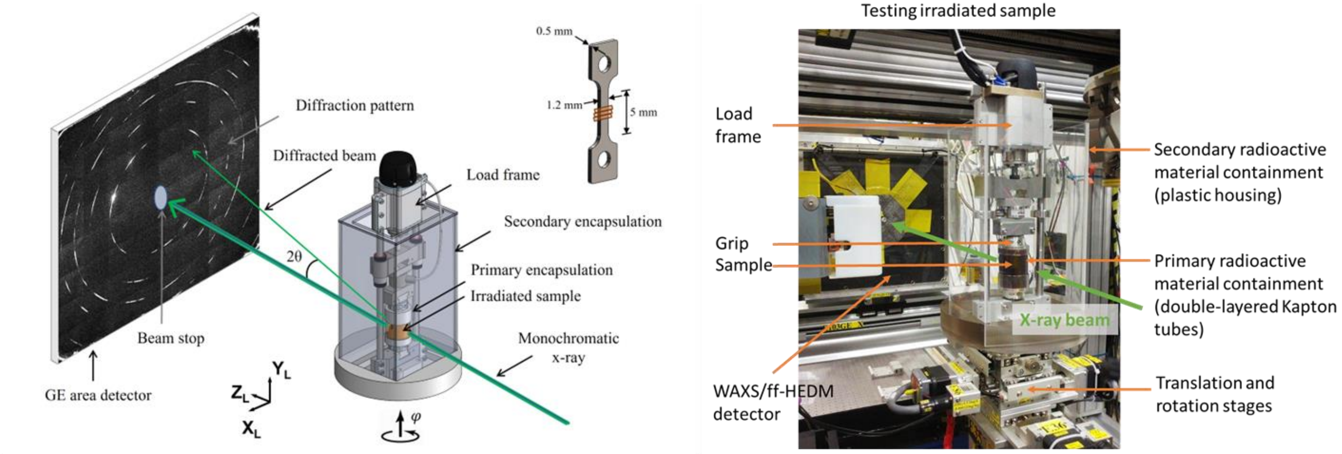
- **This process bypasses the APS General User Proposal (GUP) process.**
- **Users with activated materials can still go through the APS GUP process and the necessity of utilizing the AML can be evaluated on a case-by-case basis.**

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

iRadMat apparatus
(local shielding, high T, vaccum)



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



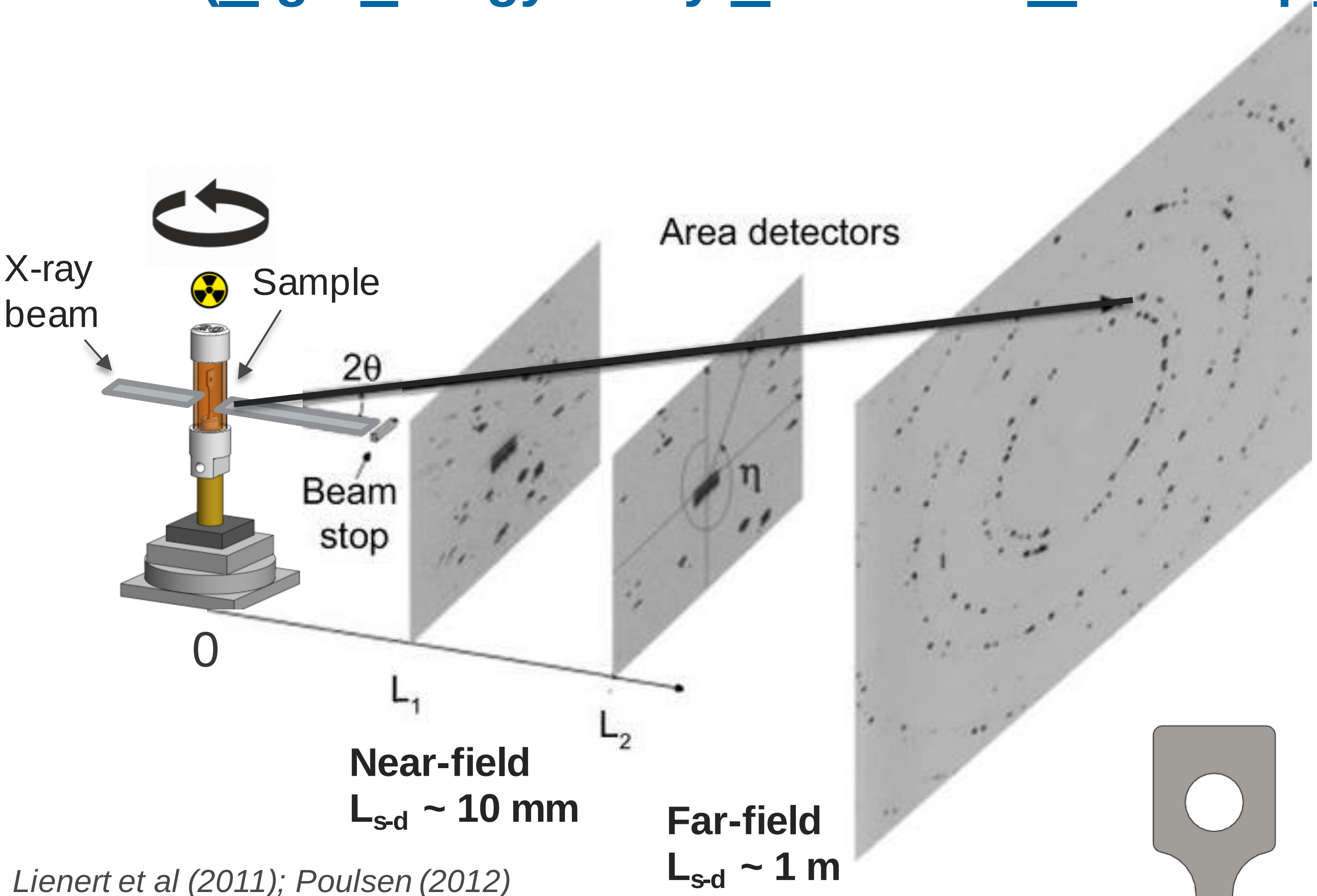
- **Lists of previous experiments on neutron-irradiated tensile specimens tested *in-situ* at the APS:**

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

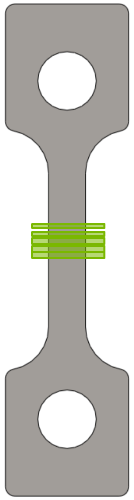
3D/4D Characterization of Radioactive Samples

HEDM (High-Energy X-ray Diffraction Microscopy)



Lienert et al (2011); Poulsen (2012)

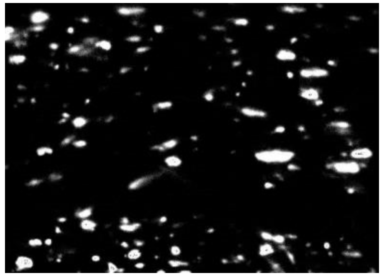
Both HEDM techniques measure the sample in "layers" to cover large volume.



