

NSUF Annual Program Review

April 15-18, 2024

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**NSUF Chief Scientist**

# 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
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# NSUF PIE Institutions in the USA

- Total 21 institutions
- Total 19 PIE institutions



# 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

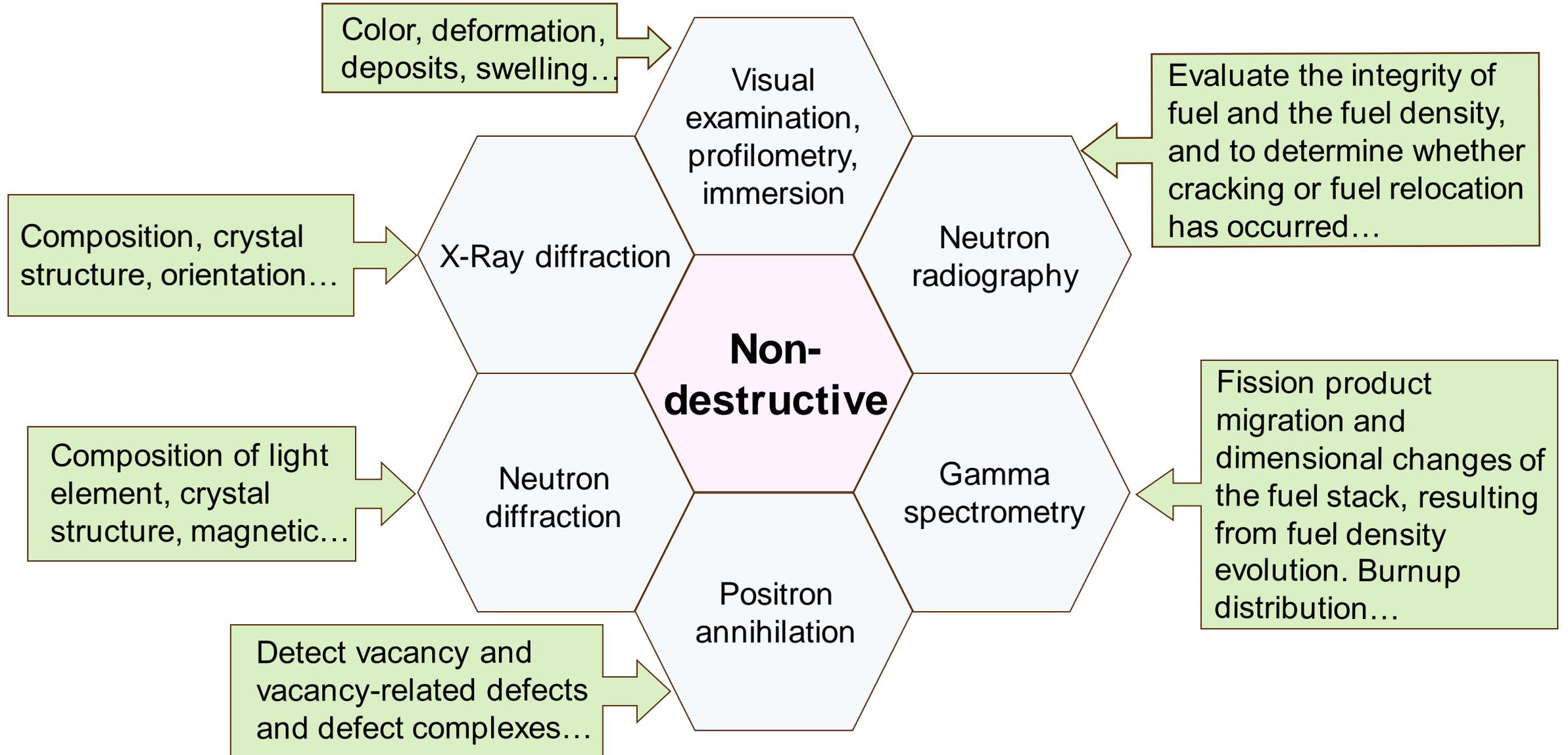
- **Techniques**

- Non-destructive
- Destructive

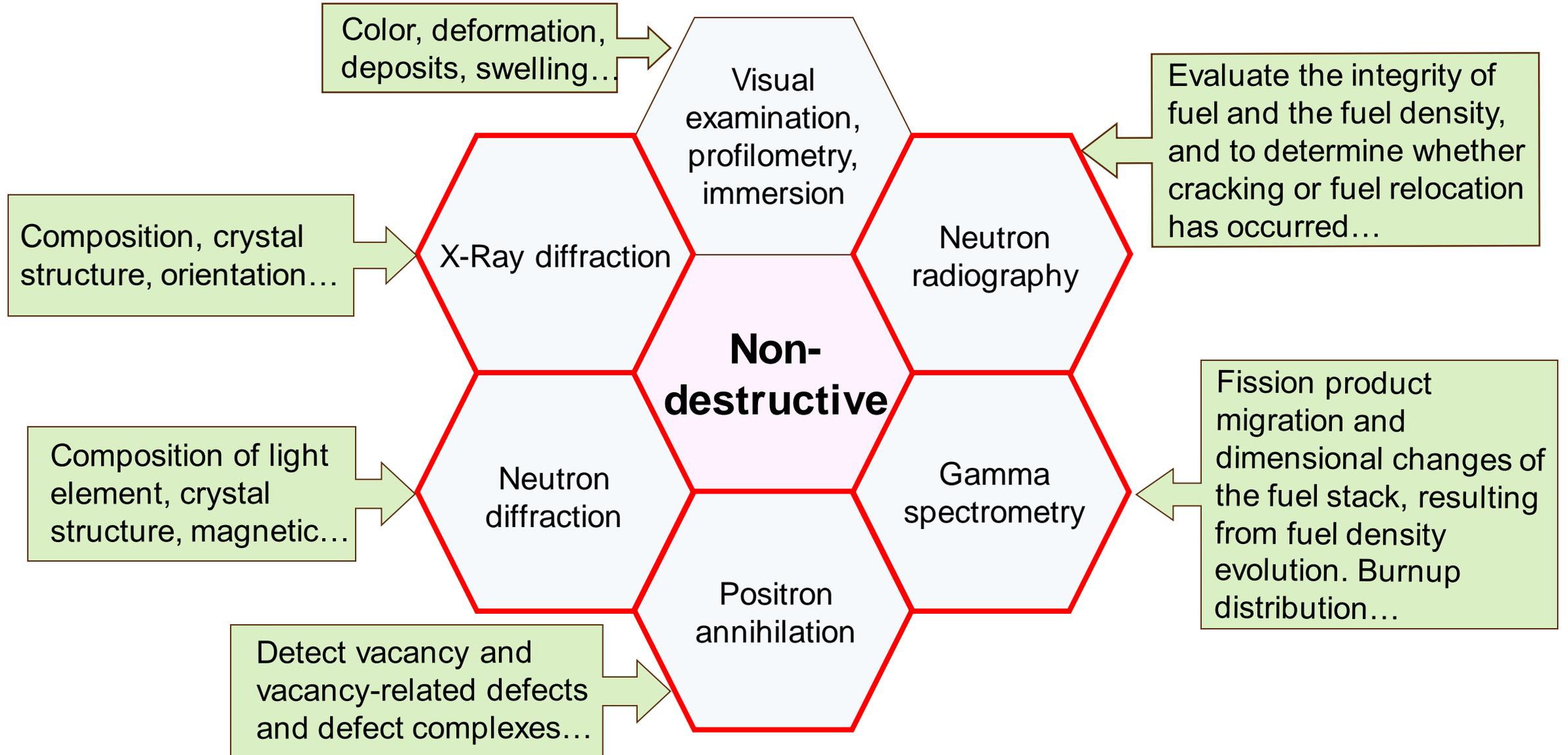
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# Non-Destructive PIE Methods

