NSUF User Development
Workshop on Irradiation Testing

May 20, 2024

Keith Jewell

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NSUF Chief Scientists

NSUF Post-Irradiation Examination (PIE) Capabilities Overview



U.S. DEPARTMENT OF ENERGY | Office of NUCLEAR ENERGY

NSUF Nuclear Science User Facilities

Content

- Introduction
- Non-destructive PIE methods
- Destructive PIE characterization
  - Physical properties
  - Surface characterization
  - Microstructure
  - Mechanical properties
- Accelerated testing
  - *In situ* irradiation + microstructure /or mechanical /or corrosion testing
  - *In situ* mechanical testing + microstructure characterization
  - *In situ* corrosion + stress
- Summary

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Post-Irradiation Examination (PIE)

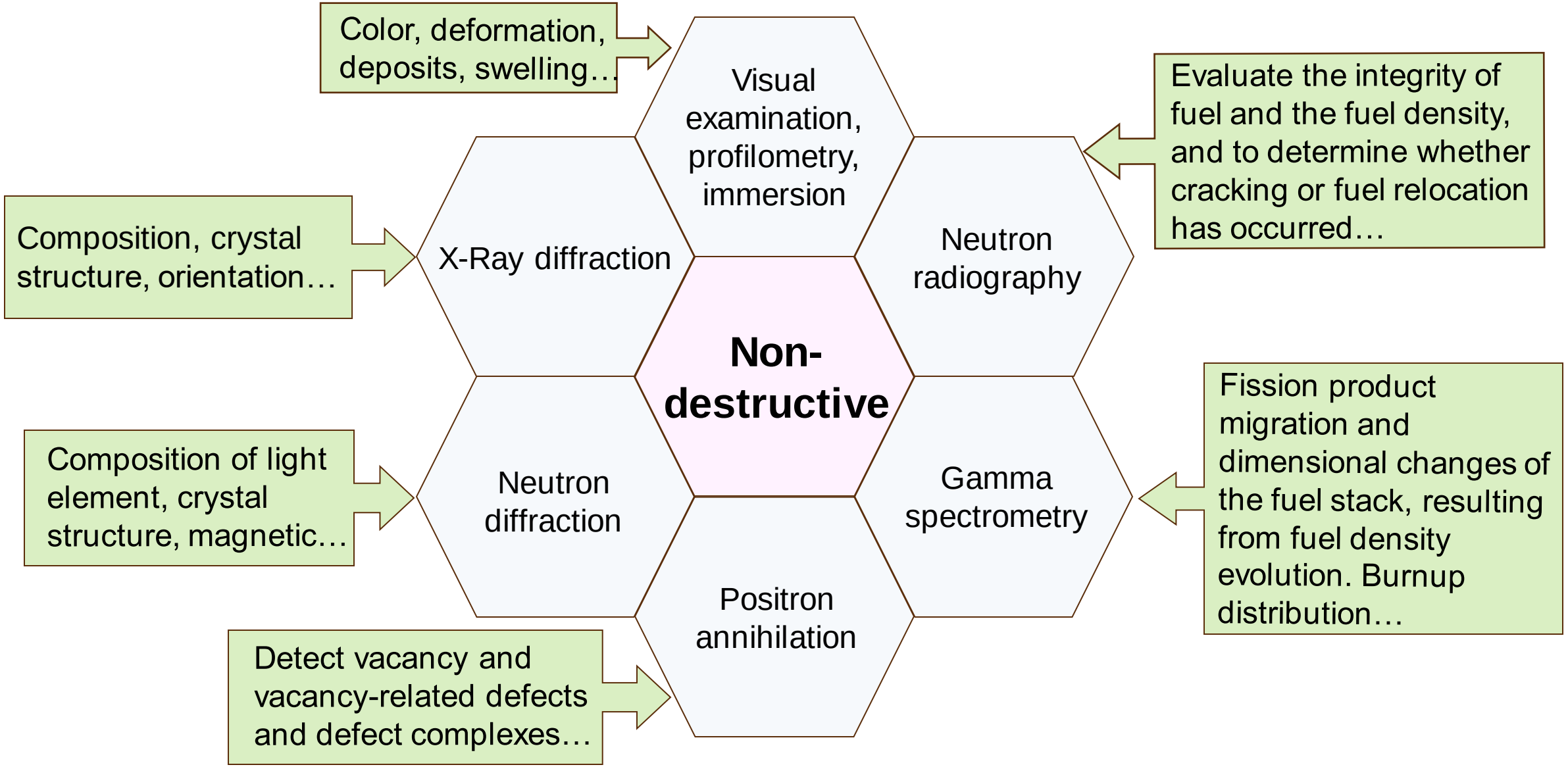
- **Objective**
  - Select new or improve nuclear materials
  - Characterize and understand in-core behavior of fuel and materials
  - Support the qualification of new research reactor materials
  - Interpretate reactor material safety tests
- **Techniques**
  - Non-destructive
  - Destructive

Content

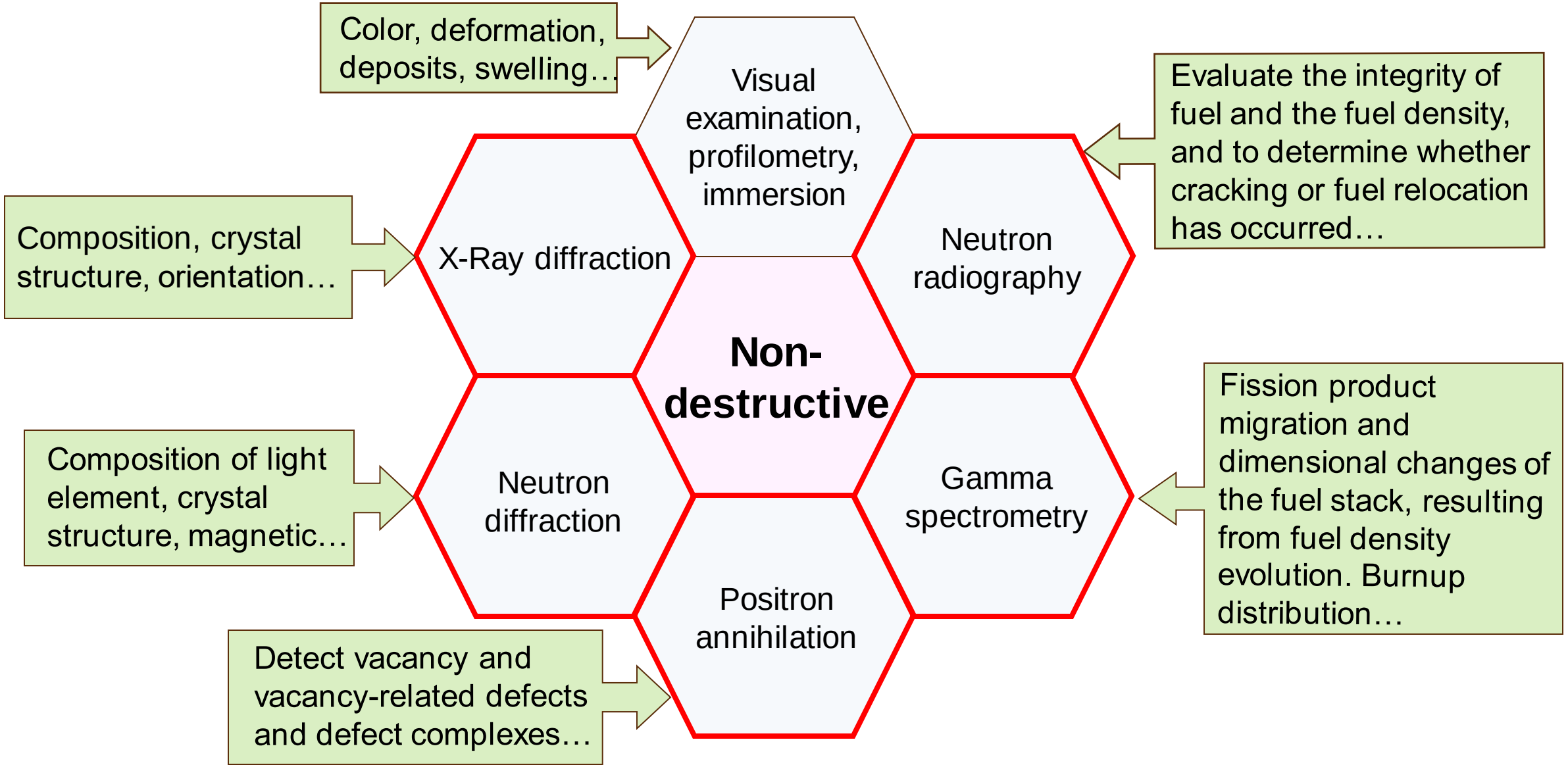
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Non-Destructive PIE Methods