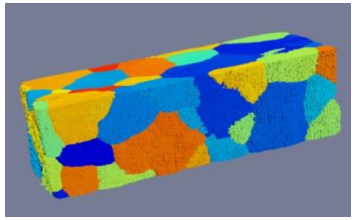
Near-field HEDM (nf-HEDM):

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

Detector images as sample rotates:



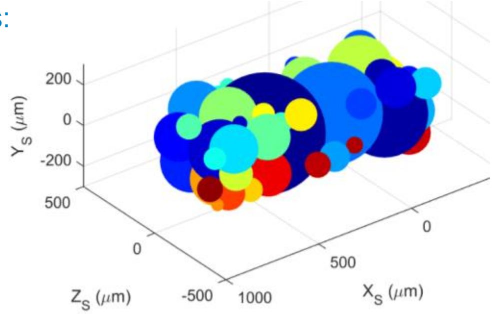
EBSD-like reconstruction



Far-field HEDM (ff-HDEM):

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

Detector images as sample rotates:



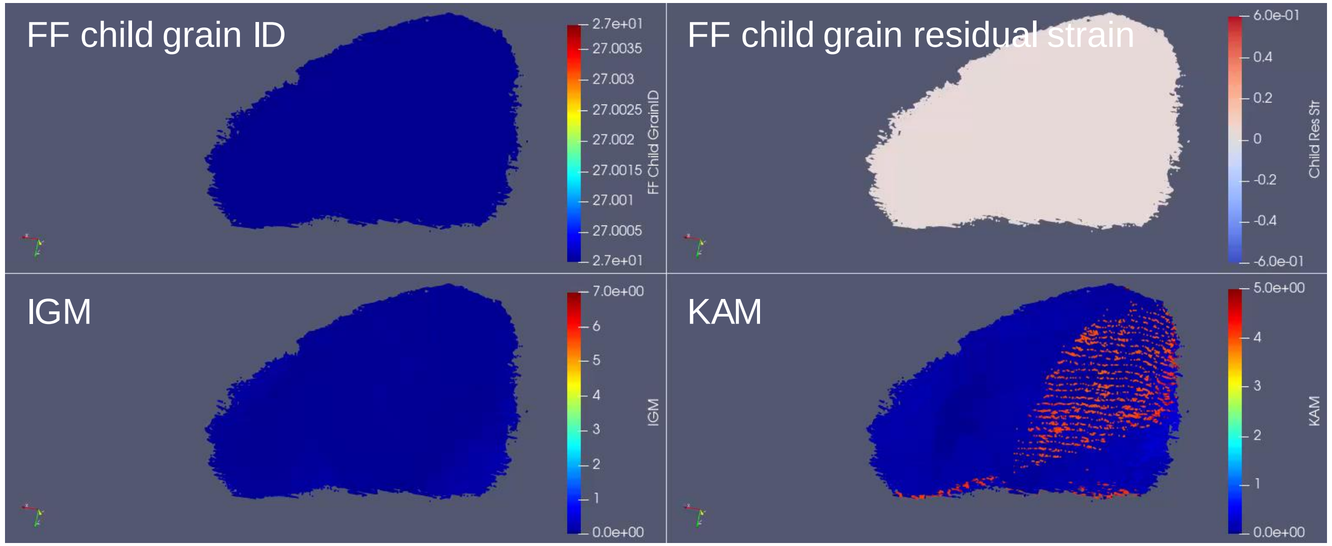
INDIVIDUAL GRAIN EVOLUTION IS VISUALIZED IN 3D

0.1 DPA AT 450°C

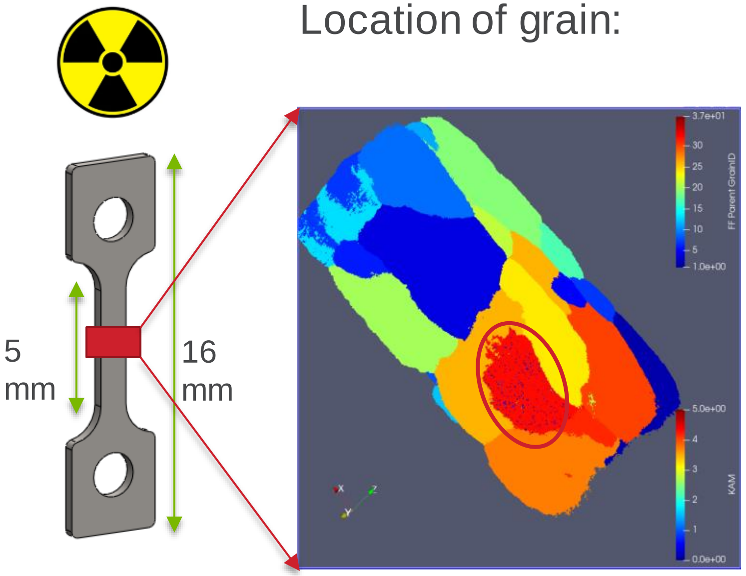
Publication in preparation

Undeformed

- **Radius = 57 µm**
- **1 child grain**

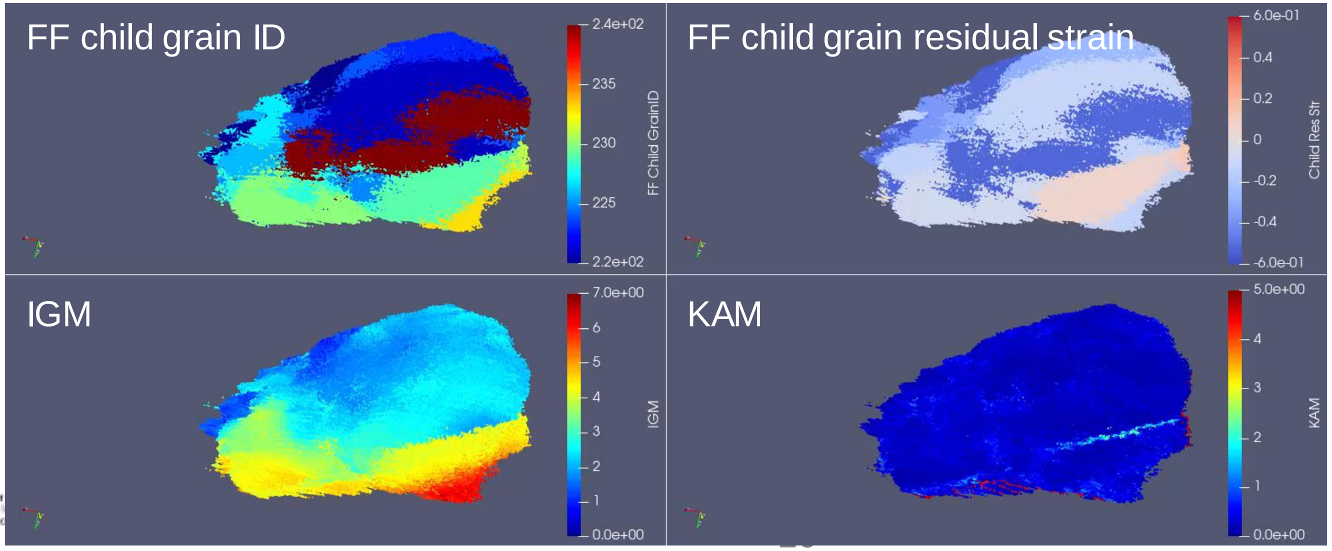


Location of grain:

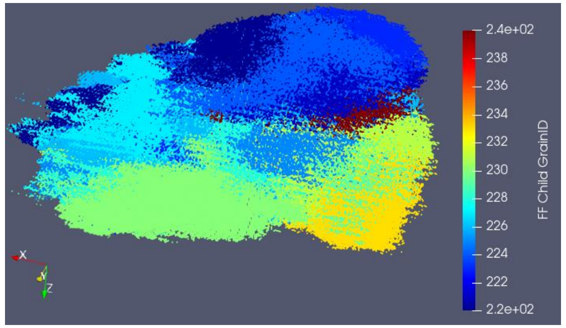


Deformed

- **Radius = 44 µm**
- **13 child grains**



Cross-section view of grain:



APS Upgrade (APS-U)

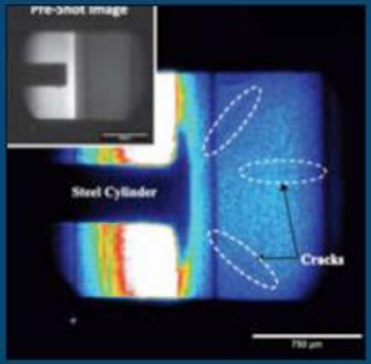
- APS dark period: 2023-04-17 to 2024-04

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

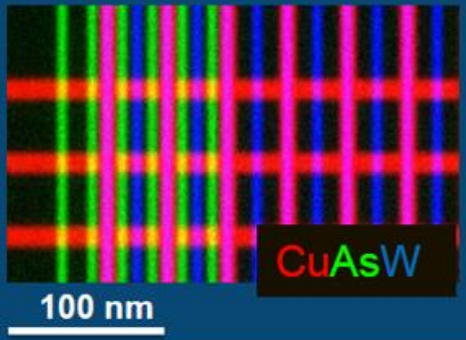
- World-class brightness for hard x-rays
- 3D mapping deep inside samples
- X-ray cinematography in previously inaccessible regimes



Brightness

Providing macroscopic fields of view with nm-scale resolution

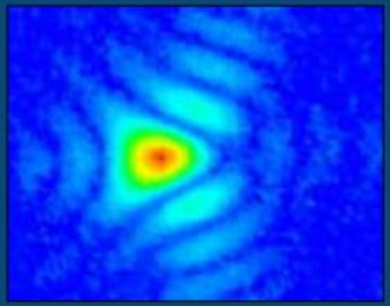
- Multi-scale imaging connecting nanometer features across macroscopic dimensions
- Fast sampling with chemical, magnetic, electronic sensitivity

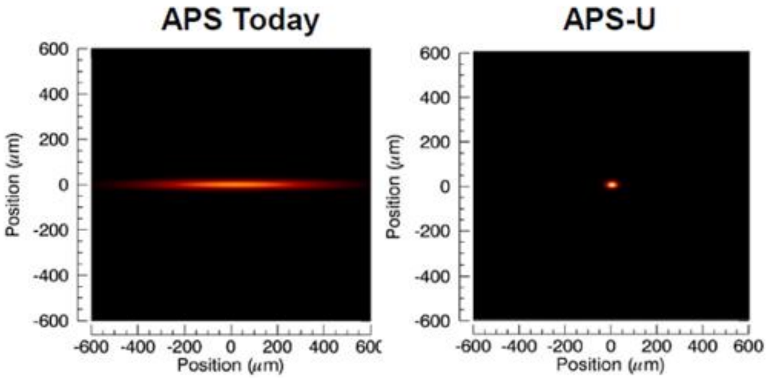


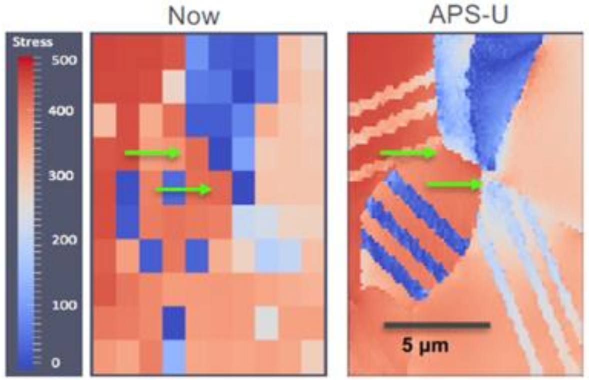
Coherence

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



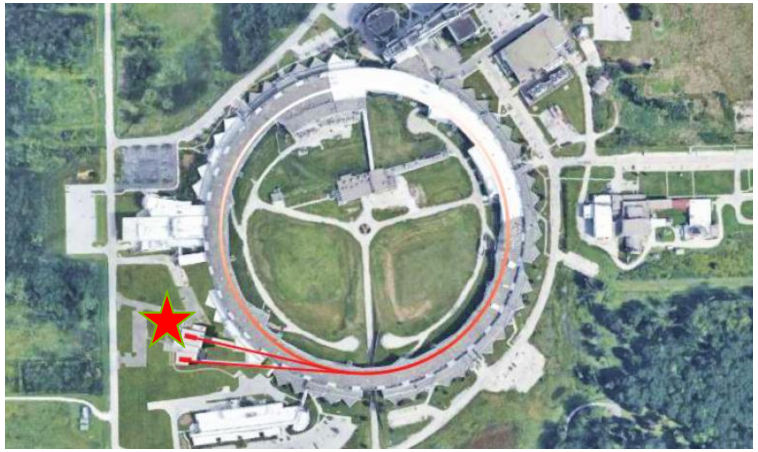


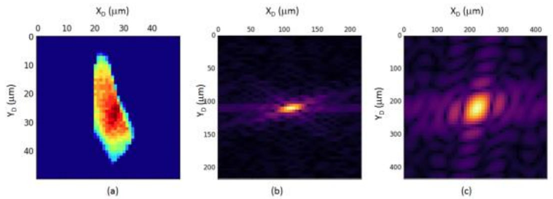


Simulation of Ni-superalloy grains under fatigue deformation; arrows show crack initiation at current (left) APS-U (right) resolutions. Courtesy M. Sangid (Purdue U).

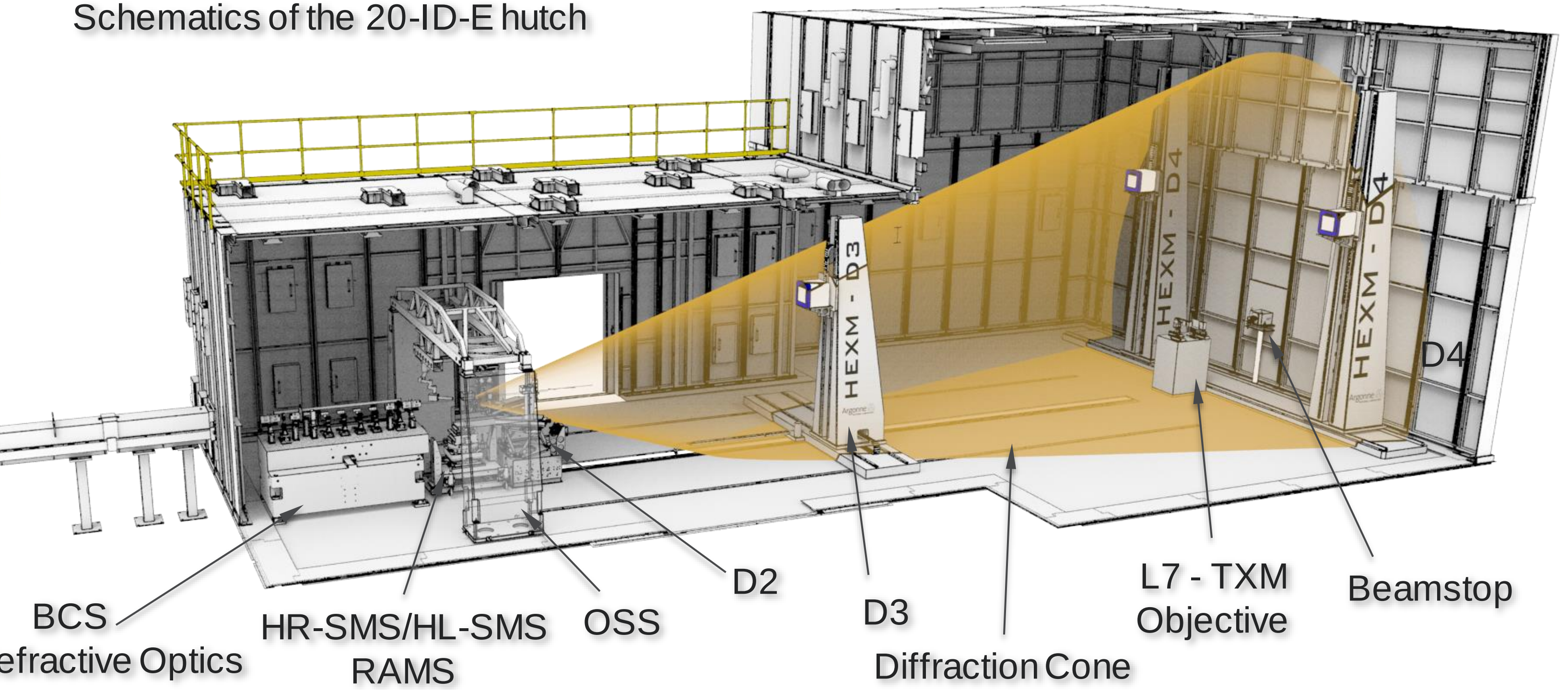
HIGH-ENERGY X-RAY MICROSCOPE (HEXM) @ 20-ID