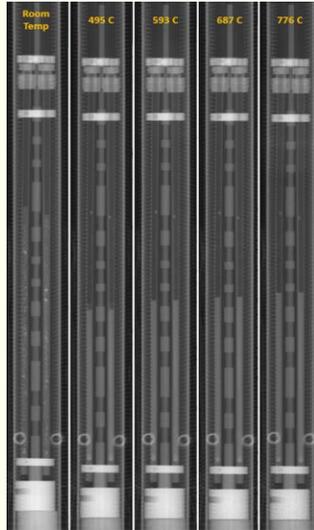


# Non-Destructive PIE Methods

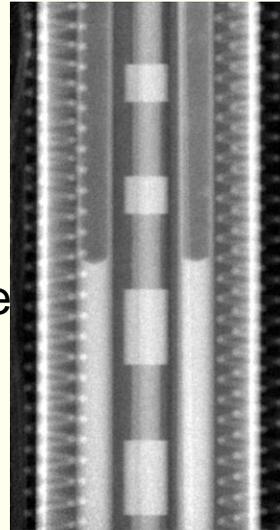


# Neutron Radiography

Thermophysical measurements of salts

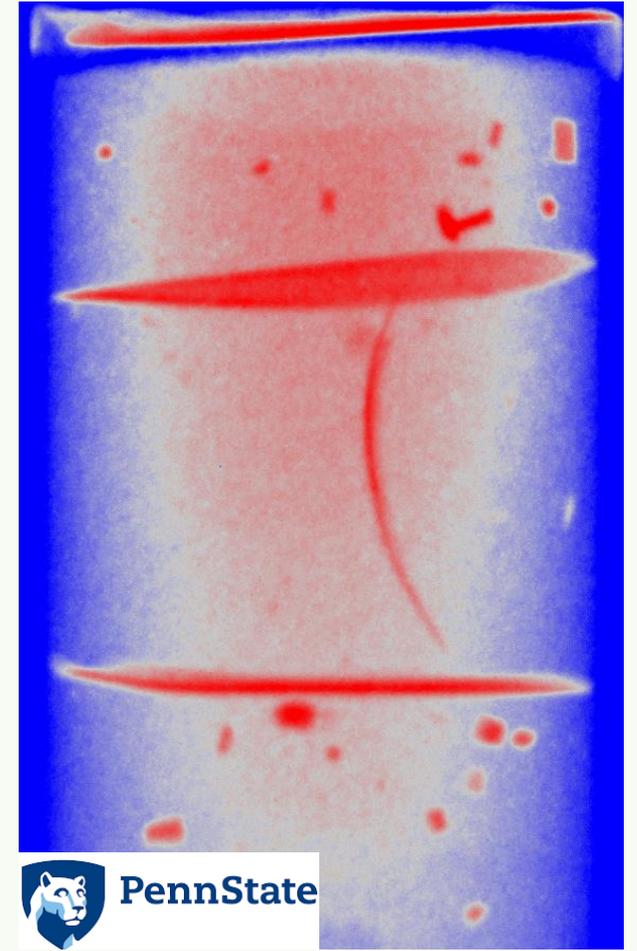


Volume expansion  
PuCl<sub>3</sub>-base samples

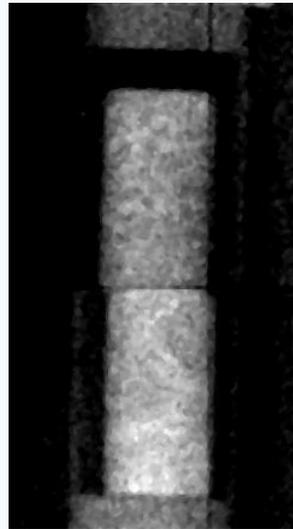


Salt is solidifying during cooling

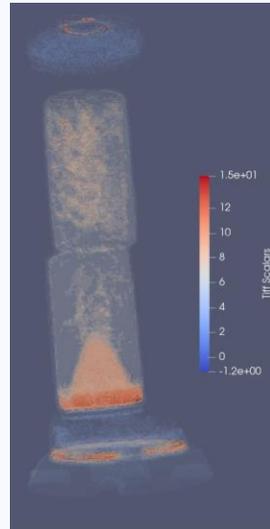
## Visualization of particles in sand



*In situ* H-distribution measurements

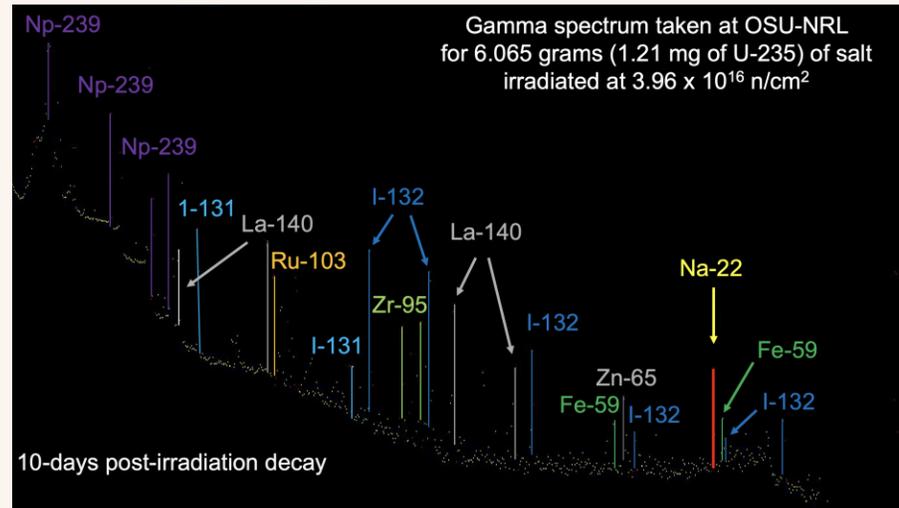


Hydrogen movement (based on temp. gradients)

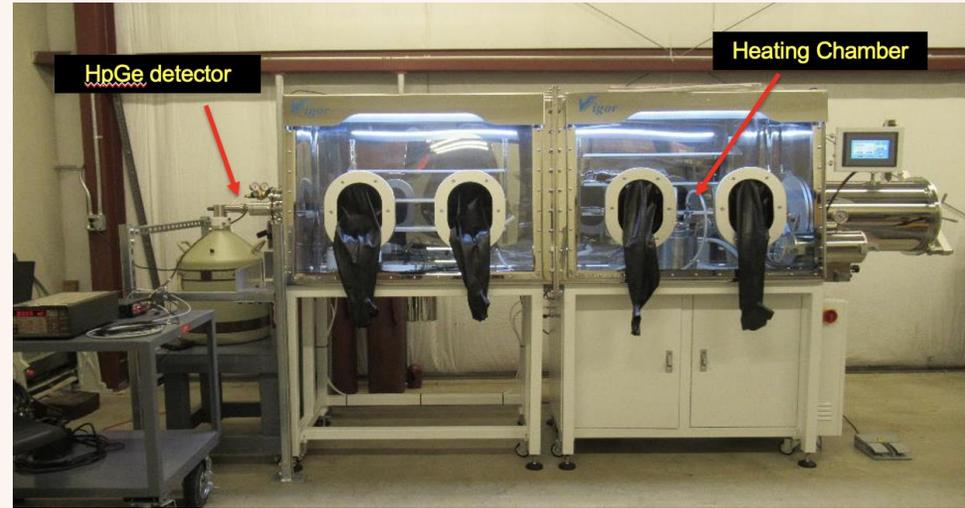


CT scan of Hydrogen after heating

# Gamma Spectrometry



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



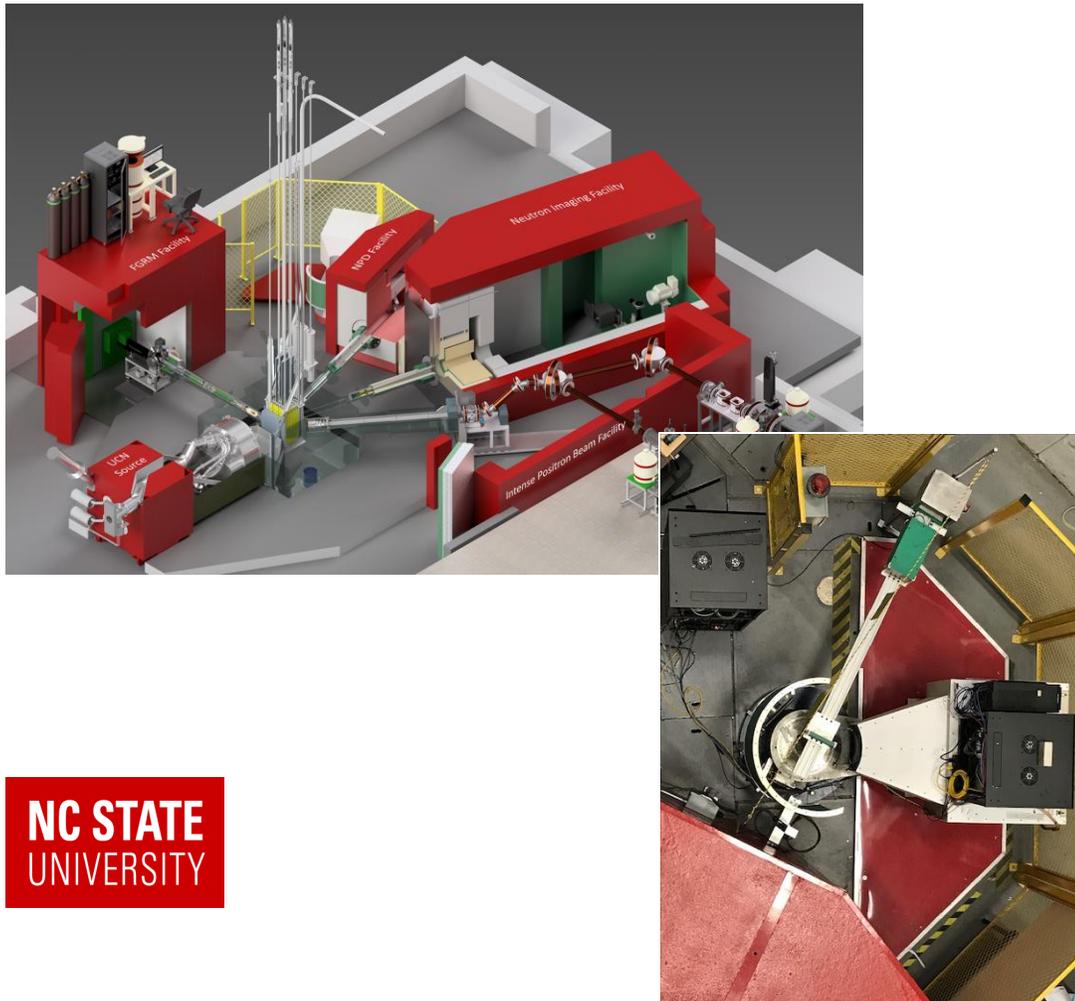
Fuel Salt Processing and Gamma Counting Facility