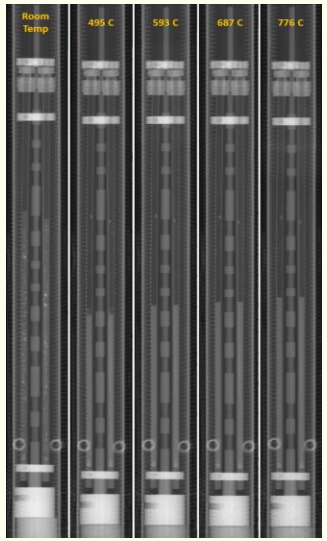


Non-Destructive PIE Methods

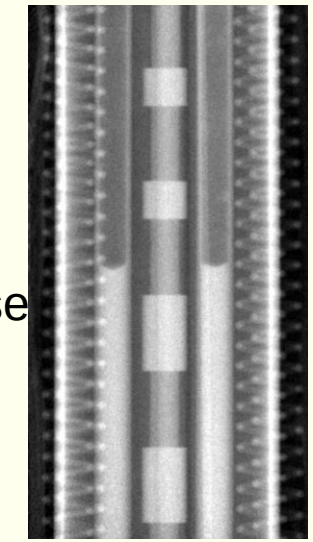


Neutron Radiography

Thermophysical measurements of salts



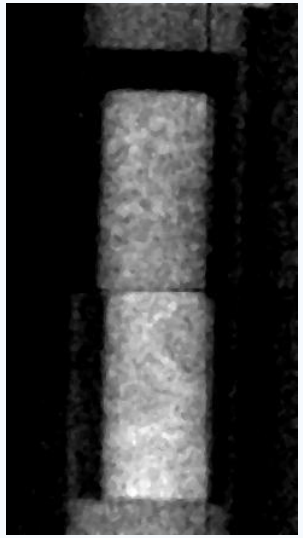
Volume expansion $PuCl_3$-base samples



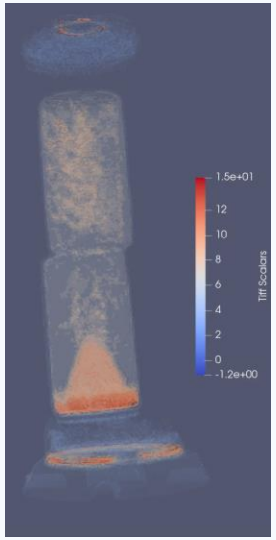
Salt is solidifying during cooling

Los Alamos NATIONAL LABORATORY EST.1943

***In situ* H-distribution measurements**



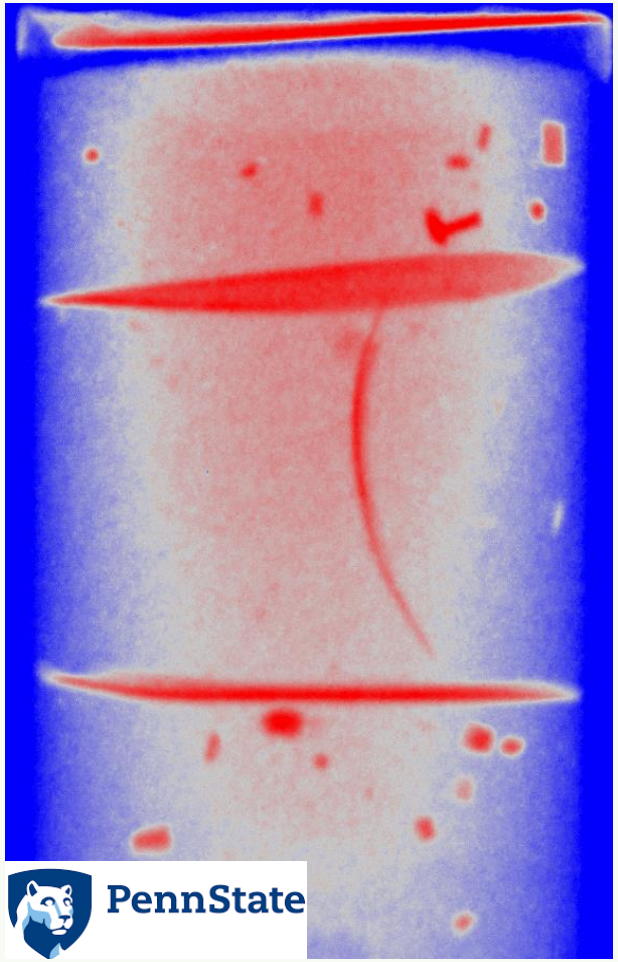
Hydrogen movement (based on temp. gradients)



CT scan of Hydrogen after heating

Los Alamos NATIONAL LABORATORY EST.1943

Visualization of particles in sand

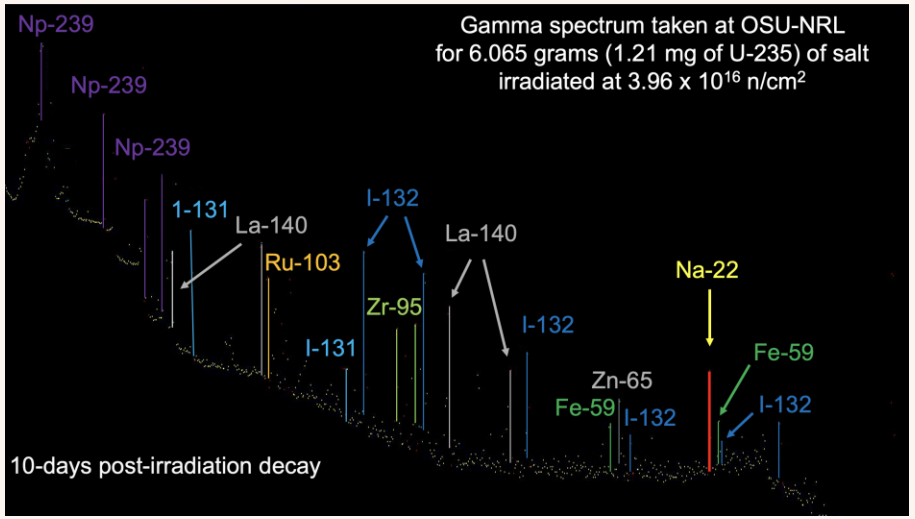


PennState

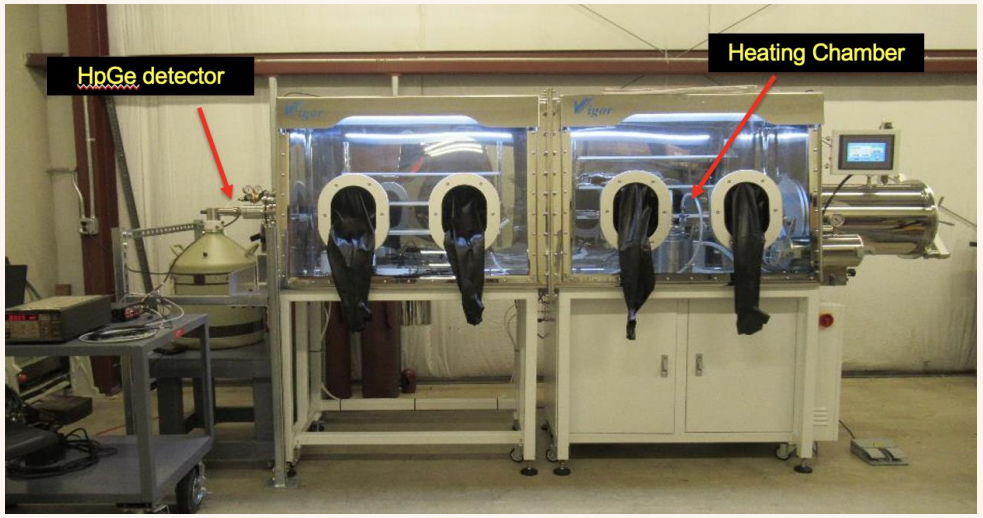
Courtesy of Alex Long, Kenan Unlu

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Gamma Spectrometry



Fission products spectrum of fuel salt [1.3 mg U-235]



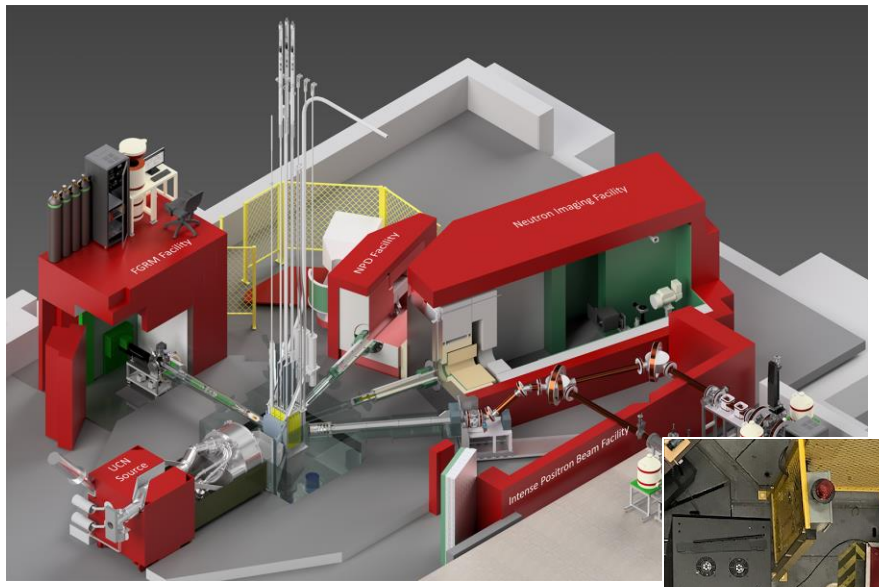
Fuel Salt Processing and Gamma Counting Facility

- The gamma counting measured a full gamma spectrum of shorted lived fission products, including those gaseous species.
- The first group who has irradiated the fuel salt (depleted U-235)

Courtesy of Raymond Cao

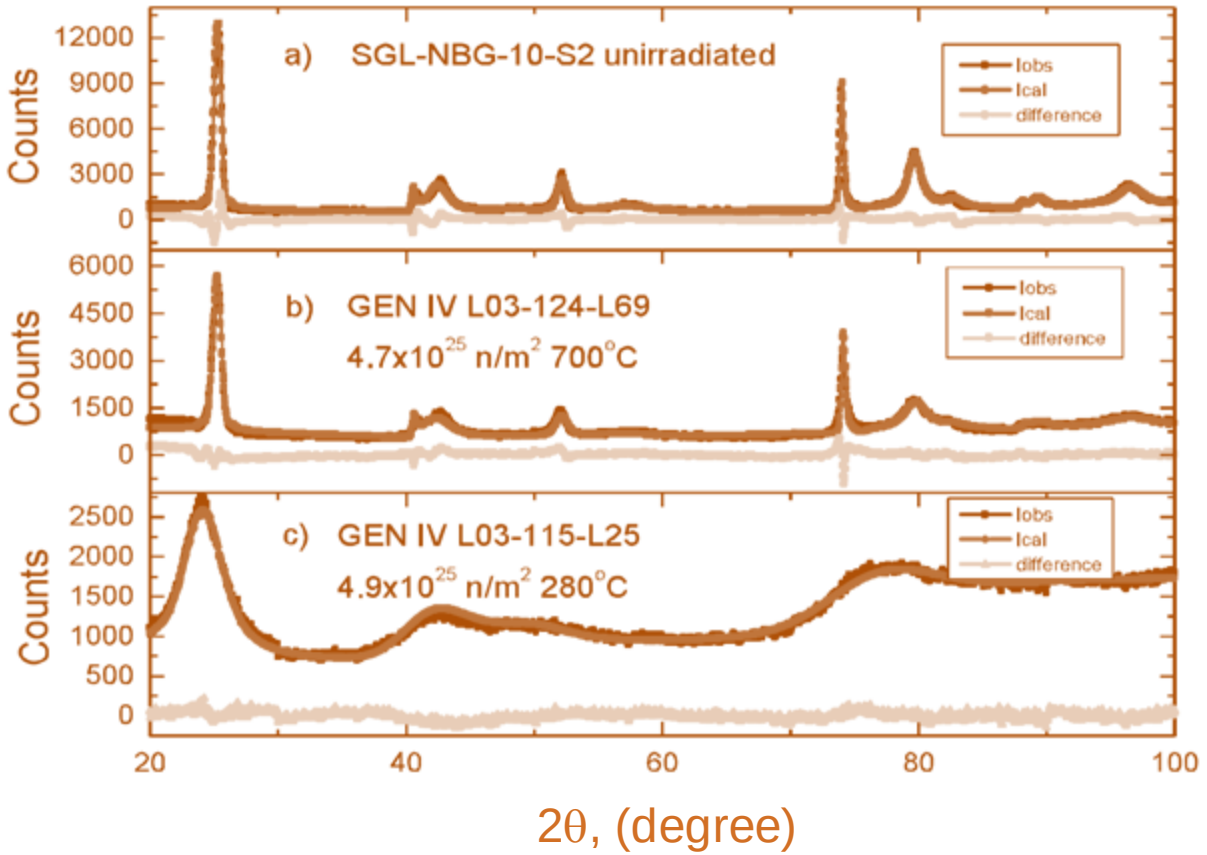
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Neutron Diffraction