- 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)

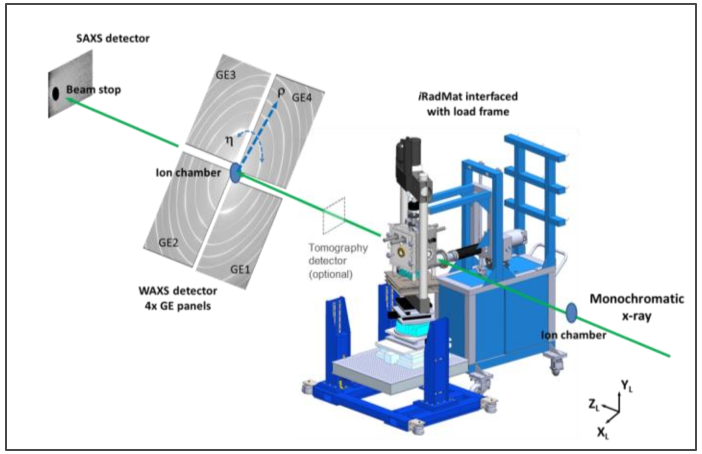


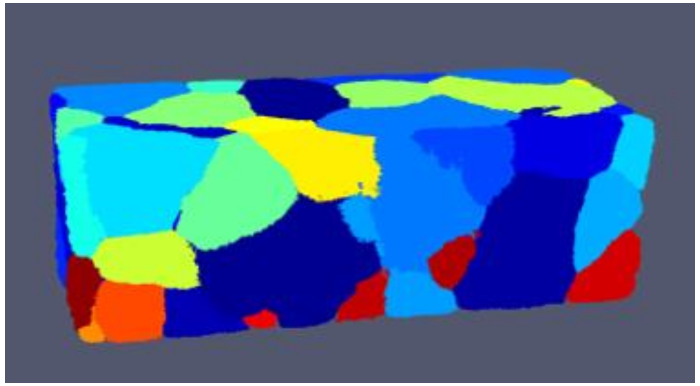


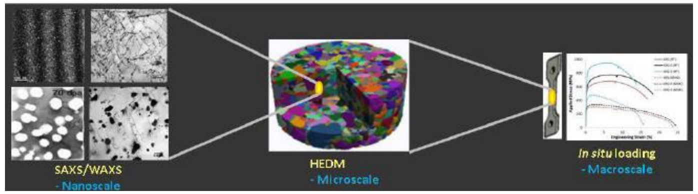
Schematics of the 20-ID-E hutch



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**

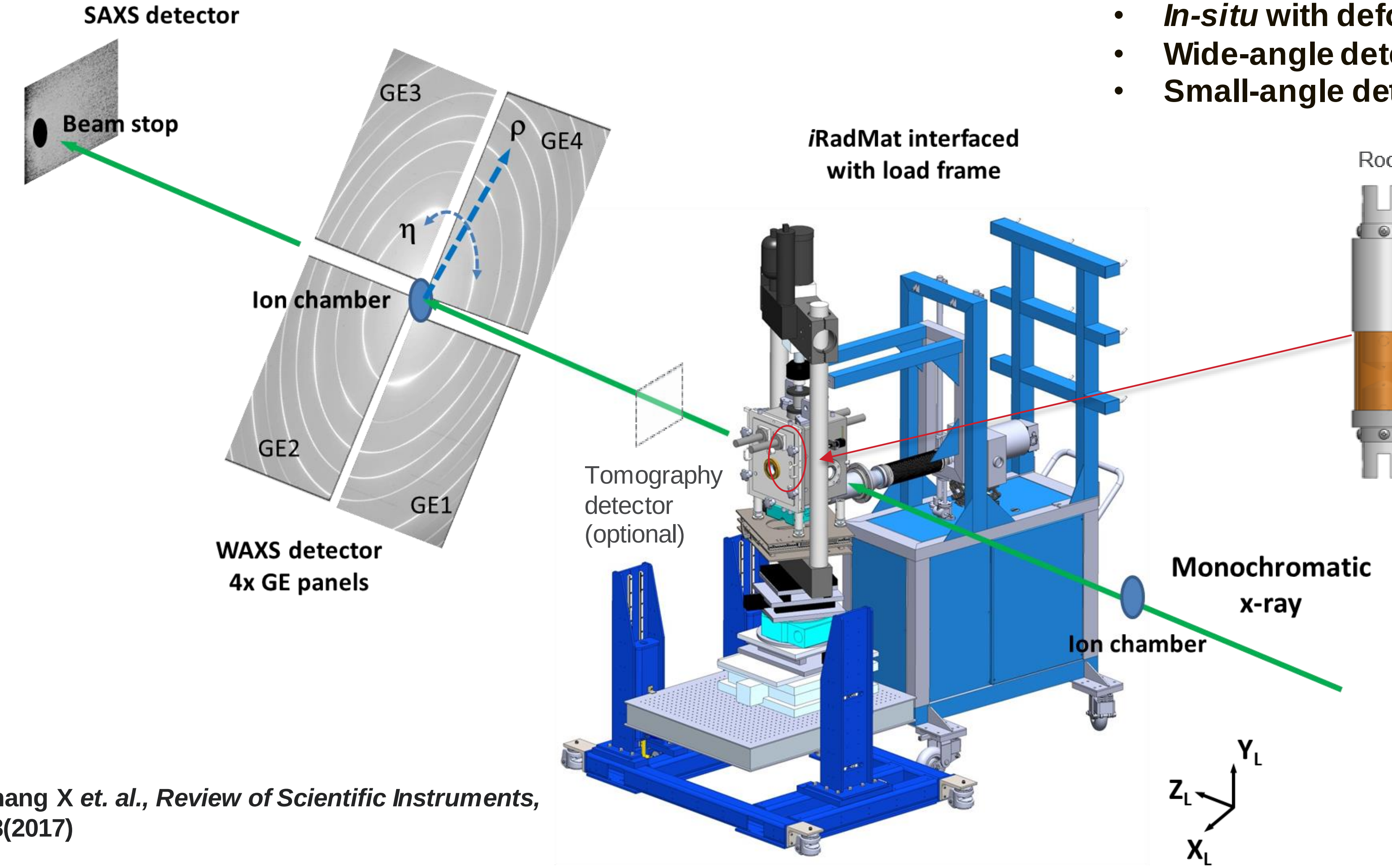
BACKUP SLIDES

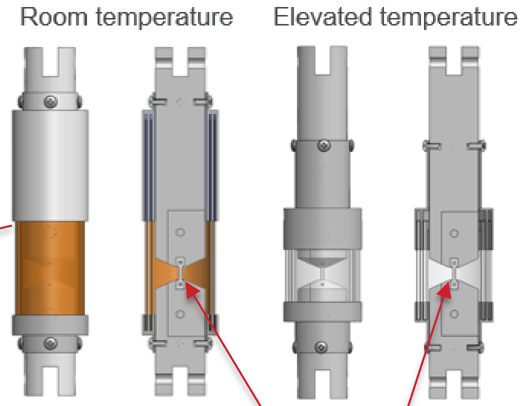
*I*RADMAT INTERFACED WITH BEAMLINE INFRASTRUCTURE

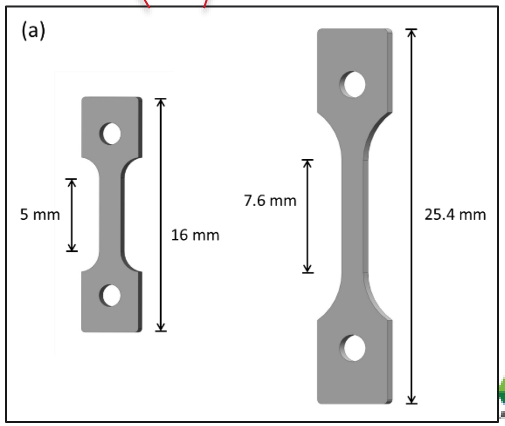
Beamline 1-ID-E @ the Advanced Photon Source

Simultaneous wide-angle x-ray scattering (WAXS) and small-angle x-ray scattering (SAXS):

- ***In-situ* with deformation.**
- **Wide-angle detector array** (1.0m - 4.5m downstream)