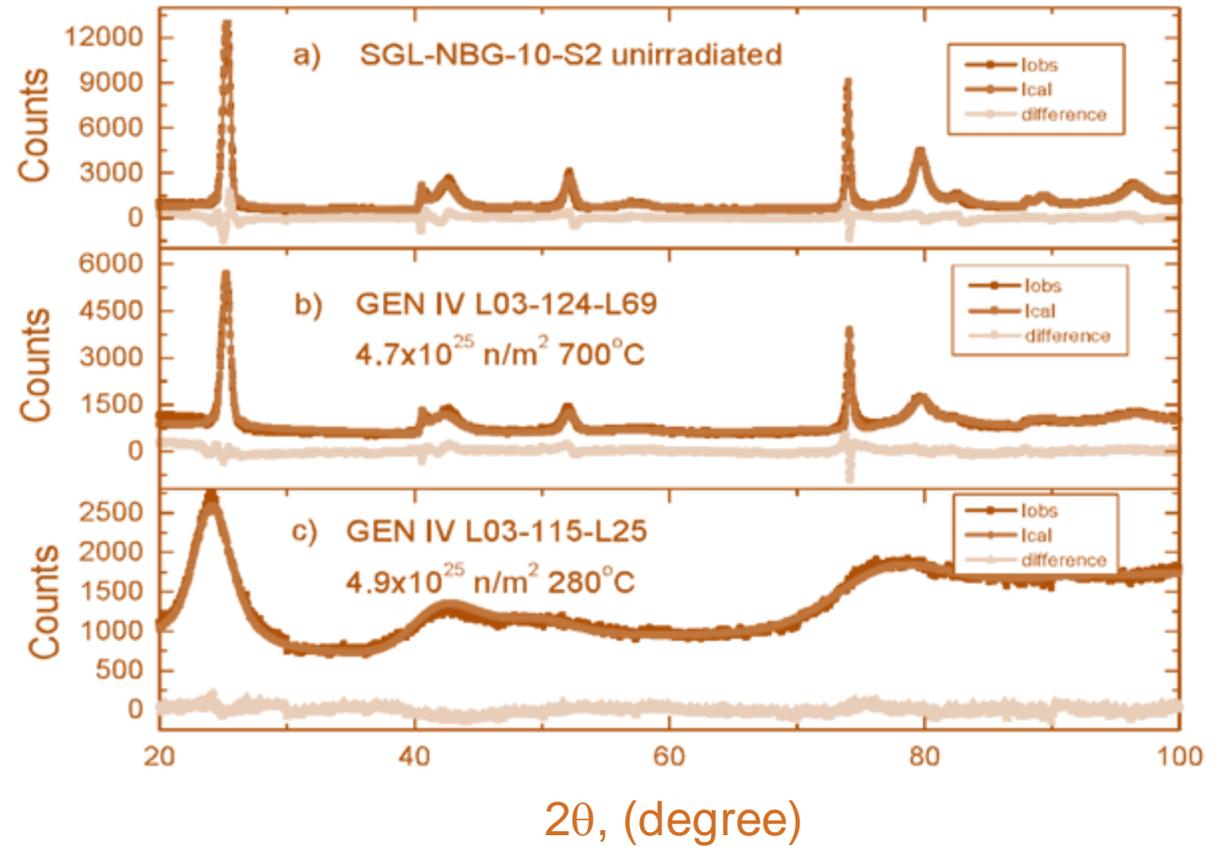
- The gamma counting measured a full gamma spectrum of shorted lived fission products, including those gaseous species.
- The first group who has irradiated the fuel salt (depleted U-235)

Courtesy of Raymond Cao

# Neutron Diffraction



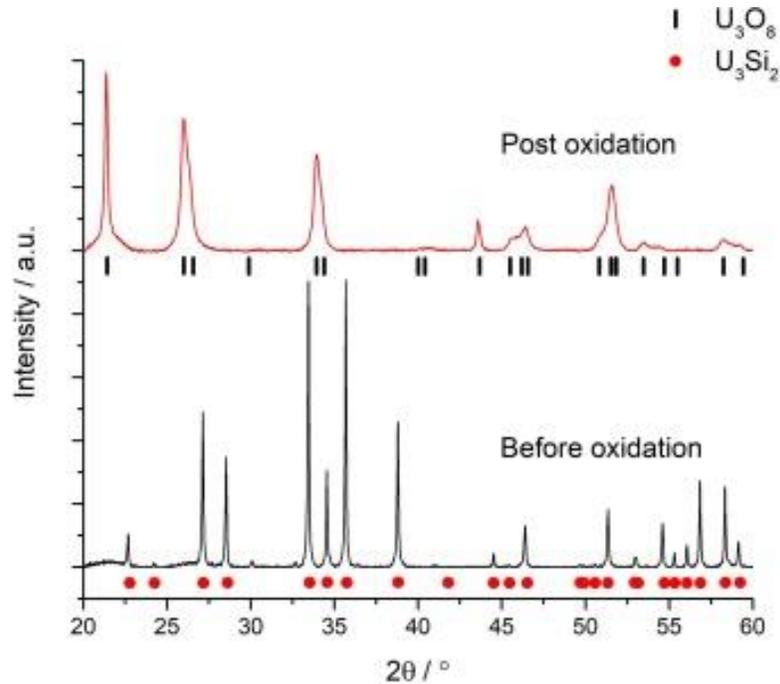
Neutron diffractometer



Impact of microstructure on neutron thermalization in graphite

# X-ray Diffraction

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



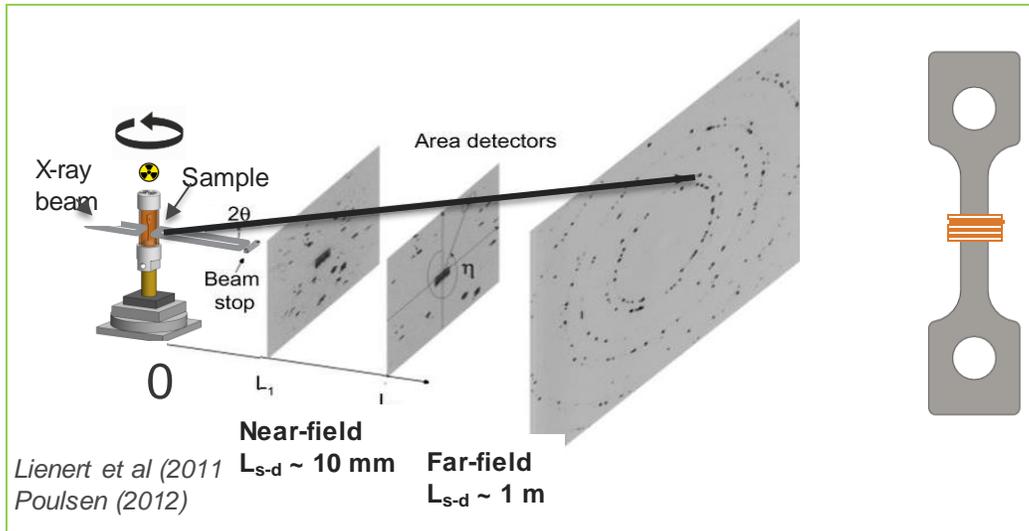
XRD patterns of  $U_3Si_2$  before oxidation and after oxidation showing the formation of  $U_3O_8$ .

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

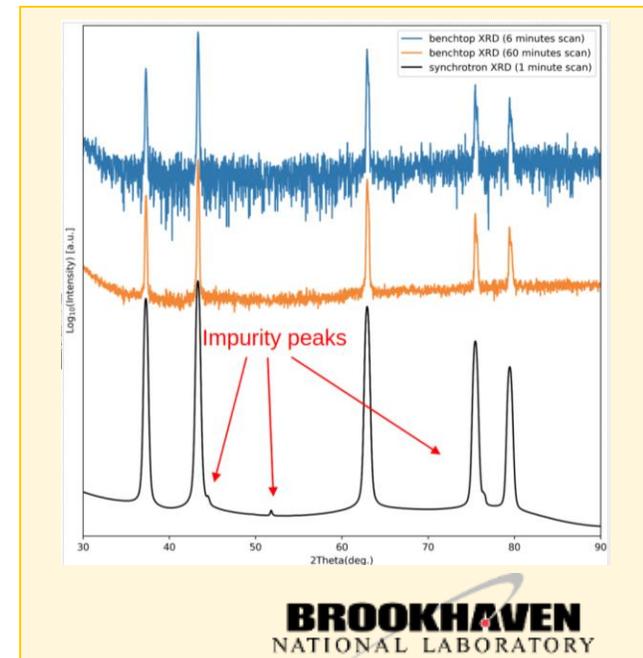


Courtesy of Catou Cmar

# X-ray Diffraction: Beamline



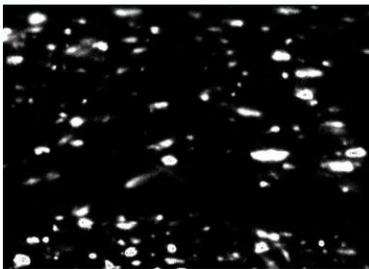
- *In situ* dynamic measurements
- Non-destructive 3D characterization
- Multiple length scales in one experiment



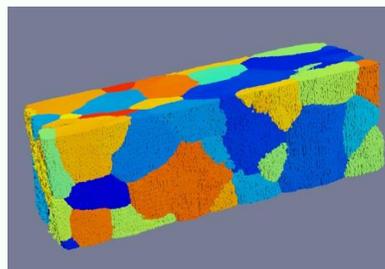
## Near-field HEDM (nf-HEDM):

- Reconstructs grain morphology and crystallographic orientation **voxel by voxel** (1-2  $\mu\text{m}$  resolution).