Neutron diffractometer

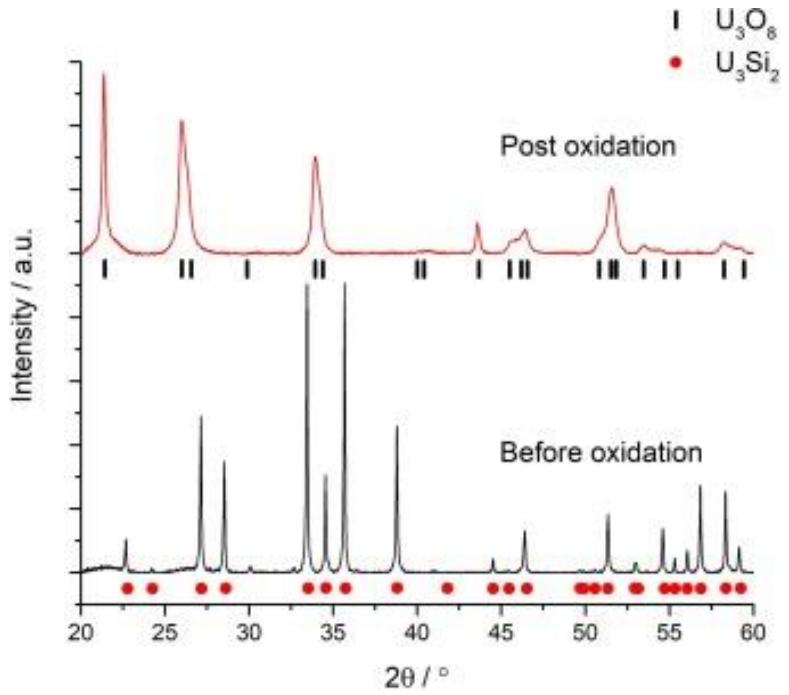


Impact of microstructure on neutron thermalization in graphite

Courtesy of Ayman Hawari

X-ray Diffraction

- XRD can reveal chemical composition, lattice structure and stresses in samples.



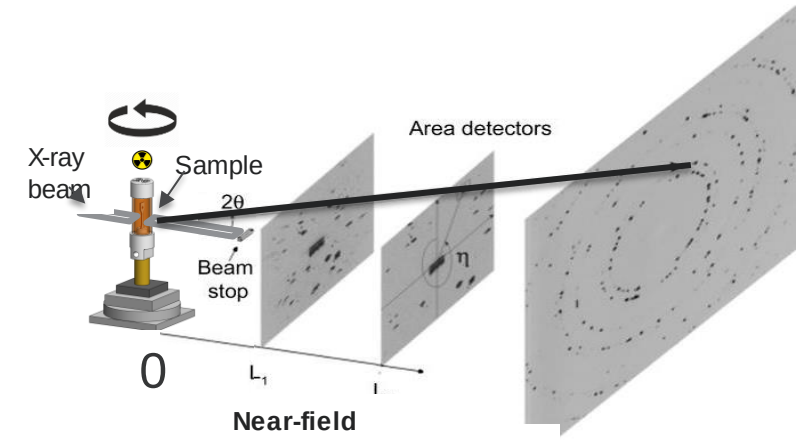
XRD patterns of U_3Si_2 before oxidation and after oxidation showing the formation of U_3O_8.



Courtesy of Catou Cmar

https://doi.org/10.1016/j.corsci.2020.108822

X-ray Diffraction: Beamline

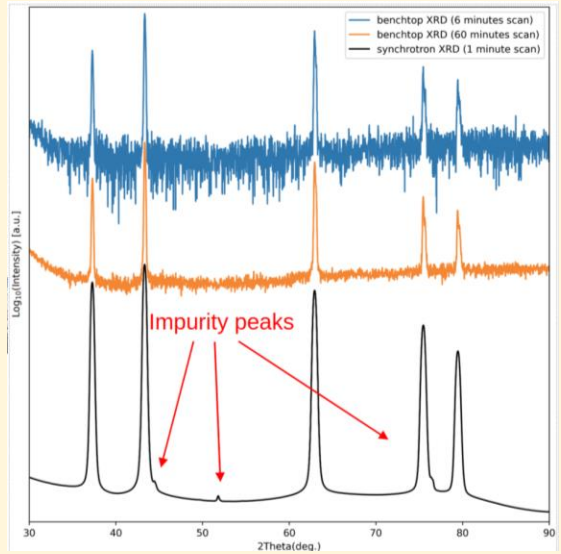


Lienert et al (2011
Poulsen (2012)



Argonne NATIONAL LABORATORY

- ***In situ*** **dynamic measurements**
- **Non-destructive 3D characterization**
- **Multiple length scales in one experiment**

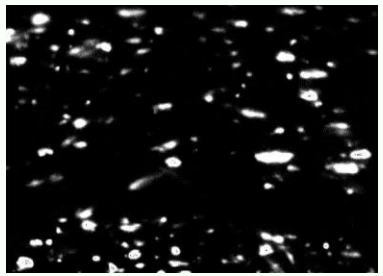


BROOKHAVEN NATIONAL LABORATORY

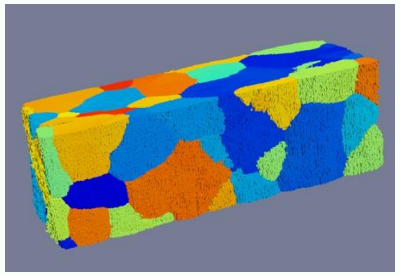
Near-field HEDM (nf-HEDM):

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

Detector images as sample rotates:



EBSD-like reconstruction

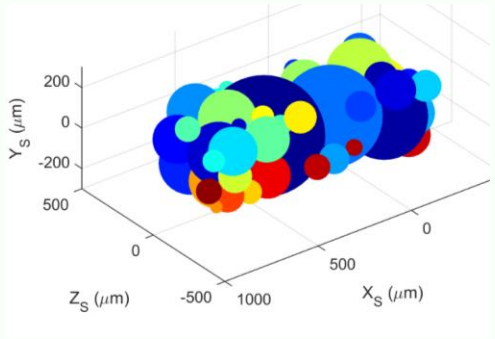


Far-field HEDM (ff-HDEM):

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

Detector images as sample rotates:

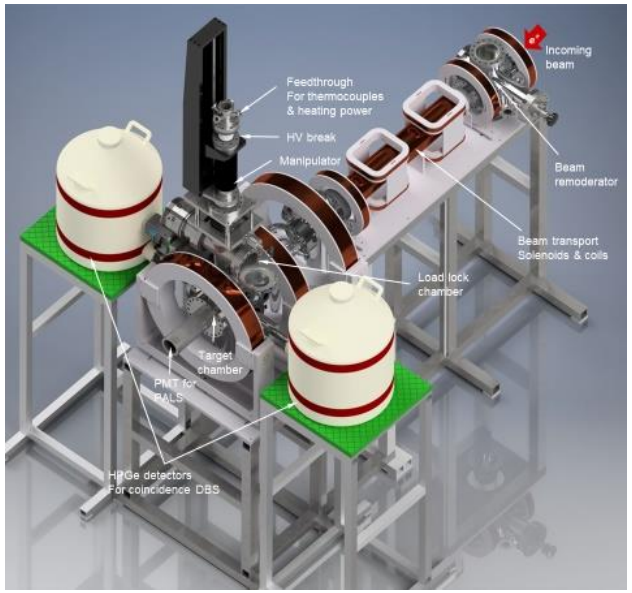


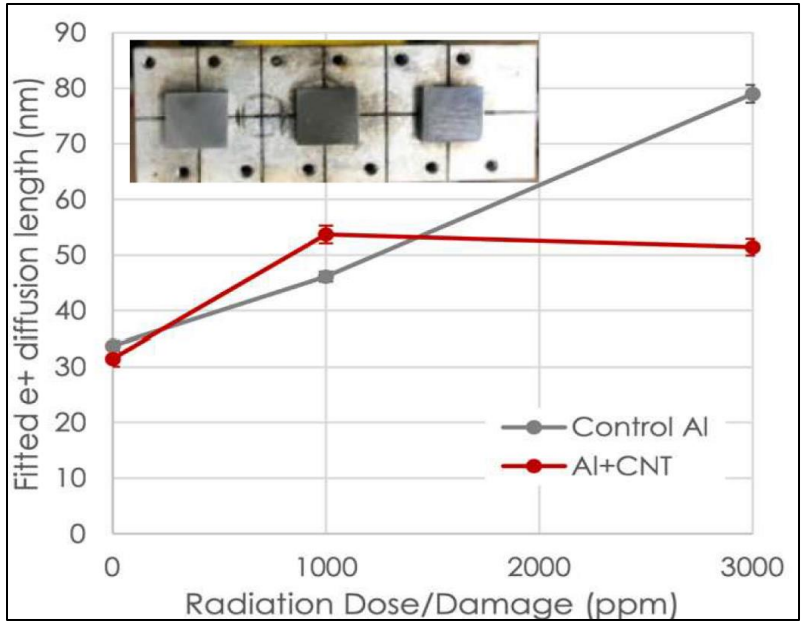


Courtesy of Xuan Zhang, Mehmet Topsakal

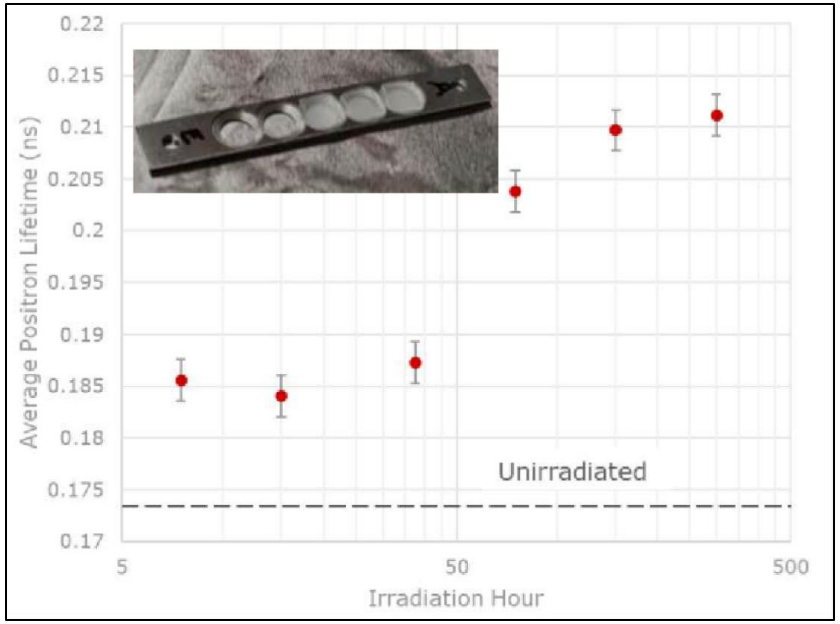
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Positron Annihilation