- **Small-angle detector** (6.6m downstream)







Zhang X *et. al., Review of Scientific Instruments,* 88(2017)

INHOMOGENEOUS DEFORMATION IN IRRADIATED SAMPLES

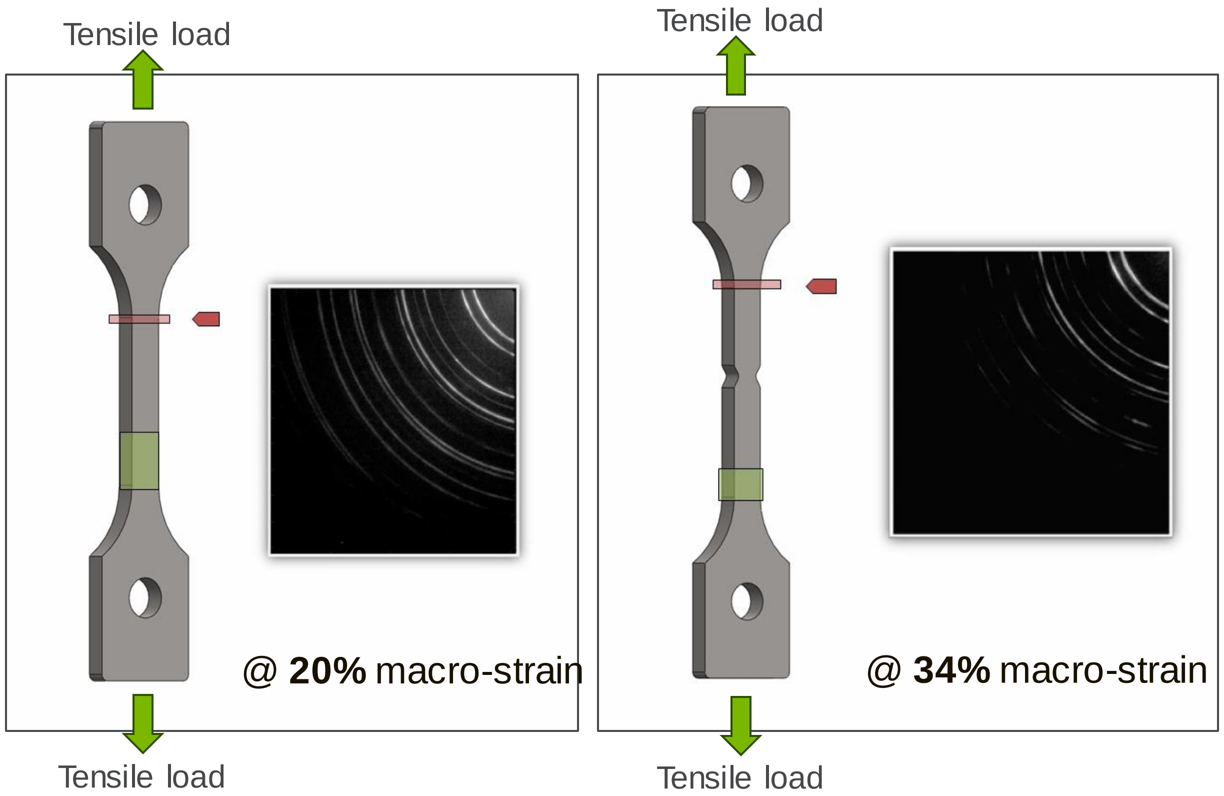
Material:
- Solution annealed 316 SS

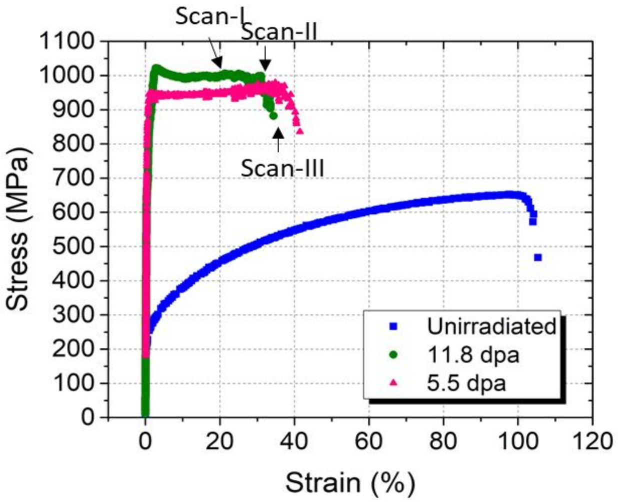
Specimen:
- Sheet-type tensile specimen (gauge 7.62x1.52x0.76 mm)

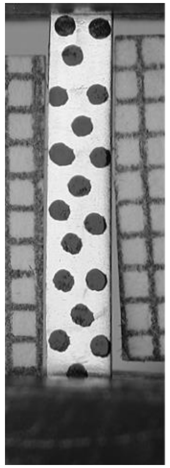
Neutron irradiation:
- 320°C to 10 dpa (NRC archive sample)

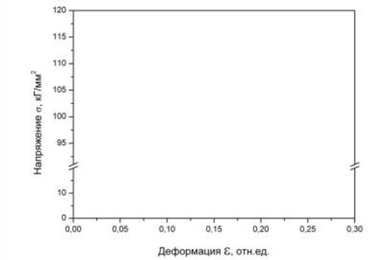
In situ X-ray test:
- Energy: 123 keV (0.01008 nm)
- Beam size: 100x100 μm
- Strain rate: 1-3x10^{-5}/s

In the 320°C,10 dpa irradiated sample:









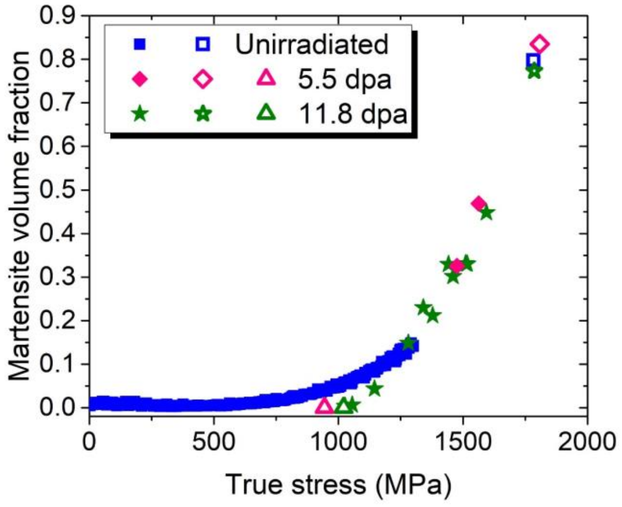
55 dpa, 310°C in BN-350, Russian analog to 321 SS, Room temperature test

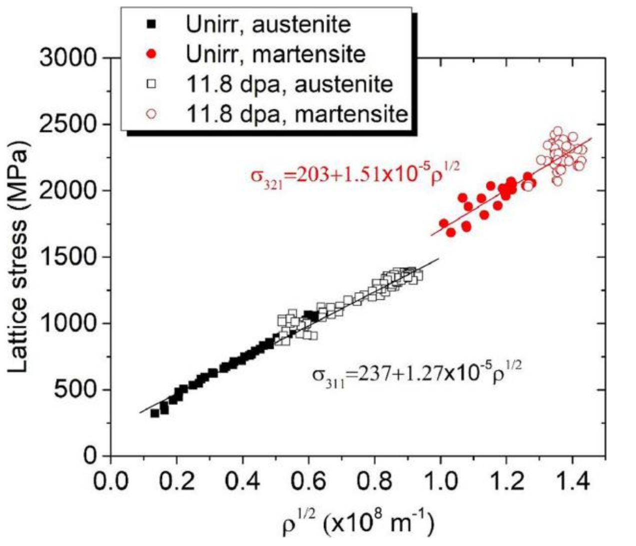
Animation from Dr. F. Garner; presented with permission.

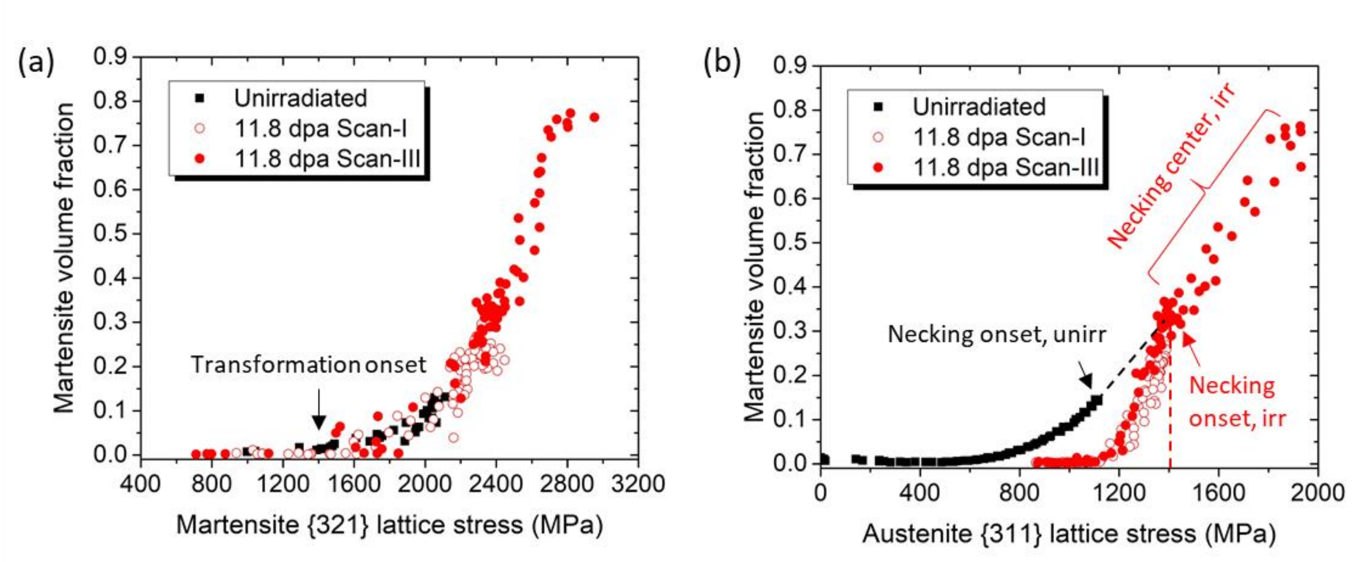
Observations:
- Deformation starts from the top and propagates to the bottom (**deformation wave**).
- Necking occurred before band fully propagating through.

AN INSIGHT TO DEFORMATION MECHANISM

Zhang X *et. al.*, *Acta Materialia,* 200(2020) 315







- **Things align!** The irradiation did not alter the dislocation hardening and the martensitic transformation mechanisms.
- **A magic 1400 MPa!** It is the stress required to bring the *Ms* to room temperature.

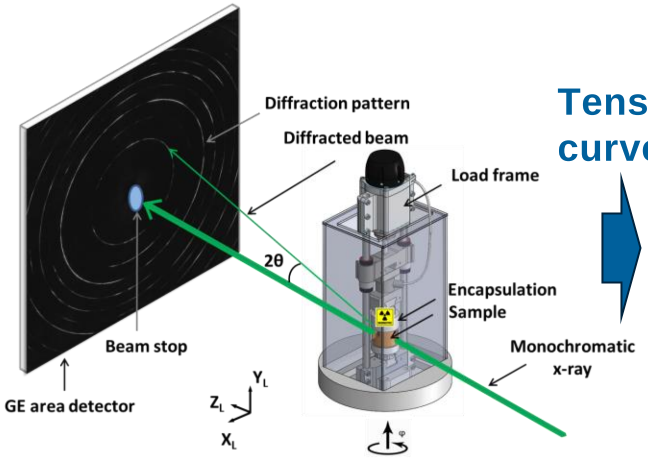
  σ_{thresh}=(T-Ms)/0.16
  T: test temperature
  Ms: martensite start temperature, -193°C
  0.16: increase in *Ms* per MPa [*]
  σ_{thresh} is calculated to be 1350 MPa

- Increased YS in irradiated samples facilitated the **martensitic transformation at the onset of plastic deformation.**
- The hardening effect of the martensites reduced the tendency towards necking, leading to the deformation wave and **a combined high strength and high ductility**.
- Unfortunately, such a mechanism **cannot not be activated at typical reactor operating temperatures** (300°C) (σ_{thresh} over 3 GPa).