Detector images as sample rotates:



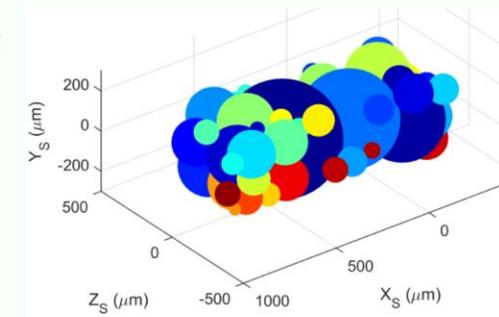
EBSD-like reconstruction



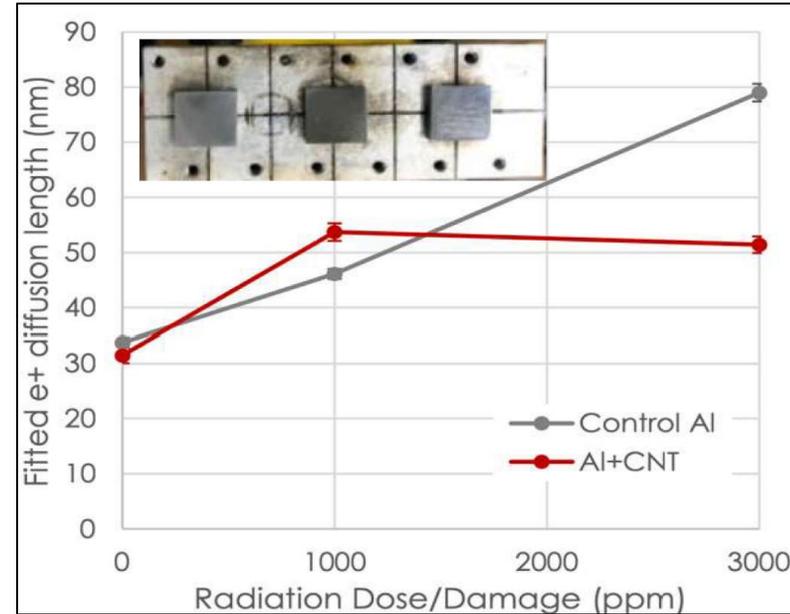
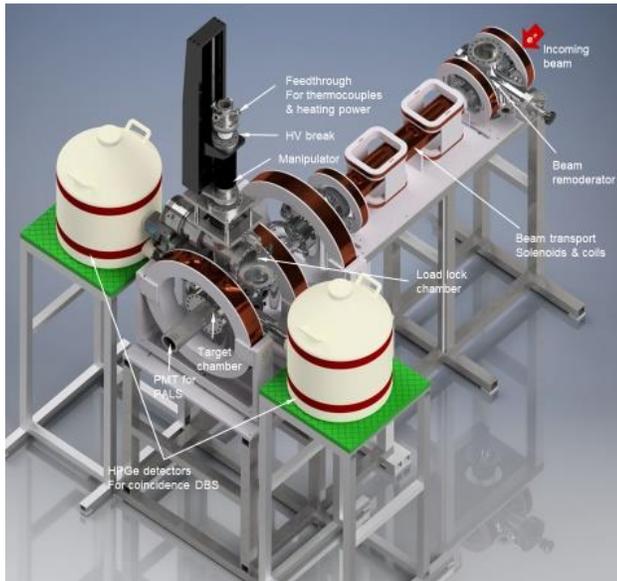
## Far-field HEDM (ff-HDEM):

- Reconstructs centroids, crystallographic orientations, and elastic strain tensors **domain by domain** (10  $\mu\text{m}$  resolution).

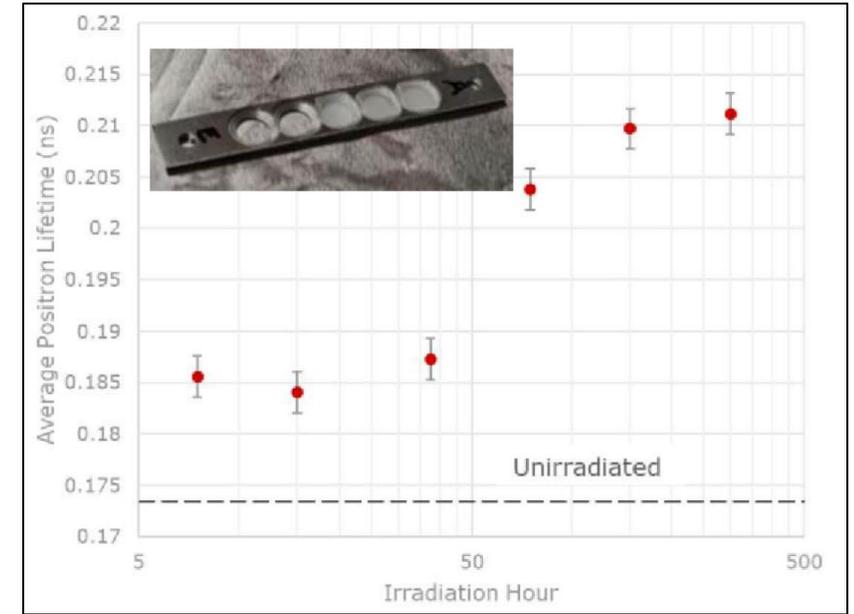
Detector images as sample rotates:



# Positron Annihilation



Quantification of porosity in irradiated metal via PAS



Average positron life of Ga<sub>2</sub>O<sub>3</sub> before/after irradiation

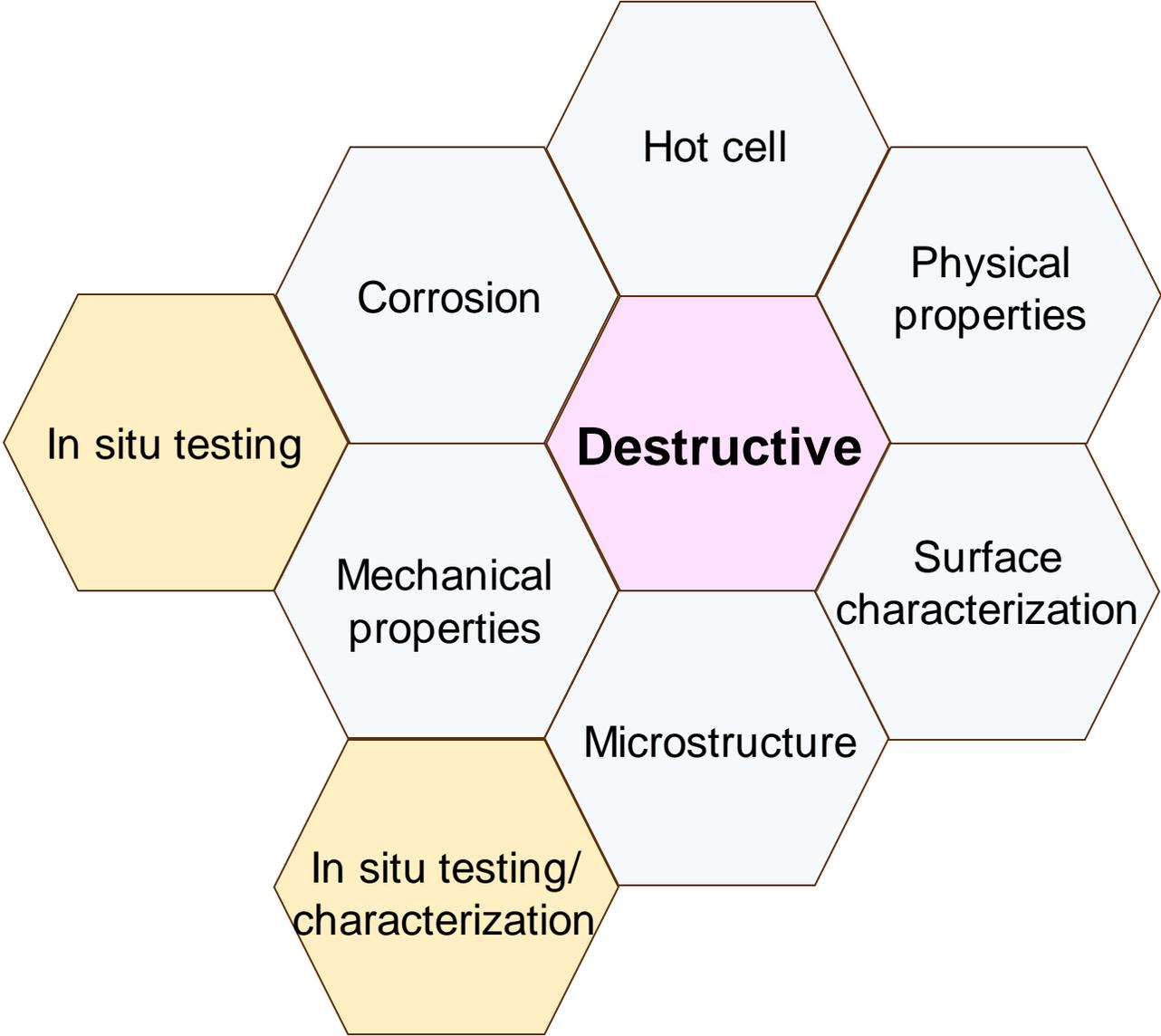


Courtesy of Ayman Hawari

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  - Surface characterization
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# Destructive PIE Characterization Capabilities