Quantification of porosity in irradiated metal via PAS



Average positron life of Ga_2O_3 before/after irradiation

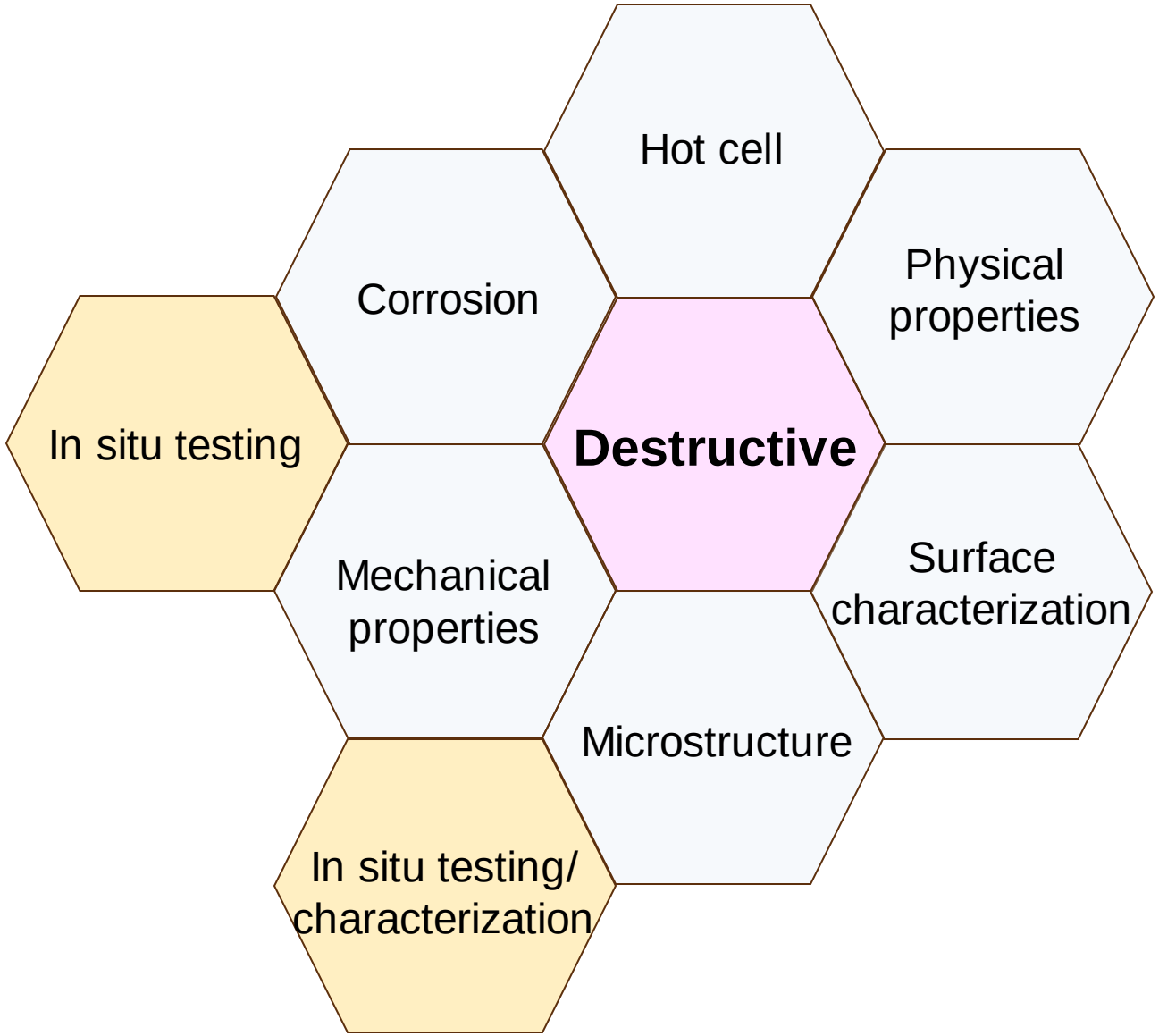
NC STATE UNIVERSITY

Courtesy of Ayman Hawari

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Destructive PIE Characterization Capabilities



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Hot Cell Facilities









Idaho National Laboratory

OAK RIDGE National Laboratory

Westinghouse

Massachusetts Institute of Technology









PNNL

Argonne NATIONAL LABORATORY

PennState

NC STATE UNIVERSITY

Physical Properties Measurement

- **Thermal stability:**
  - Thermogravimetric analysis (TGA)
- **Thermal expansion:**
  - Dilatometer
- **Thermal conductivity, thermal diffusivity, specific heat capacity:**
  - Laser flash analyzer (LFA)
  - Differential scanning calorimeter (DSC)

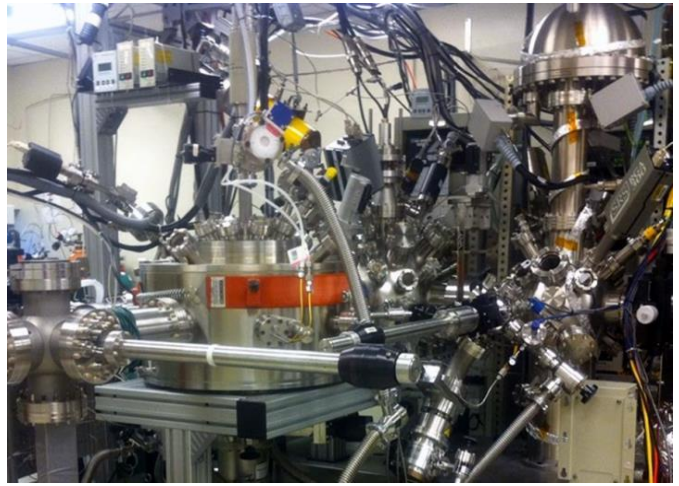


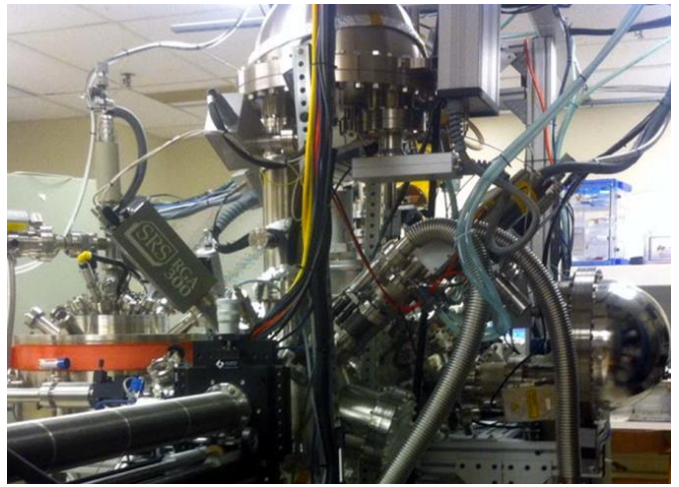
Simultaneous thermal analyzer, dilatometer, LFA, DSC inside of the fresh fuels glovebox (FFG)

Surface Characterization

At Interaction of Materials with Particles and Components Testing (IMPACT)

- In-situ characterization to study dynamic heterogeneous surfaces at the nano-scale exposed to varied environments that modify surface and interface properties
- Characterization of the evolution of elemental, chemical, and thermodynamic states of ultra-thin film surface and interfaces using complementary surface-sensitive characterization techniques
  - Thin film & multilayer (precise four-pocket e-beam evaporator)
  - Ion beam sputter cleaning
  - Ion irradiation (300 eV - 2 keV @ LN_2 - 1200º C)
  - X-ray Photoelectron Spectroscopy (XPS: surface chemistry)
  - Auger Electron Spectroscopy (AUGER: properties of elements and compounds)
  - EUV (13.5 nm) Photoelectron Spectroscopy (EUPS: electron density)
  - Ultraviolet Photoelectron Spectroscopy (UPS: composition)
  - Low-Energy Ion Scattering Spectroscopy (LEISS: atomic composition)



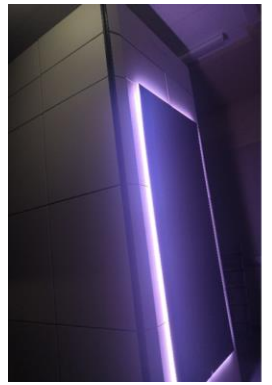


PURDUE UNIVERSITY

Courtesy of Ahmed Hassanein

Microstructure — Morphology

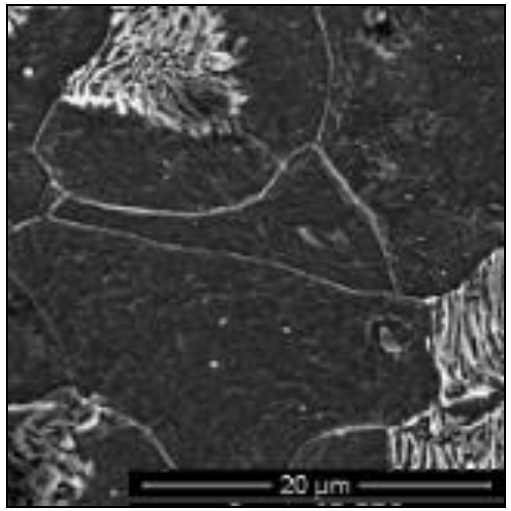
- Optical metallography
- Scanning electron microscopy (SEM)/BSE/EBSD/FIB
- Transmission electron microscopy (TEM)