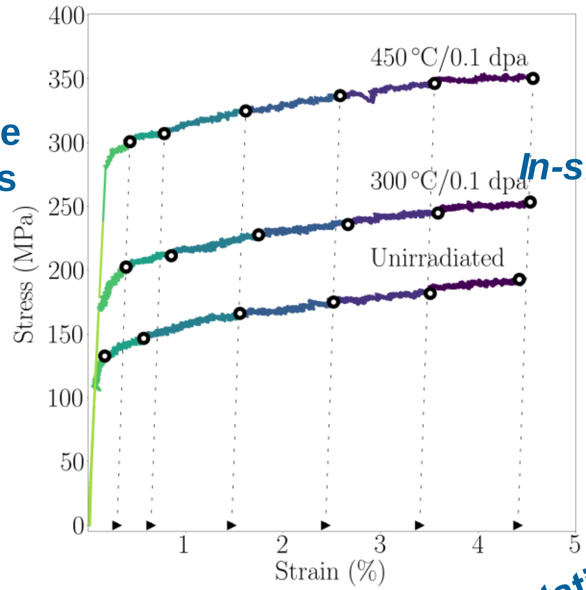
* Patel and Cohen, *Acta Metallurgica (1953)*

4D Study of Radiation-induced Deformation Heterogeneity with *In-Situ* FF-HEDM

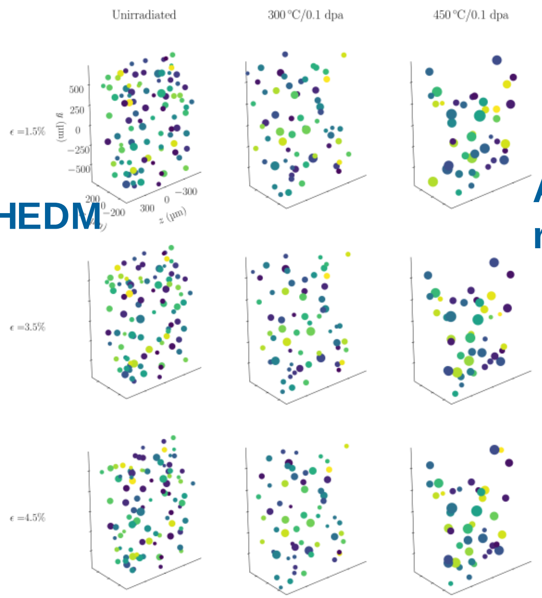
- **Specimens:** unirradiated and irradiated Fe-9Cr tensile specimens
- **Experiment:** *in-situ* room temperature tensile test with ff-HEDM
- **Results:**