# Hot Cell Facilities



# 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)

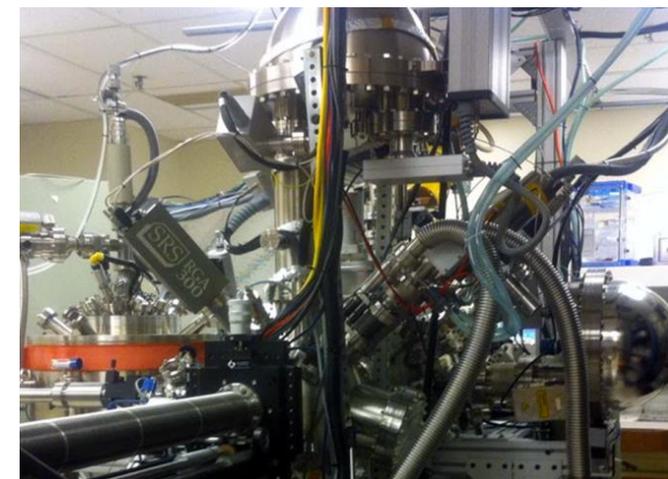
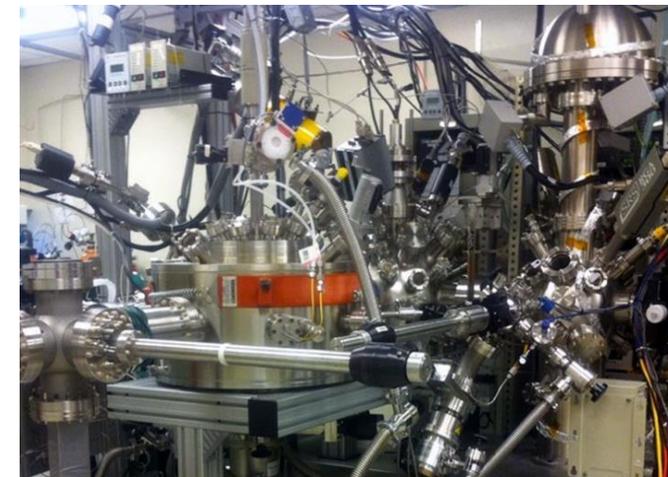


Simultaneous thermal analyzer, dilatometer, LFA, DSC inside of the fresh fuels glovebox (FFG)

# Surface Characterization

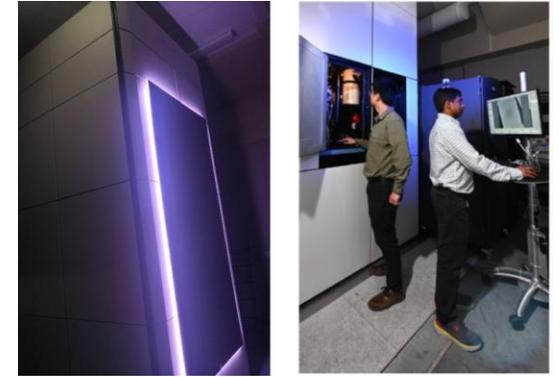
## At Interaction of Materials with Particles and Components Testing (IMPACT)

- 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<sub>2</sub> - 1200° 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)



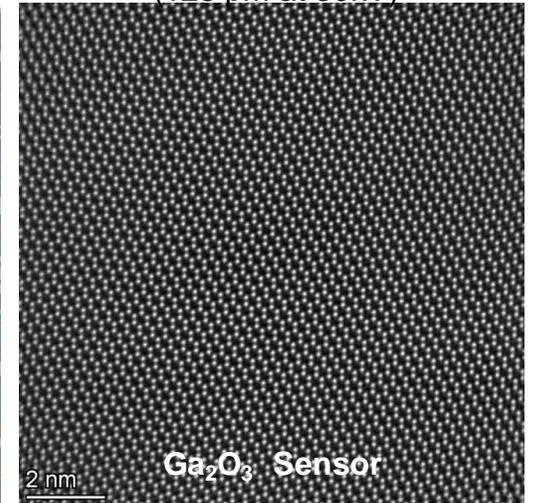
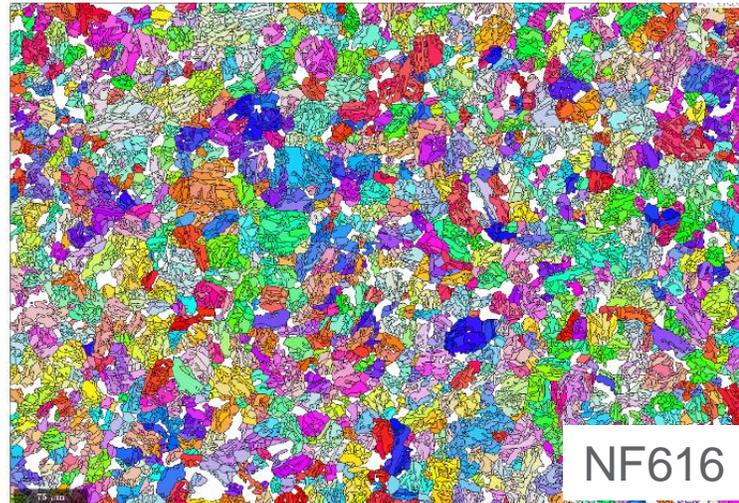
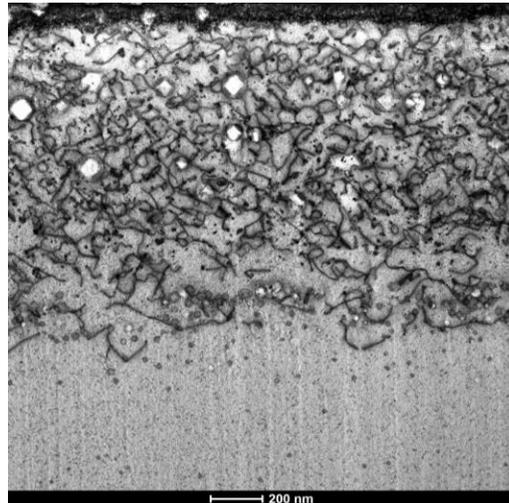
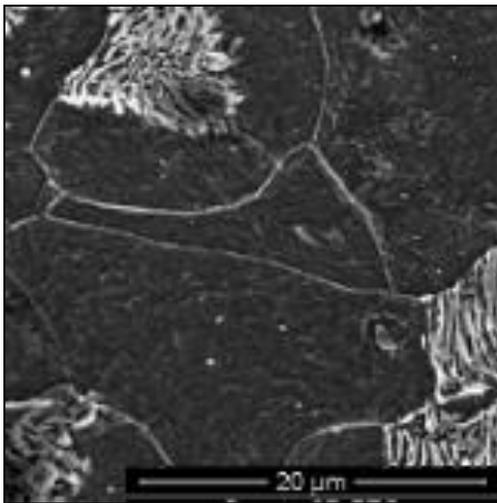
# Microstructure — Morphology