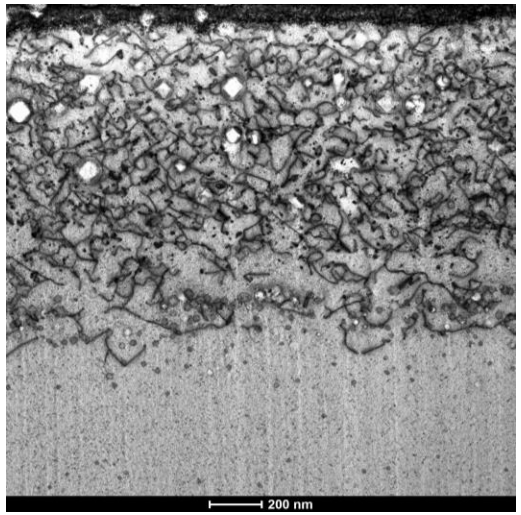


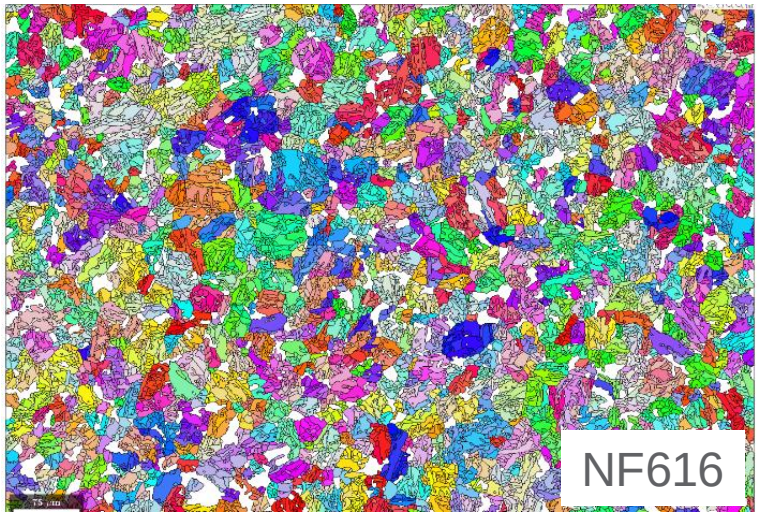


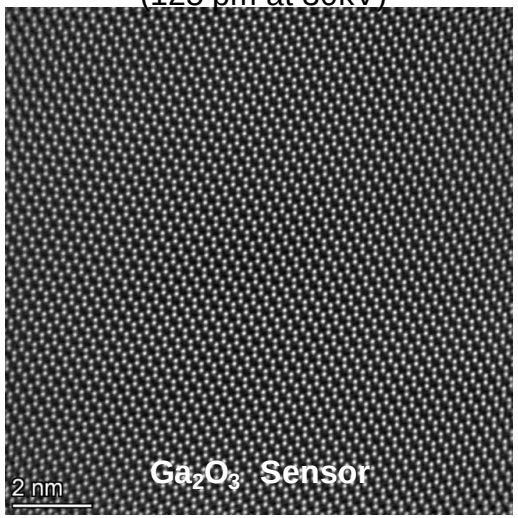
Spectra 300 STEM

STEM resolution 50 pm
(125 pm at 30kV)









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INL Idaho National Laboratory

PNNL

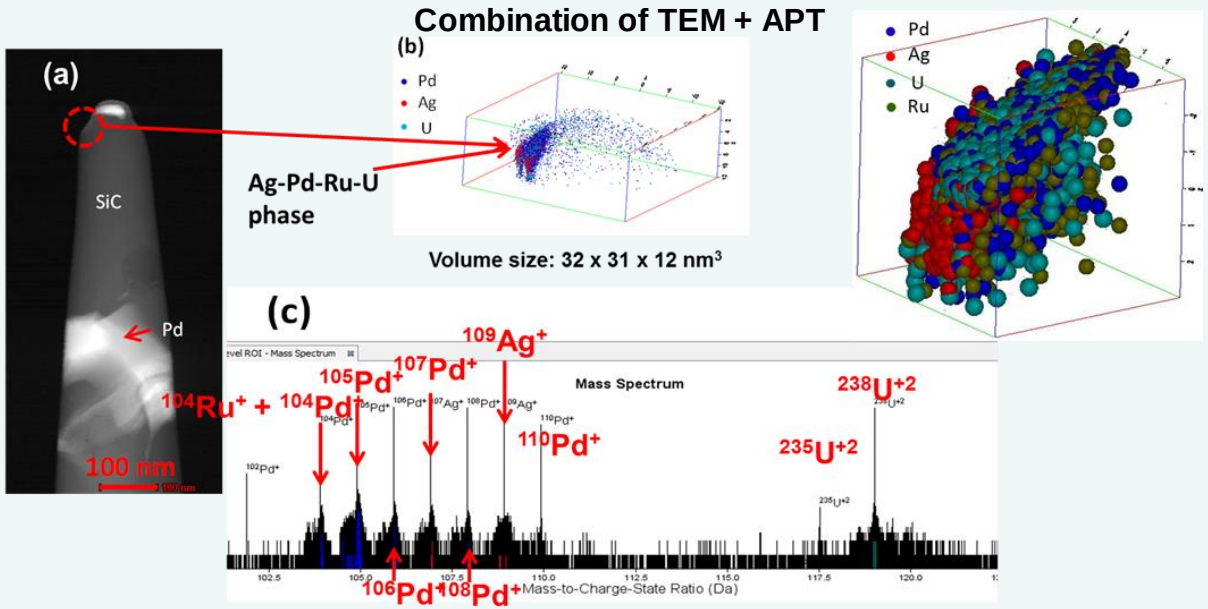
CAES Center for Advanced Energy Studies

Courtesy of Yaqiao Wu, Peter Hosemann, Stuart Maloy

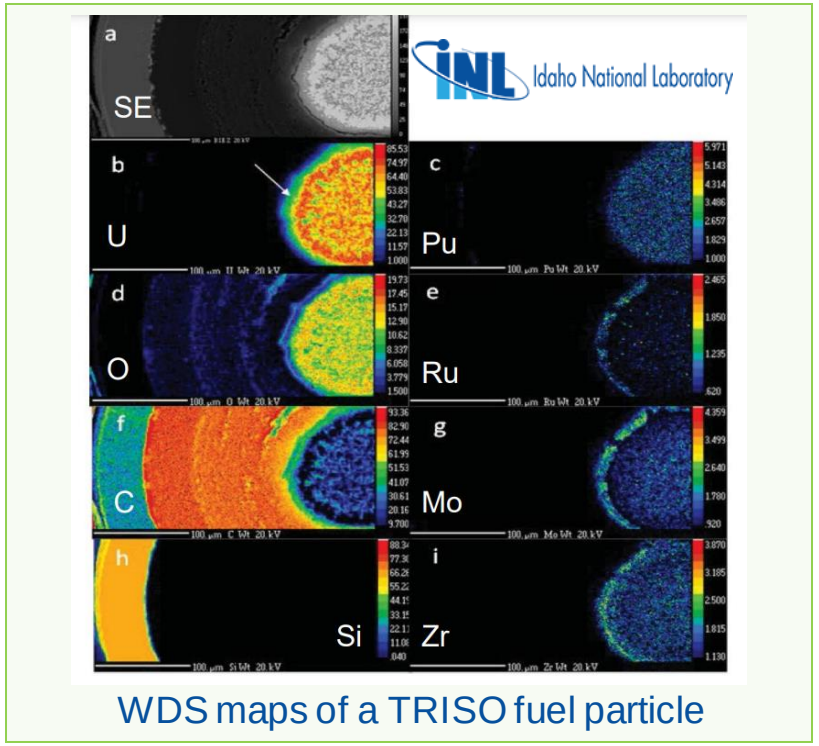
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Microstructure — Composition Analysis

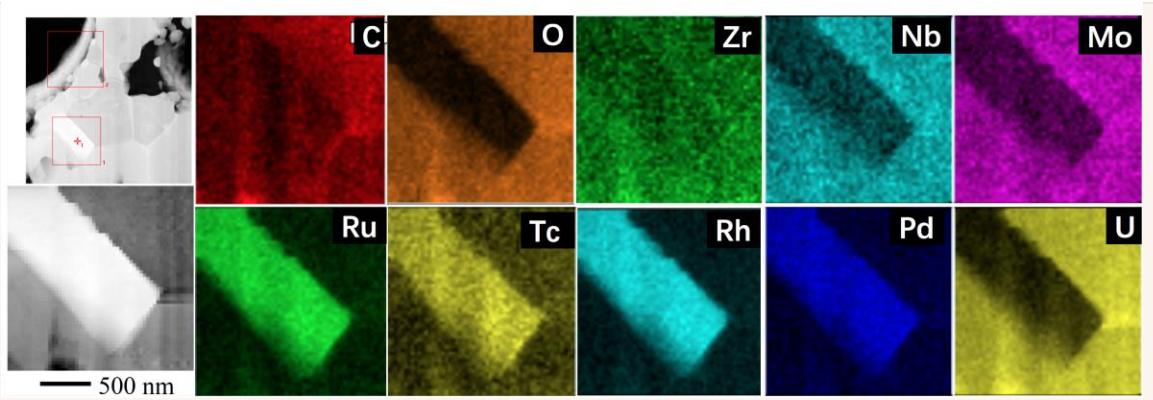
- Atom probe tomography (APT)
- Electron Probe MicroAnalysis (EPMA)
- Energy Dispersive Spectroscopy (EDS)
- Electron Energy Loss Spectroscopy (EELS)



STEM HAADF image of an irradiated TRISO APT sample tip (a), corresponding reconstructed three-dimensional APT maps showing a Ag-Pd-Ru-U - rich phase in a volume size of 32 nm x 31 nm x 12 nm (b) and mass spectrum showing detected isotopes of Ag, Pd, Ru and U (c).



WDS maps of a TRISO fuel particle



TEM images and EDS maps of irradiated fuel particle

Courtesy of Yaqiao Wu, Yong Yang

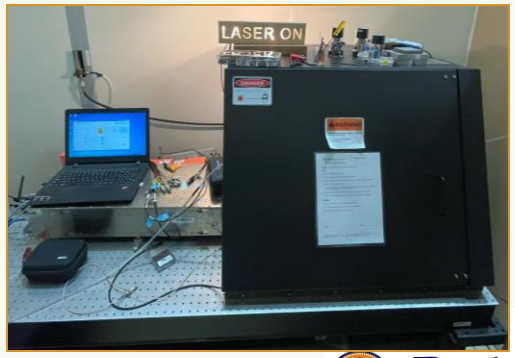
Mechanical Testing

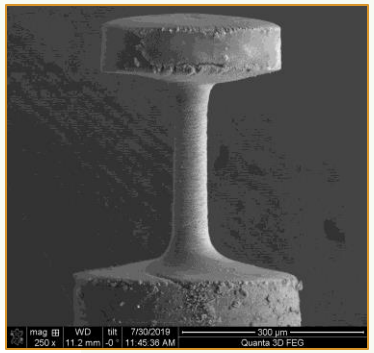
- Hardness/ Bend test/ Tensile/ Creep/ Fatigue/ Compact tension/ Charpy impact (toughness)