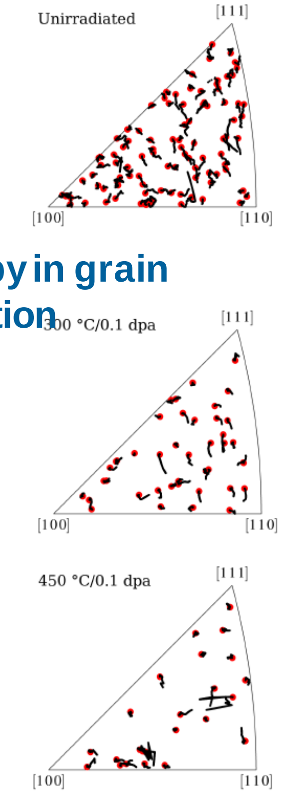
Tensile curves



In-situ HEDM



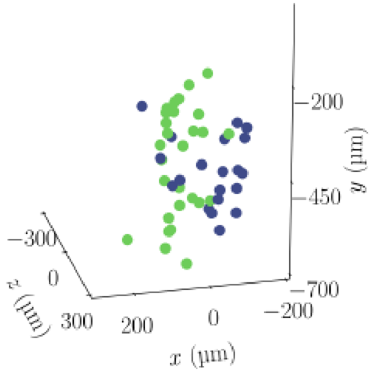
Anisotropy in grain reorientation



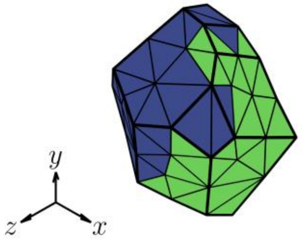
CPFEM

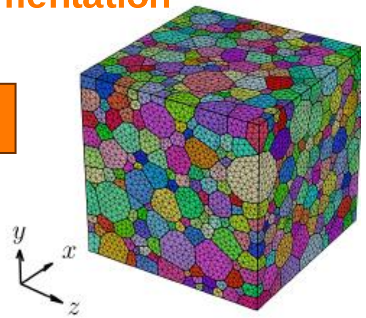
Grain fragmentation

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

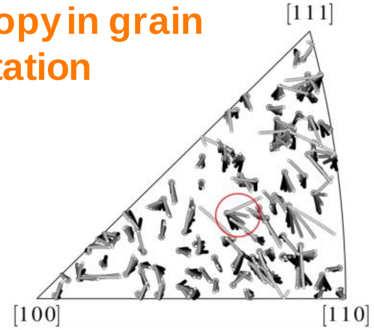


Grain fragmentation



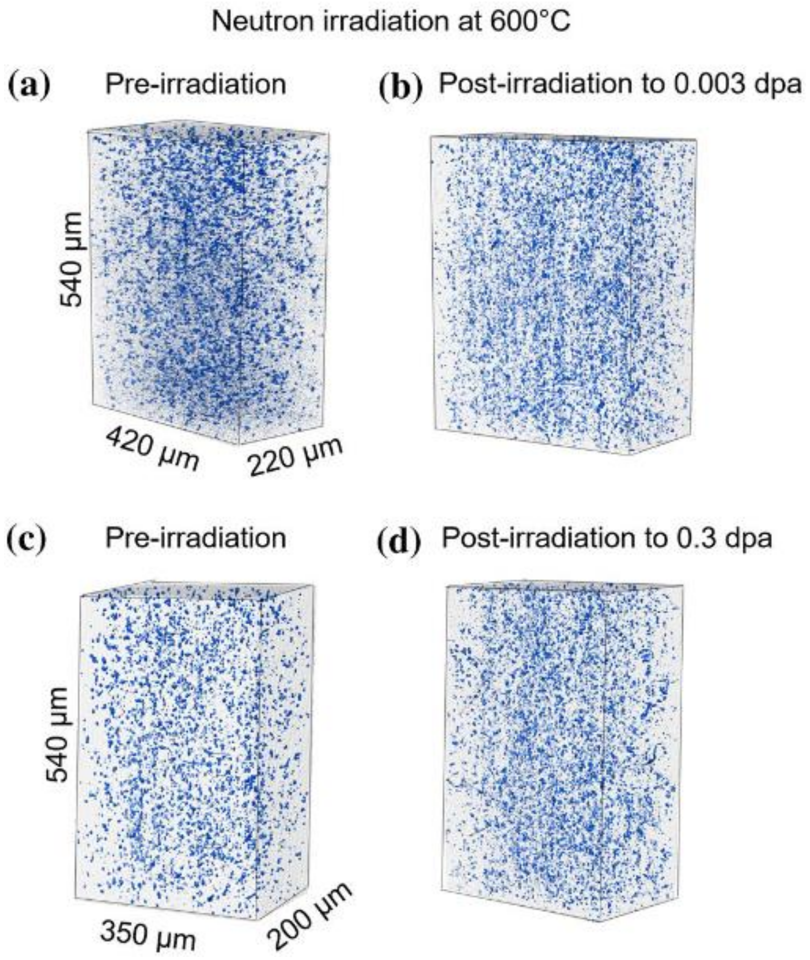


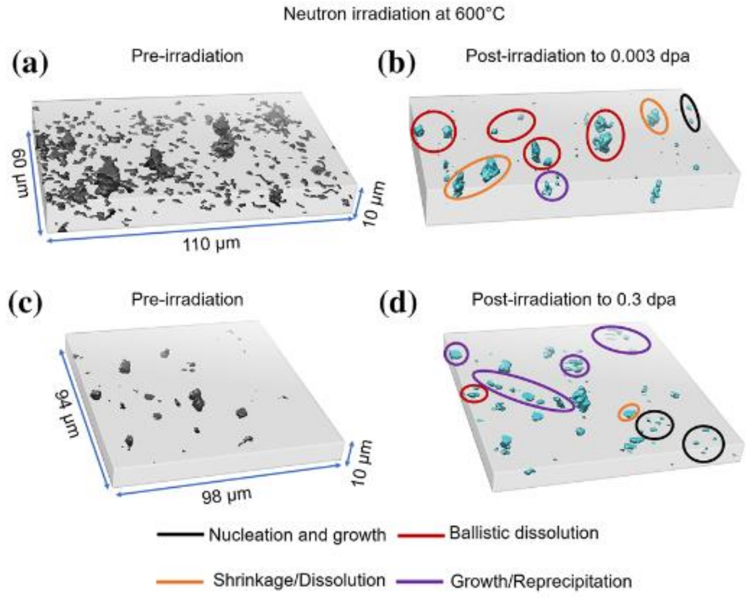
Anisotropy in grain reorientation

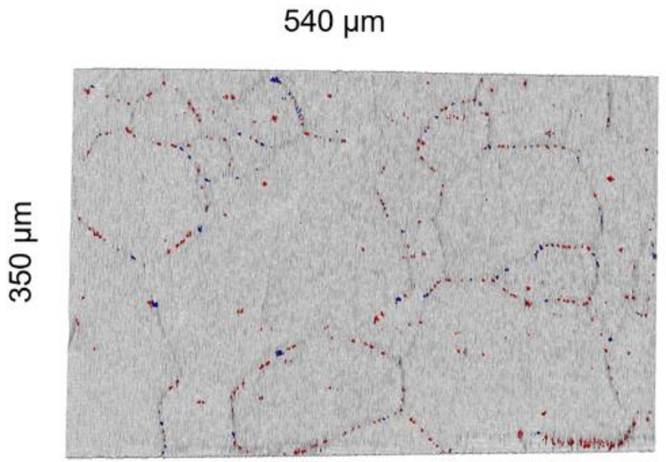


4D EVOLUTION OF $CR_{23}C_6$ PRECIPITATES IN NEUTRON-IRRADIATED HT-UPS STEEL

S. T. Nori, A. F. Bengoa, J. Thomas, J. Hunter, P. Kenesei, J.-S. Park, J. Almer, M. A. Okuniewski, *Journal of Materials Research*, 37 (2022)





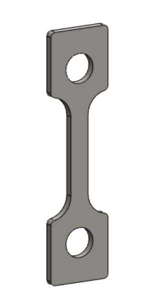


- **Sub-µm resolution** with synchrotron x-ray tomography using enhanced algorithm
- **Identical specimens** before and after irradiation
- **Conclusion:** Following neutron irradiation, the average $Cr_{23}C_6$ precipitate size reduced, affected by the synergy of nucleation and growth, ballistic dissolution, and inverse coarsening.

STAGED HEDM STUDY MEASURED SAME SAMPLE VOLUME ACROSS DEFORMATION LEVELS

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

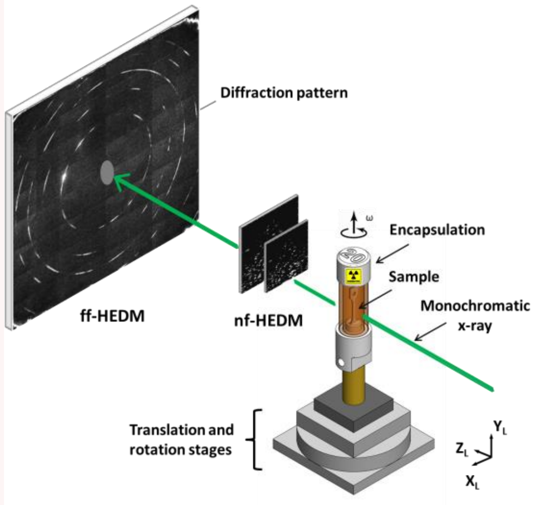
Step 0: Neutron irradiation

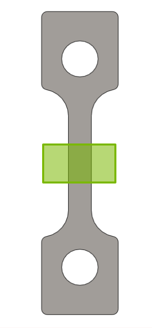


T_{irr} (°C)	300	450
dose (dpa)	0.1	0.1

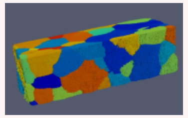
Step 1: Before tensile test

- *nf-HEDM*
- *ff-HEDM*
- *Tomography*

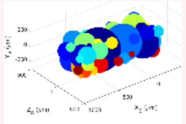




nf-HEDM

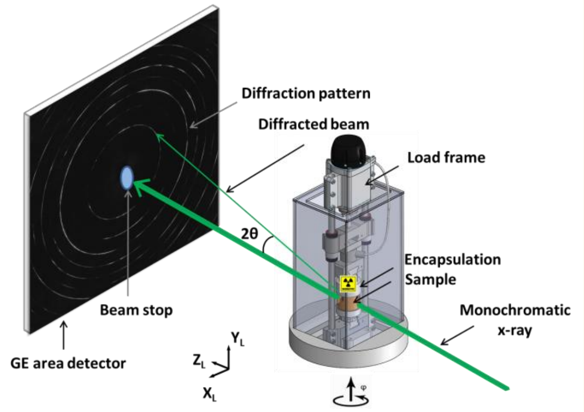


ff-HEDM

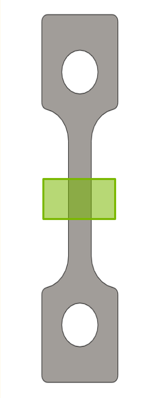


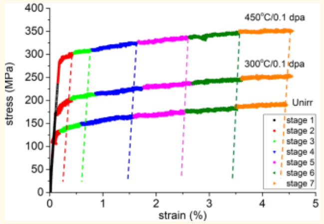
Step 2: During tensile test

- Continuous diffraction acquisition
- Interrupted ff-HEDM

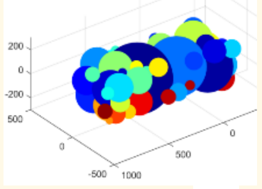


Interrupted loading



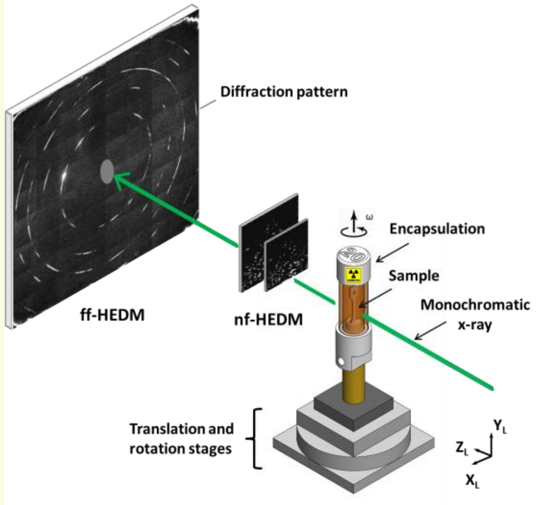


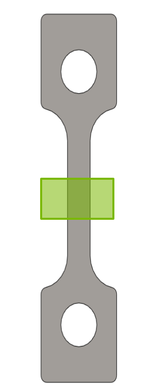
ff-HEDM



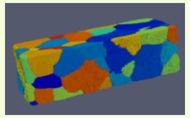
Step 3: After tensile test

- *nf-HEDM*
- *ff-HEDM*
- *Tomography*





nf-HEDM



ff-HEDM