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



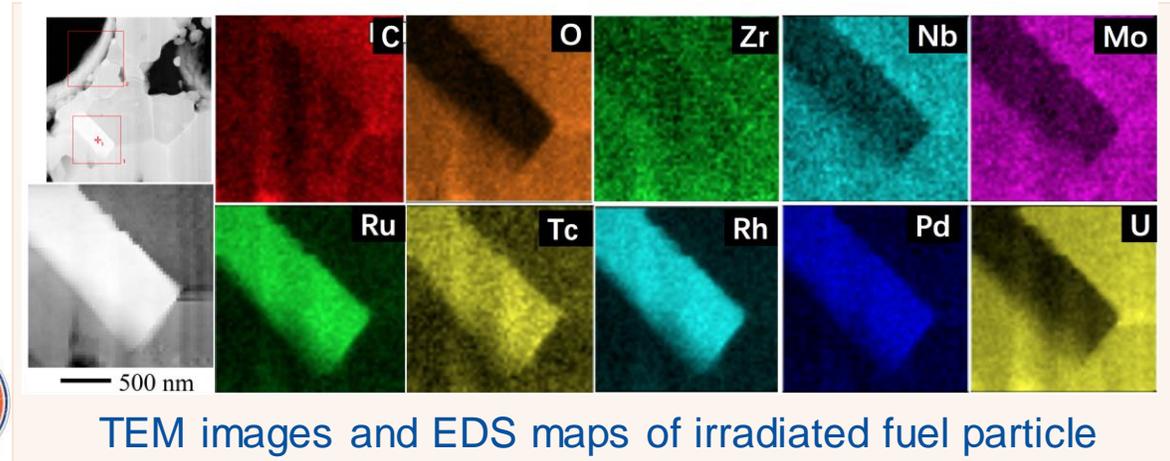
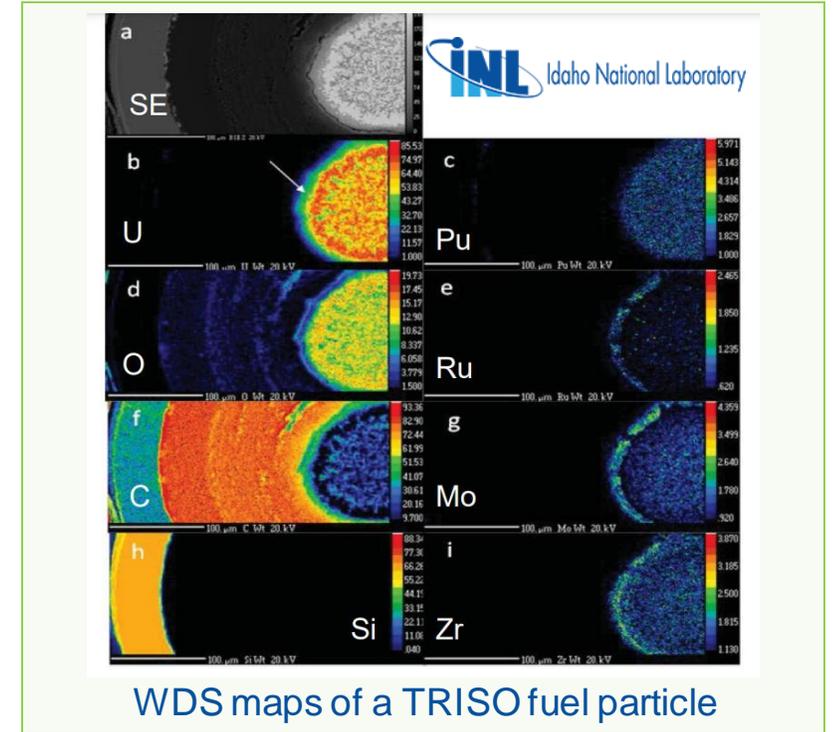
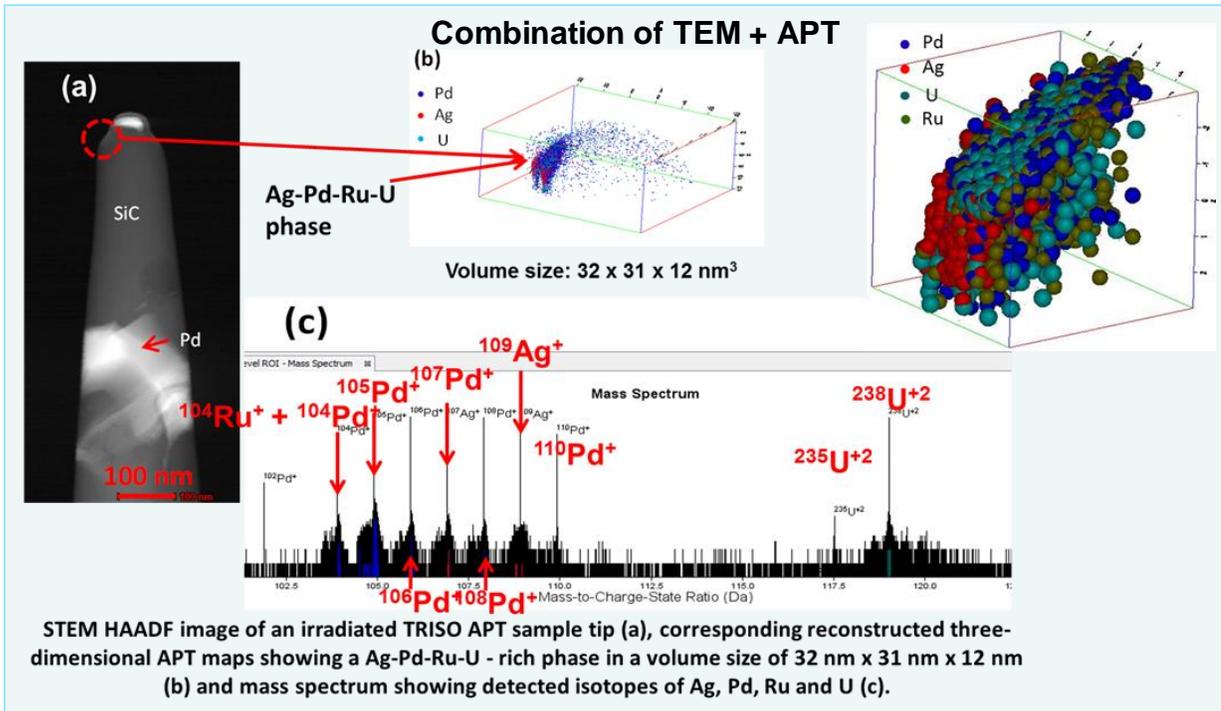
**Spectra 300 STEM**

STEM resolution 50 pm  
(125 pm at 30kV)



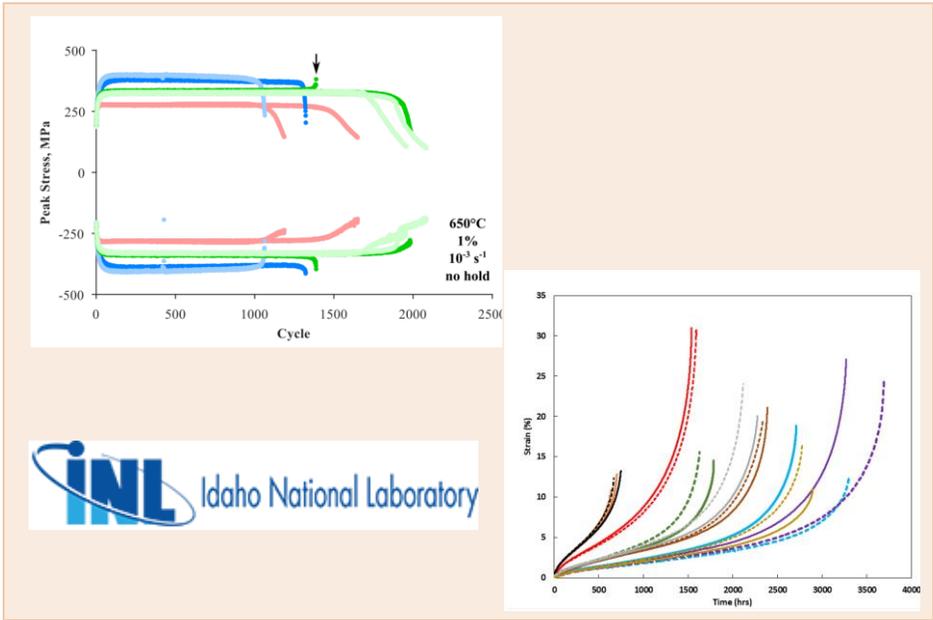
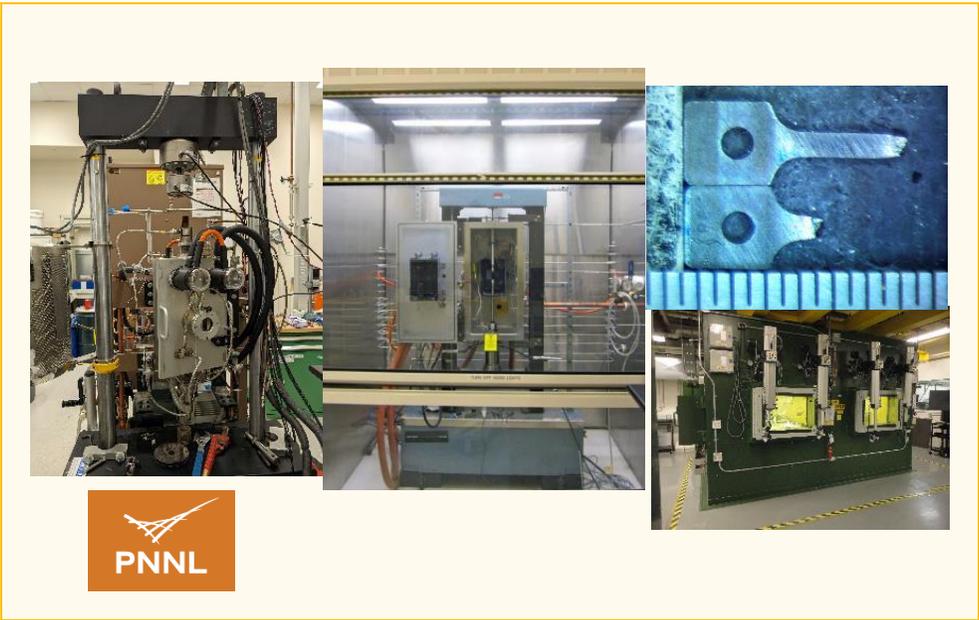
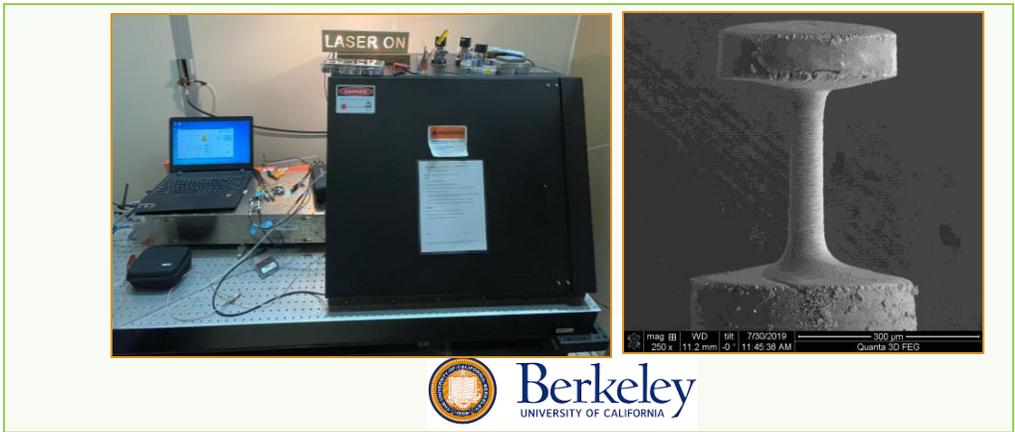
# Microstructure — Composition Analysis