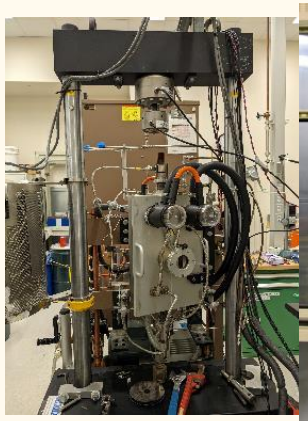


Westinghouse

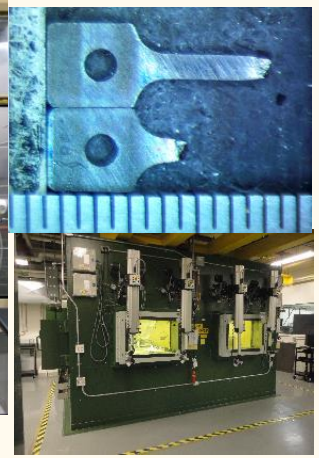




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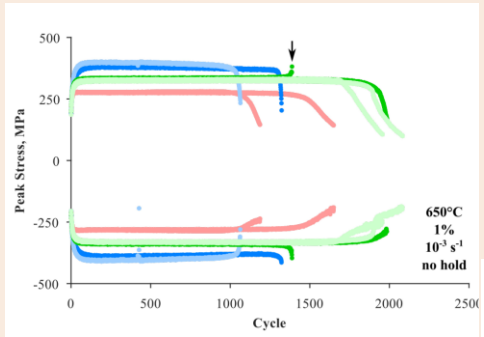




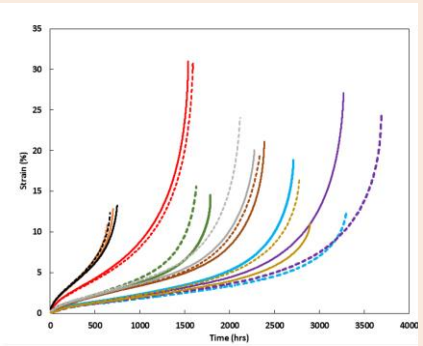
PNNL



OAK RIDGE
National Laboratory



INL Idaho National Laboratory



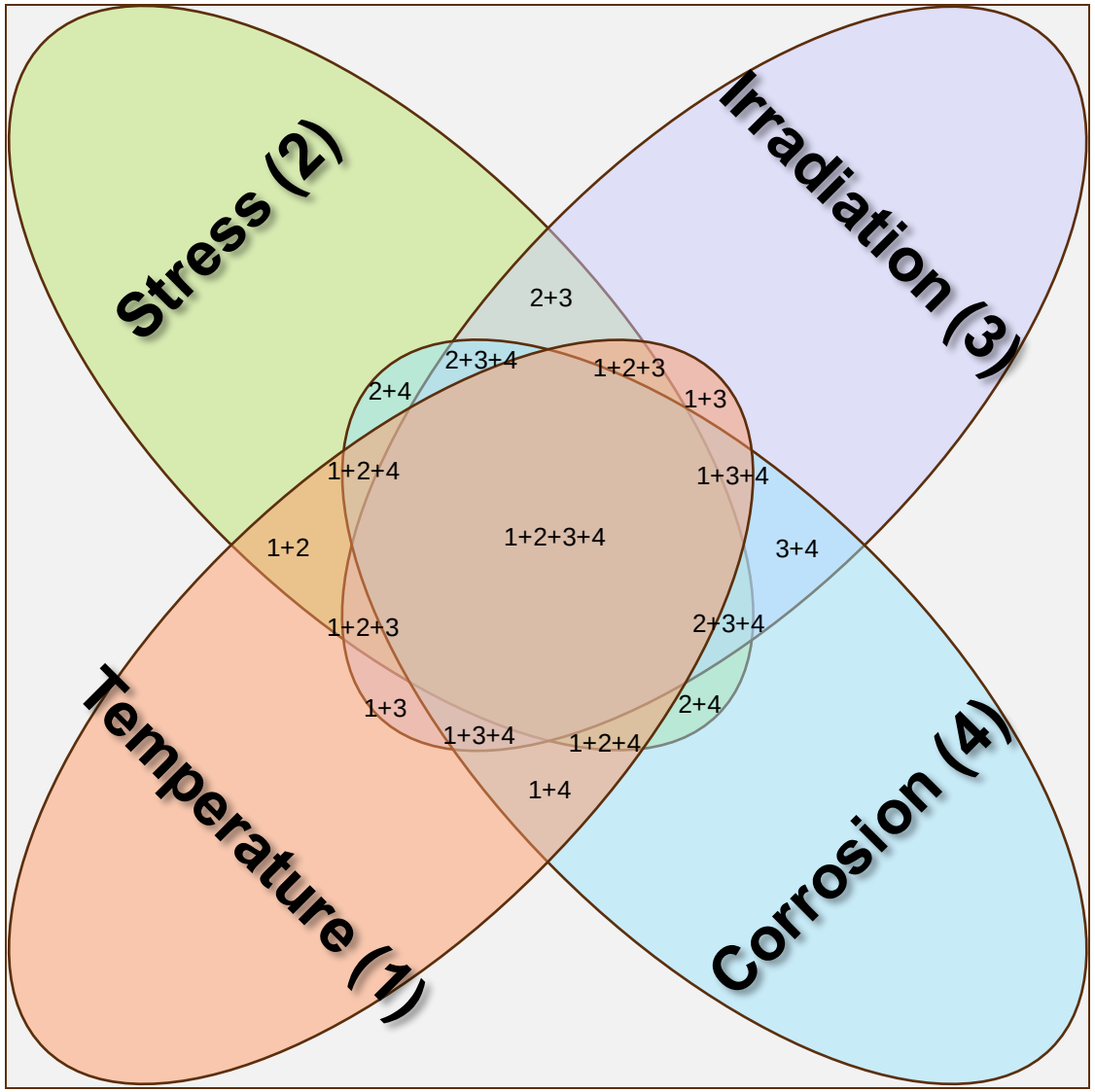
Courtesy of Catou Cmar, Peter Hosemann, Kory Linton, Stuart Maloy

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Accelerated Testing

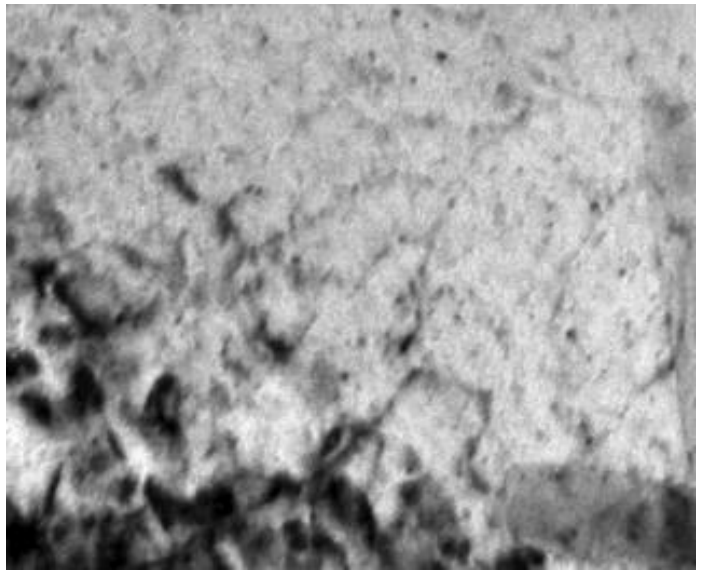


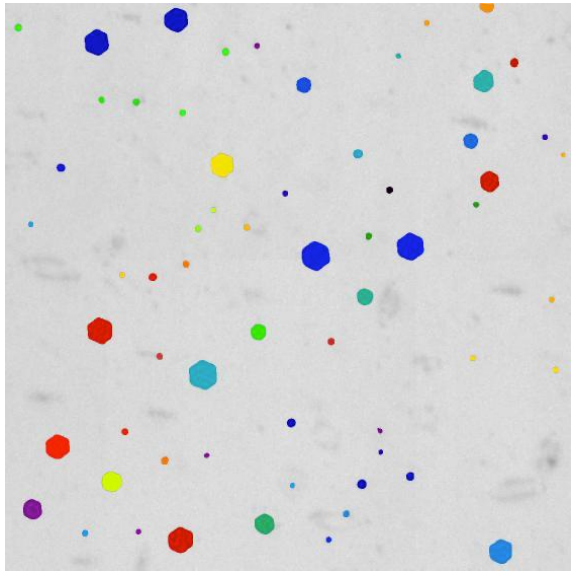
- Testing under conditions in excess of normal service parameters
- Testing under multiple factors at once

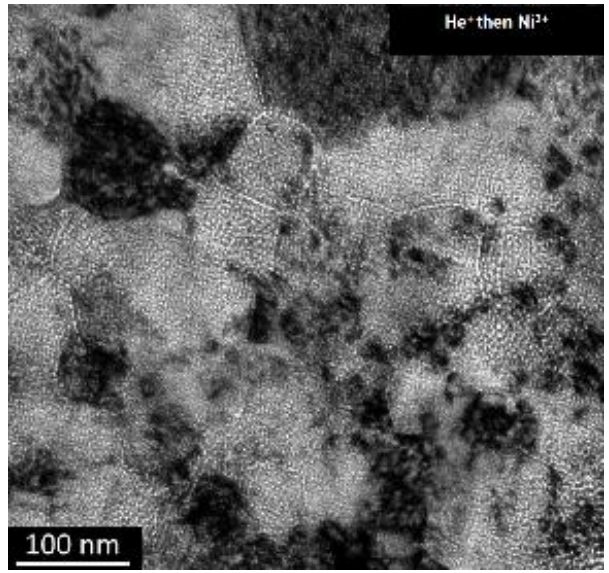
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In Situ Ion Irradiation + Microstructure Characterization