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



# Mechanical Testing

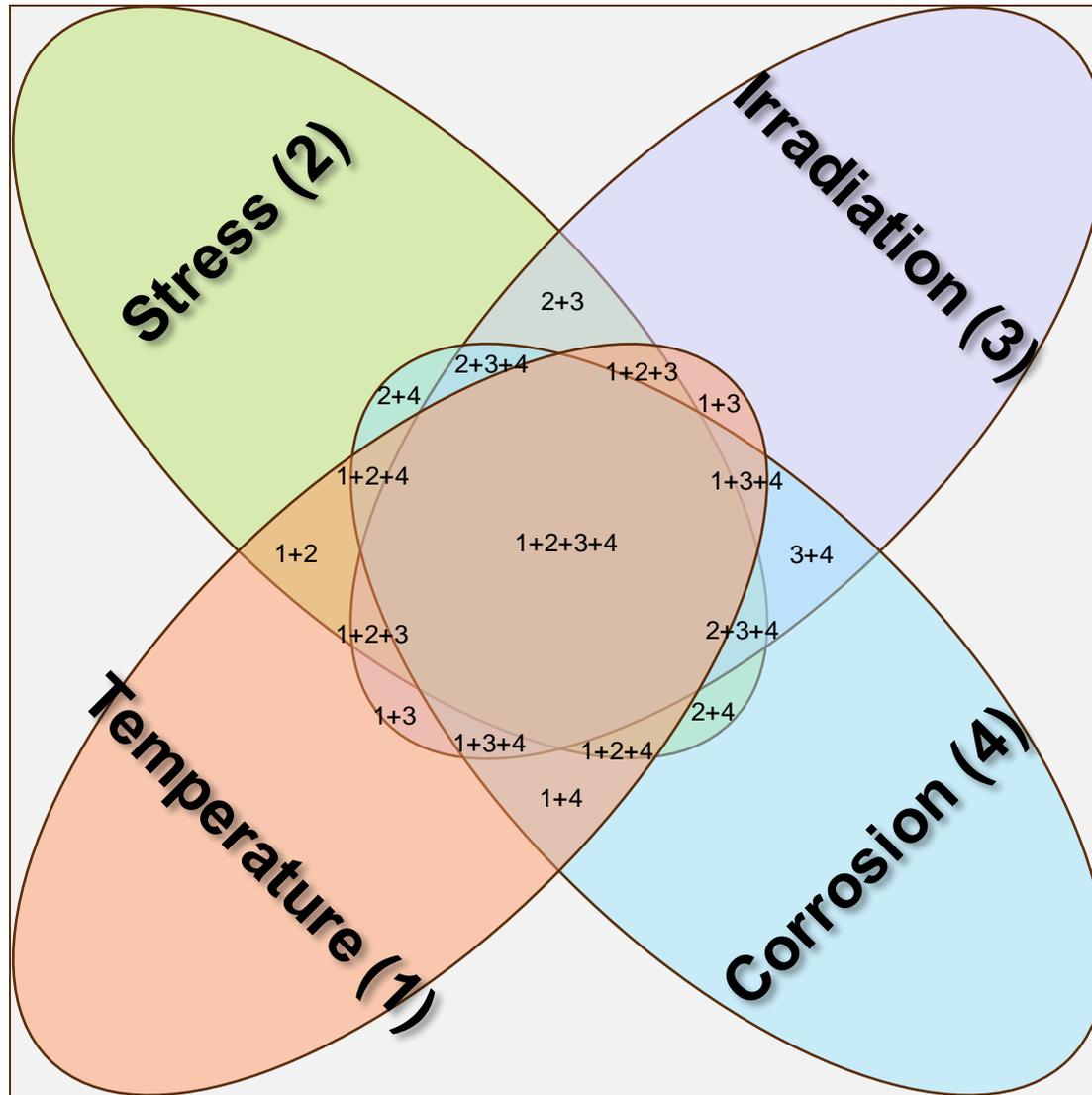
- Hardness/ Bend test/ Tensile/ Creep/ Fatigue/ Compact tension/ Charpy impact (toughness)



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# Accelerated Testing

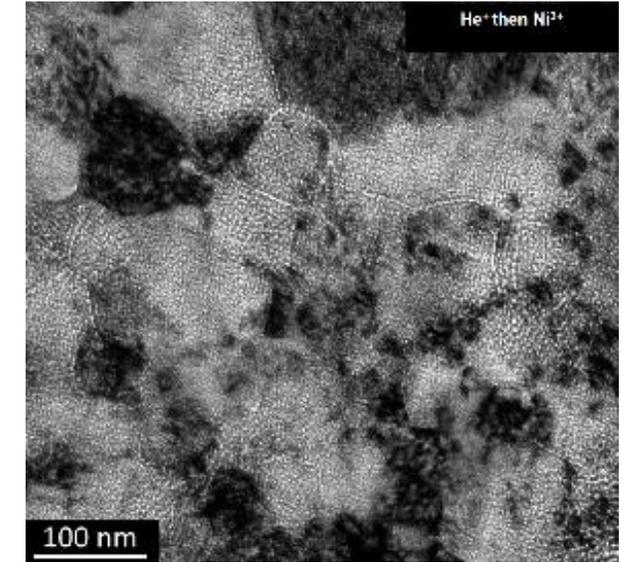
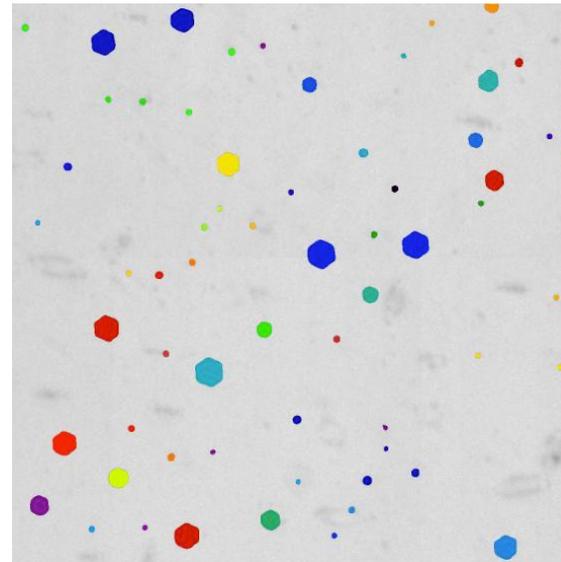
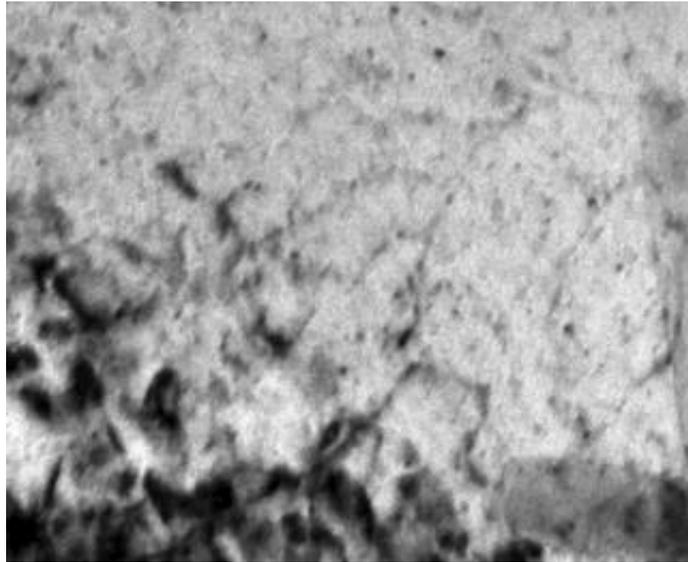


- Testing under conditions in excess of normal service parameters
- Testing under multiple factors at once