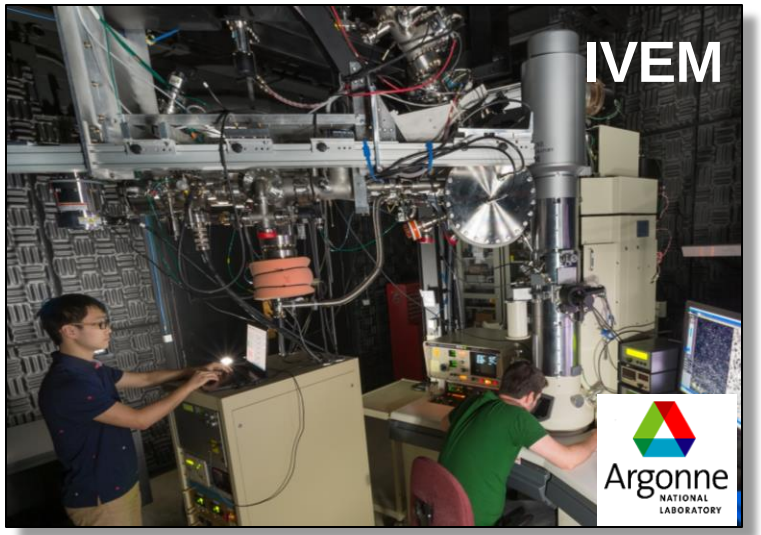


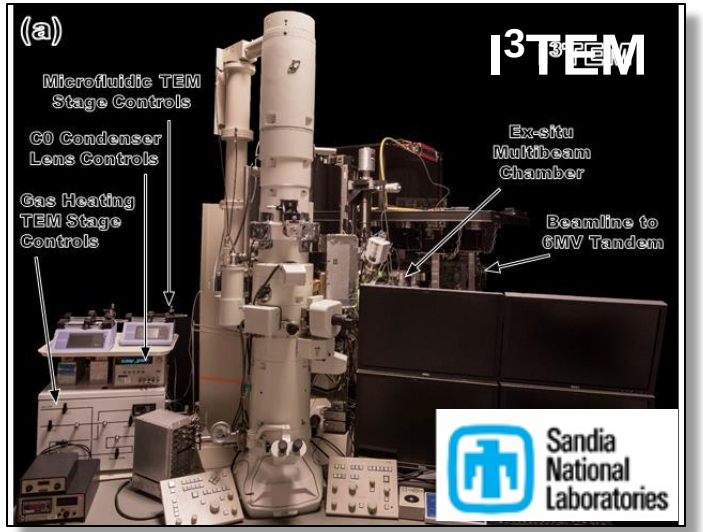




K. Hattar et al., 2021, SAND2021-2542C



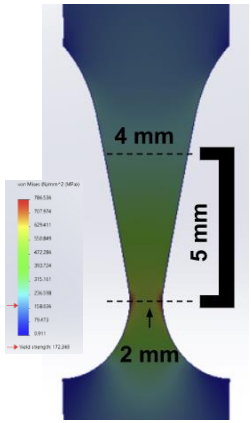




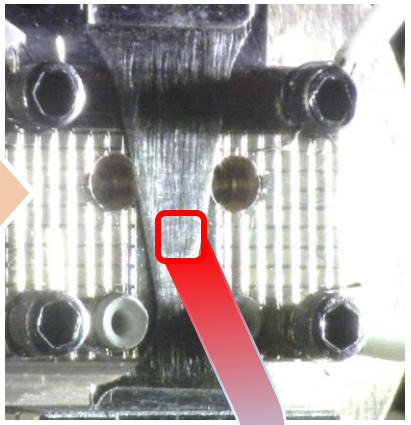
Courtesy of Kevin Field, Wei-Ying Chen

In Situ Ion Irradiation + Mechanical Testing in MIBL

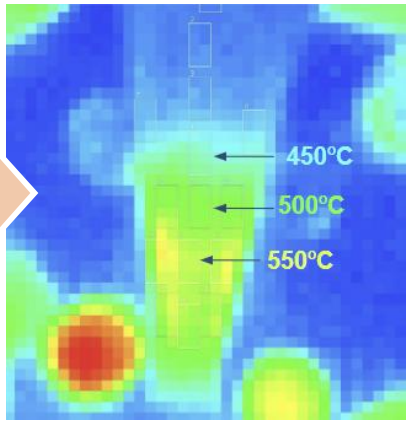
Design



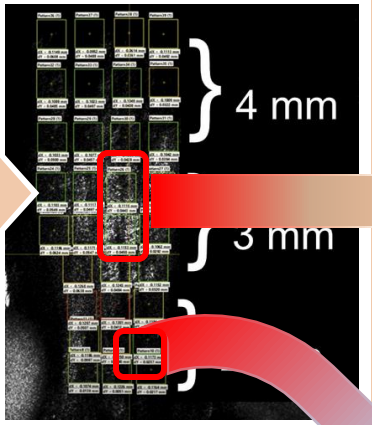
Irradiate Tapered Sample



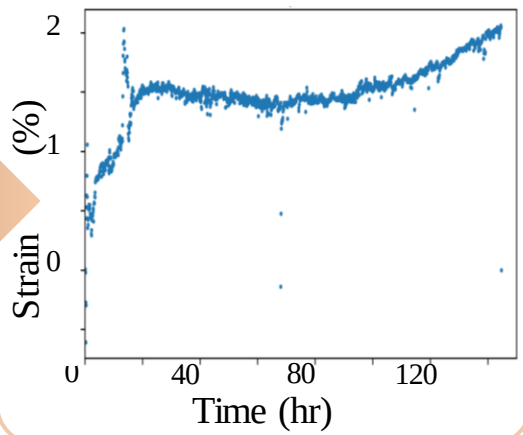
In-Beam Temp. Monitor



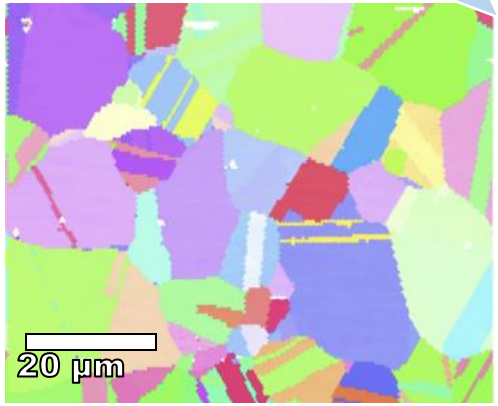
32 AOI tracking



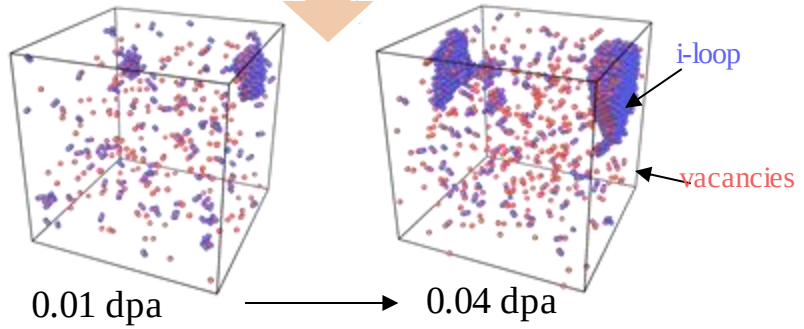
Temp.+Stress+DPA→Strain



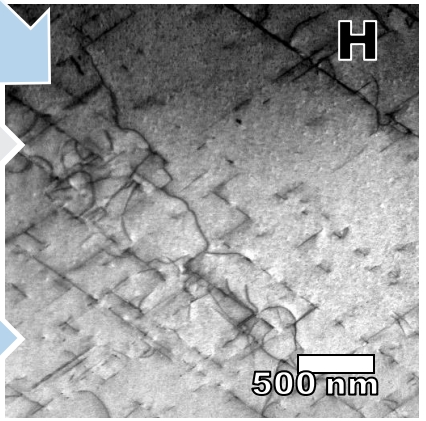
Pre-irr. Characterization



OLPD Simulations



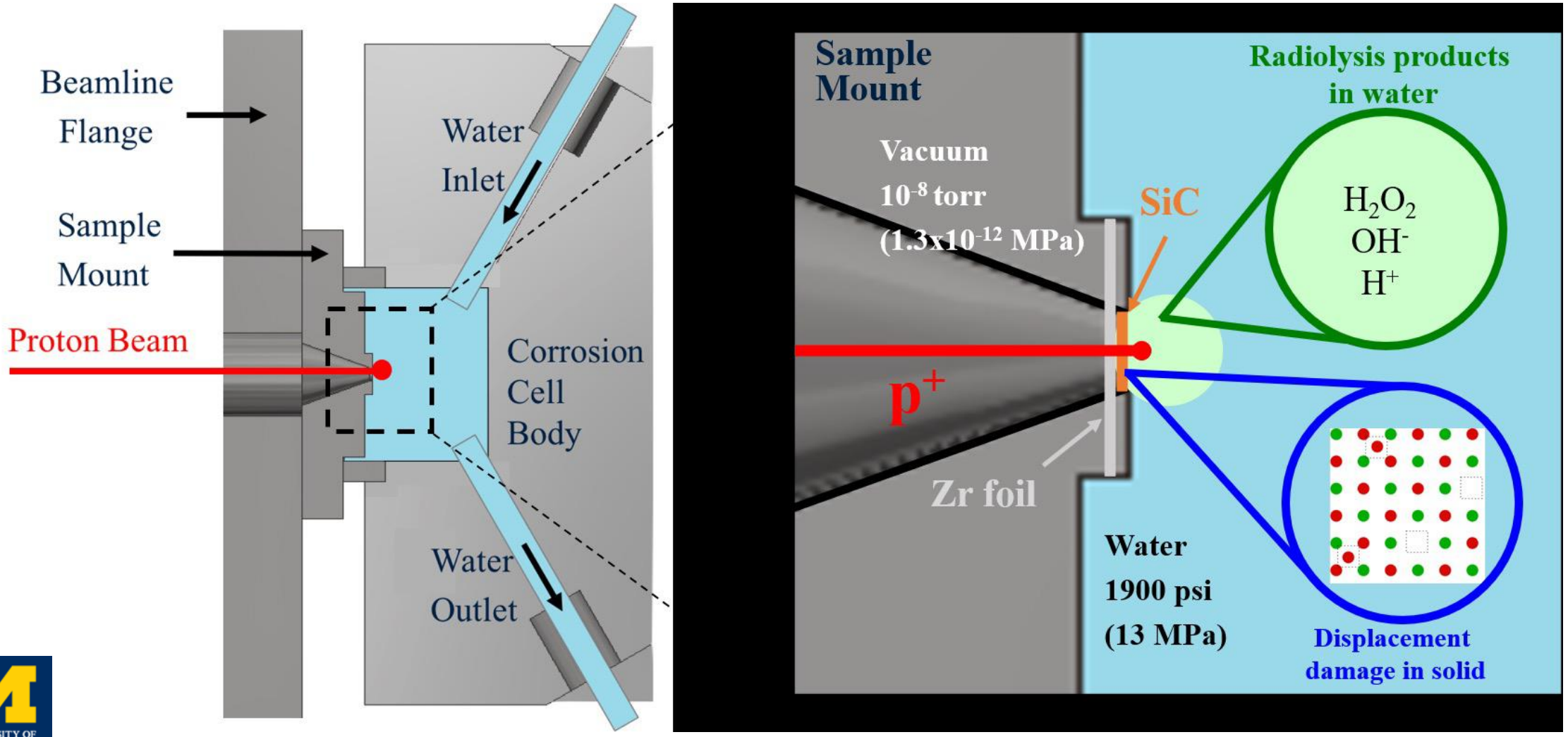
Post-irr. ML-S/TEM



Courtesy of Kevin Field, Charlie Hirst

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In Situ Proton Irradiation + Corrosion in MIBL

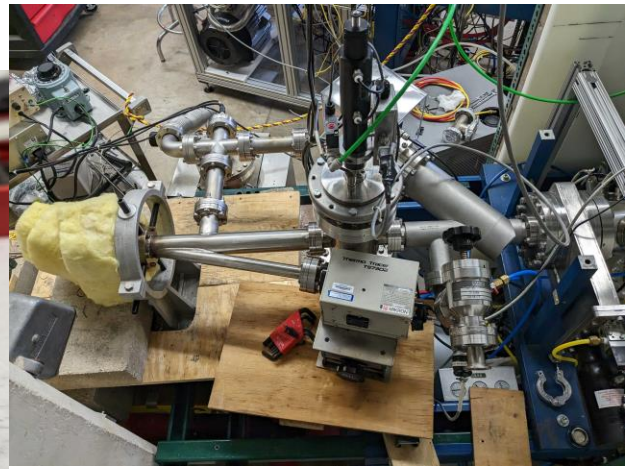


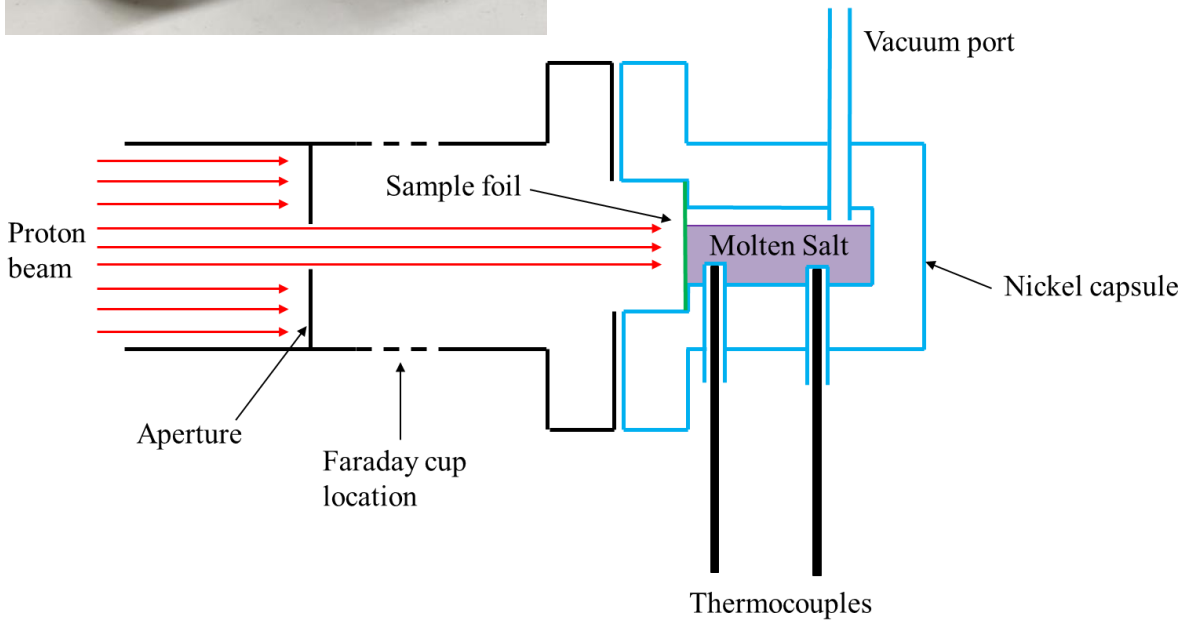
Courtesy of Peng Wang

In Situ Ion Irradiation + Molten Salt Corrosion

- Understand performance of structural alloys for molten salt reactors under simultaneous corrosion and irradiation
- Heated Ni capsule holds molten FLiNaK behind thin foil target







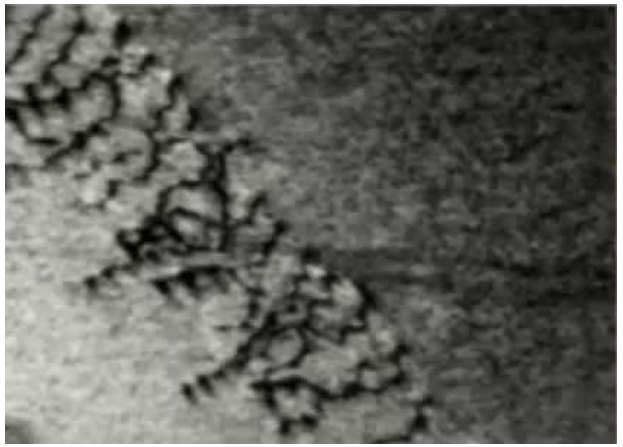
Cole Evered, et al. NEUP Project 21-24195: Enhancing Yellowjacket for Modeling the impact of Radiation and Stress on the Corrosion of Molten-Salt-Facing Structural Components, Federal Grant/Cooperative Agreement Number: DE-NE0009163

Courtesy of Cole Evered, Adrien Couet

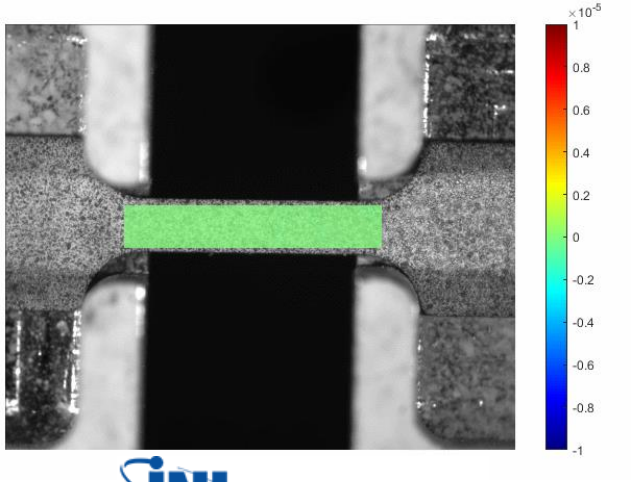
Content

- Introduction
- Non-destructive PIE methods
- Destructive PIE characterization
  - Physical properties
  - Surface characterization
  - Microstructure
  - Mechanical properties
- Accelerated testing
  - *In situ* irradiation + microstructure /or mechanical /or corrosion testing
  - *In situ* mechanical testing + microstructure characterization
  - *In situ* corrosion + stress
- Summary

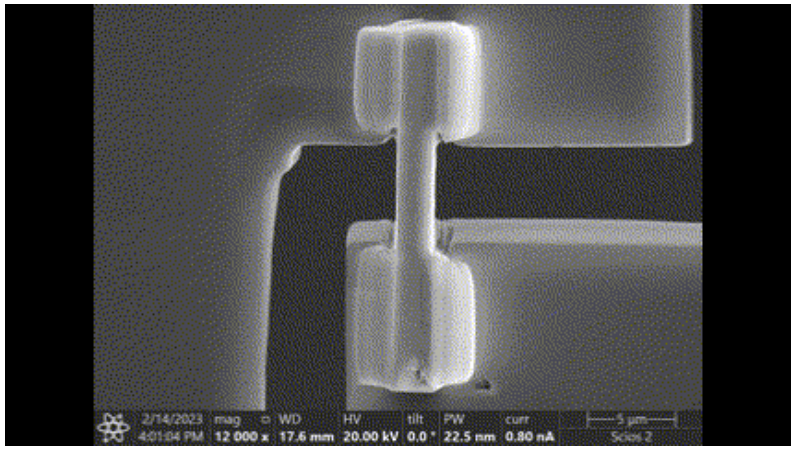
In situ Mechanical Testing + Microstructure Characterization



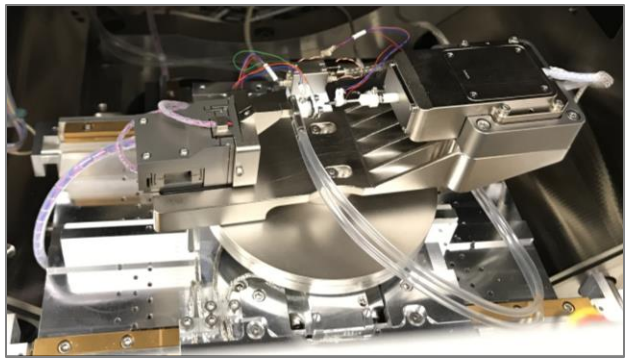
Argonne NATIONAL LABORATORY



INL Idaho National Laboratory

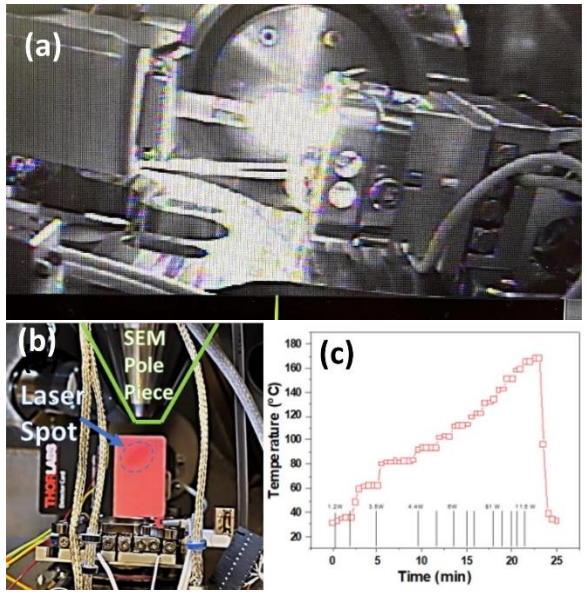


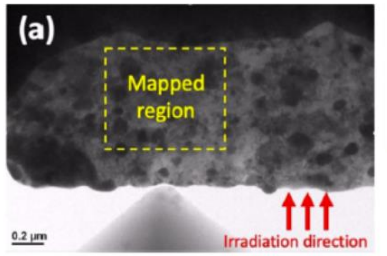
Berkeley UNIVERSITY OF CALIFORNIA



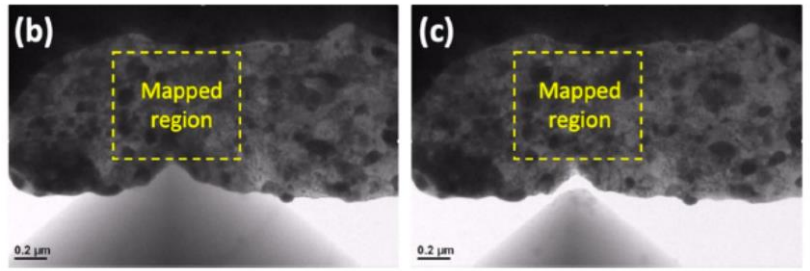
UNIVERSITY OF FLORIDA

Sandia National Laboratories





CAES Center for Advanced Energy Studies



NSUF Nuclear Science User Facilities

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In situ Stress + Corrosion: IASCC

- Stress corrosion testing on irradiated material at Materials in High Temperature Extreme Environments (MiHTEE) Lab





Courtesy of Peng Wang, Stephen Raiman

UNIVERSITY OF MICHIGAN
NSUF Nuclear Science User Facilities

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Summary

- NSUF has comprehensive PIE capabilities
  - Non-destructive capabilities
  - Destructive characterization methods
  - Accelerated testing capabilities for various reactors environments
- NSUF provides all these world-class capabilities to support cutting-edge research

No other user facility in the United States offers as wide a variety of PIE capabilities as are available through the NSUF.



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