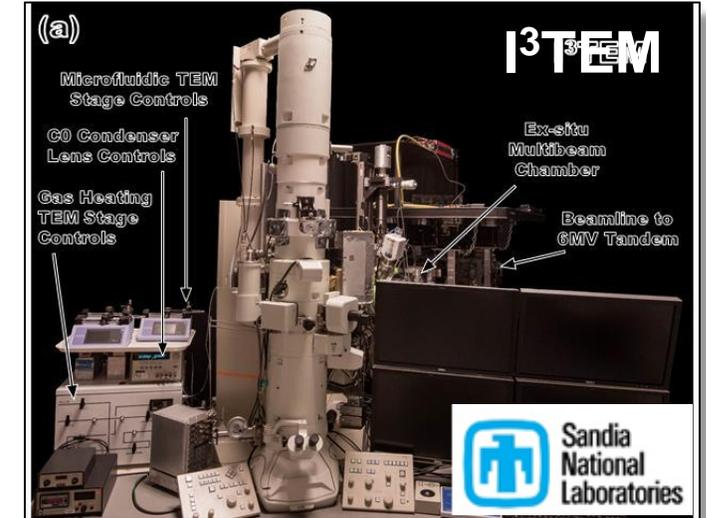
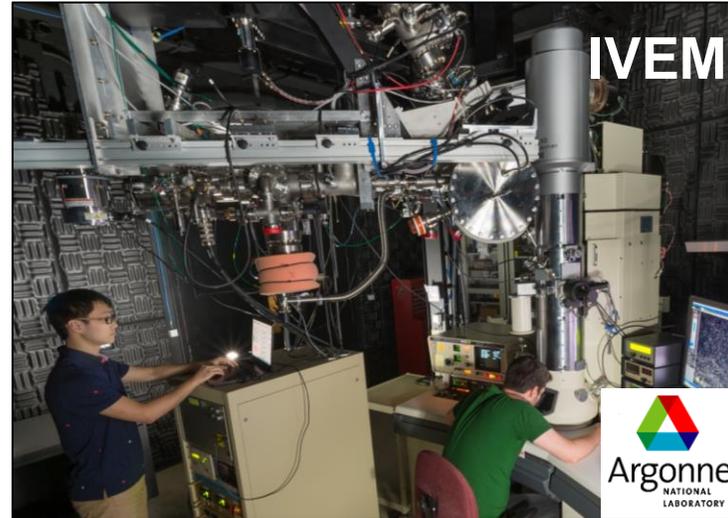
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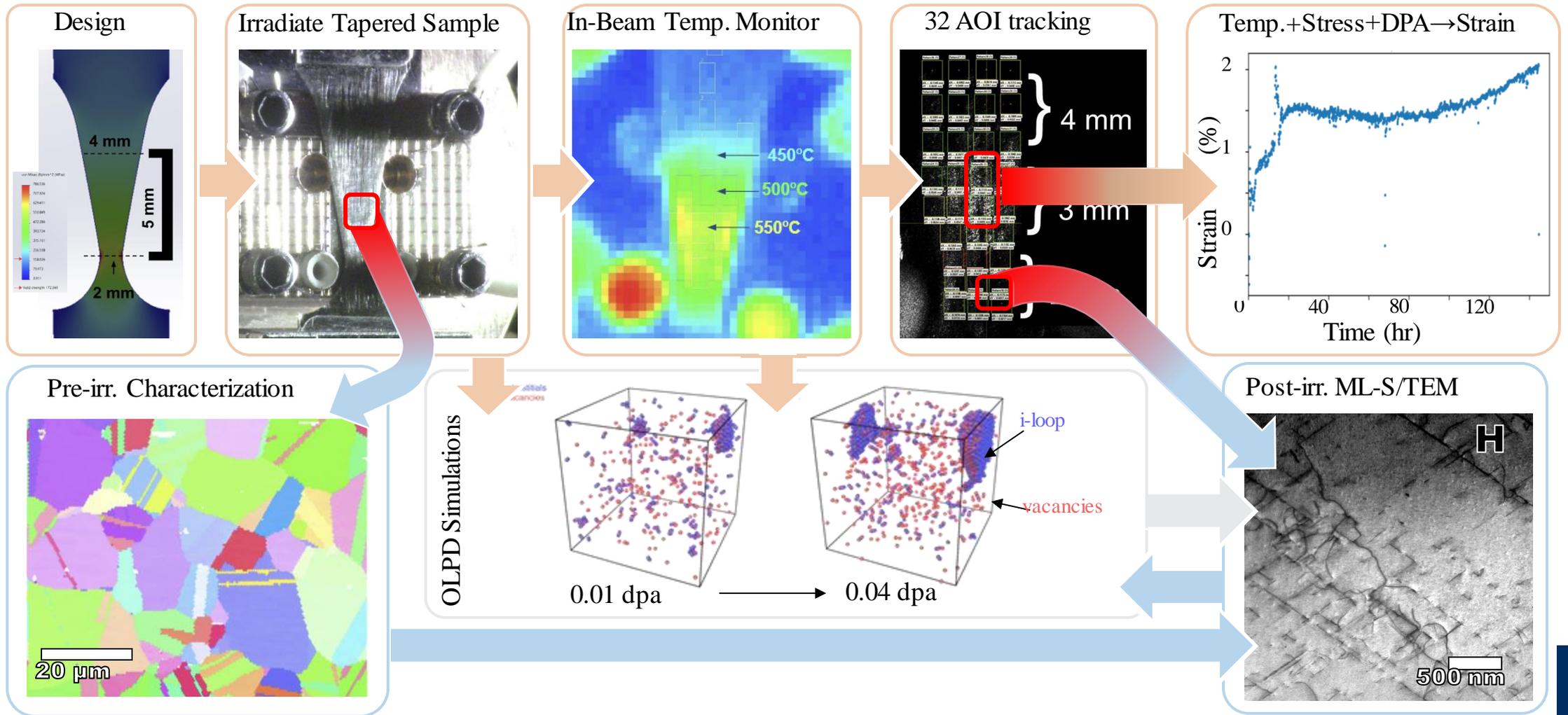
# In Situ Ion Irradiation + Microstructure Characterization



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

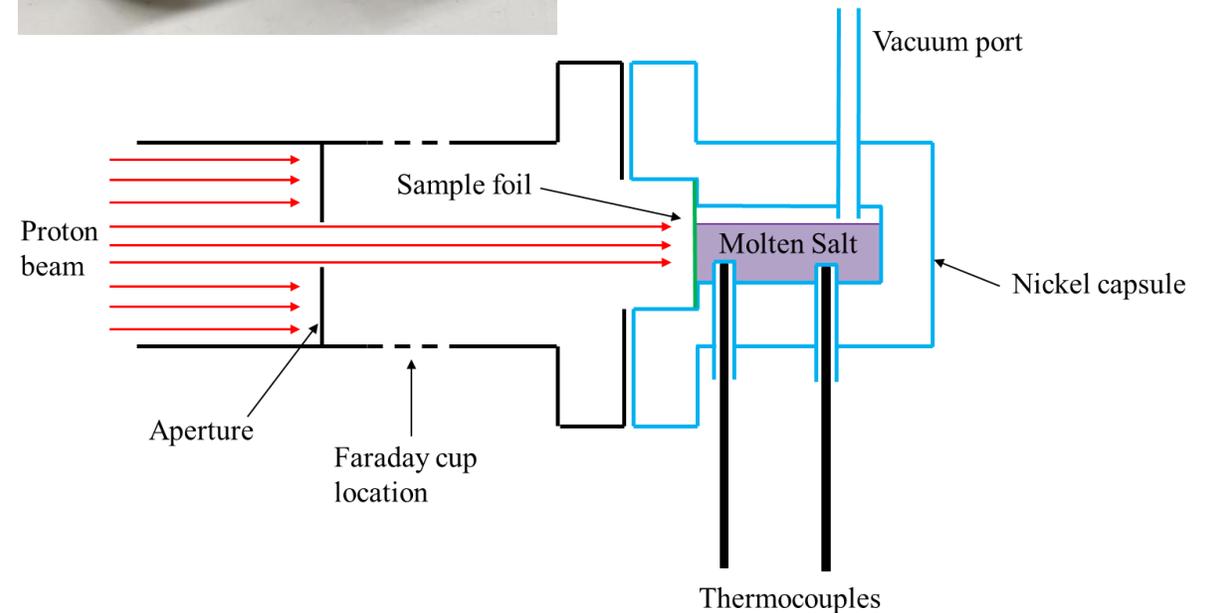


# In Situ Ion Irradiation + Mechanical Testing in MIBL



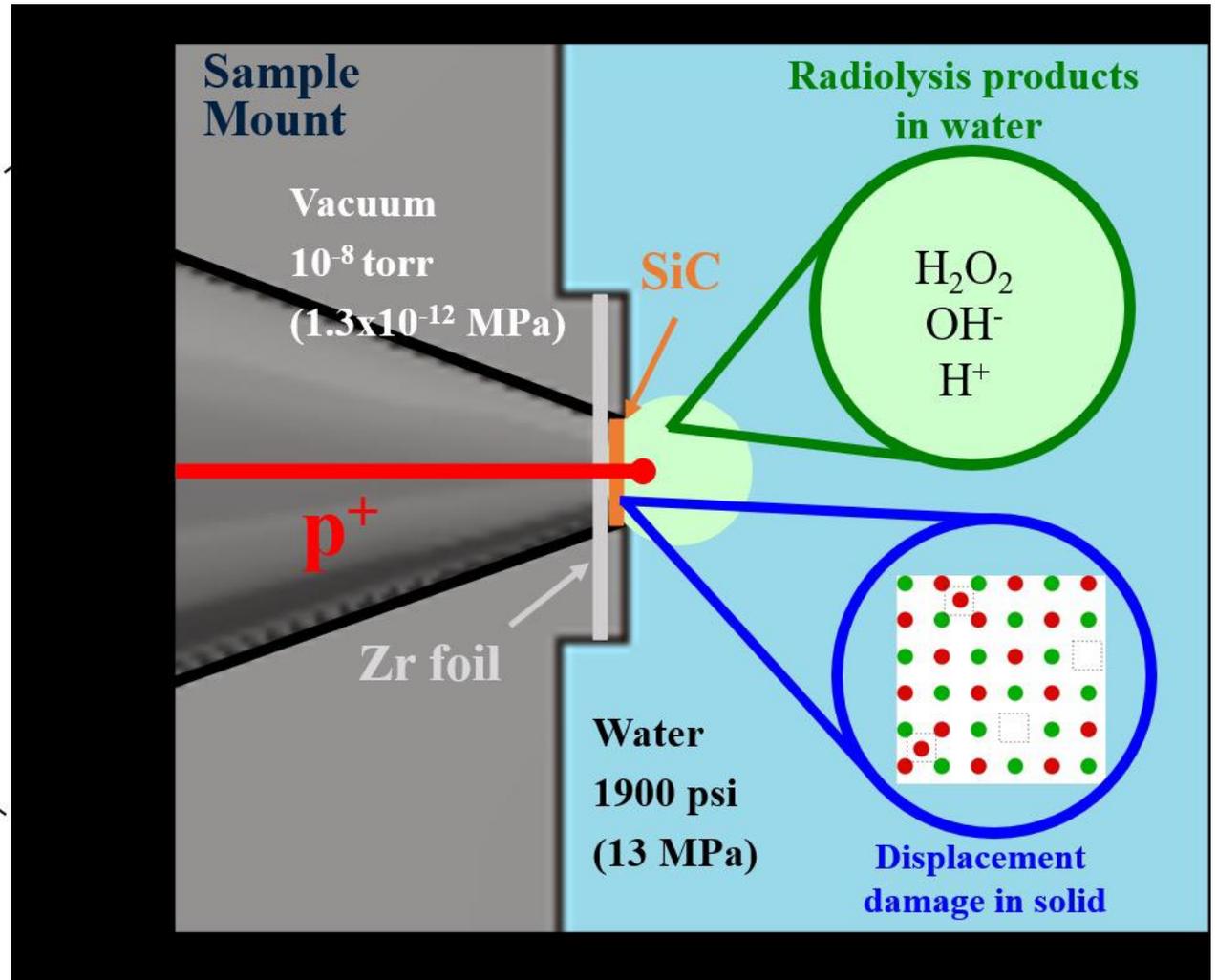
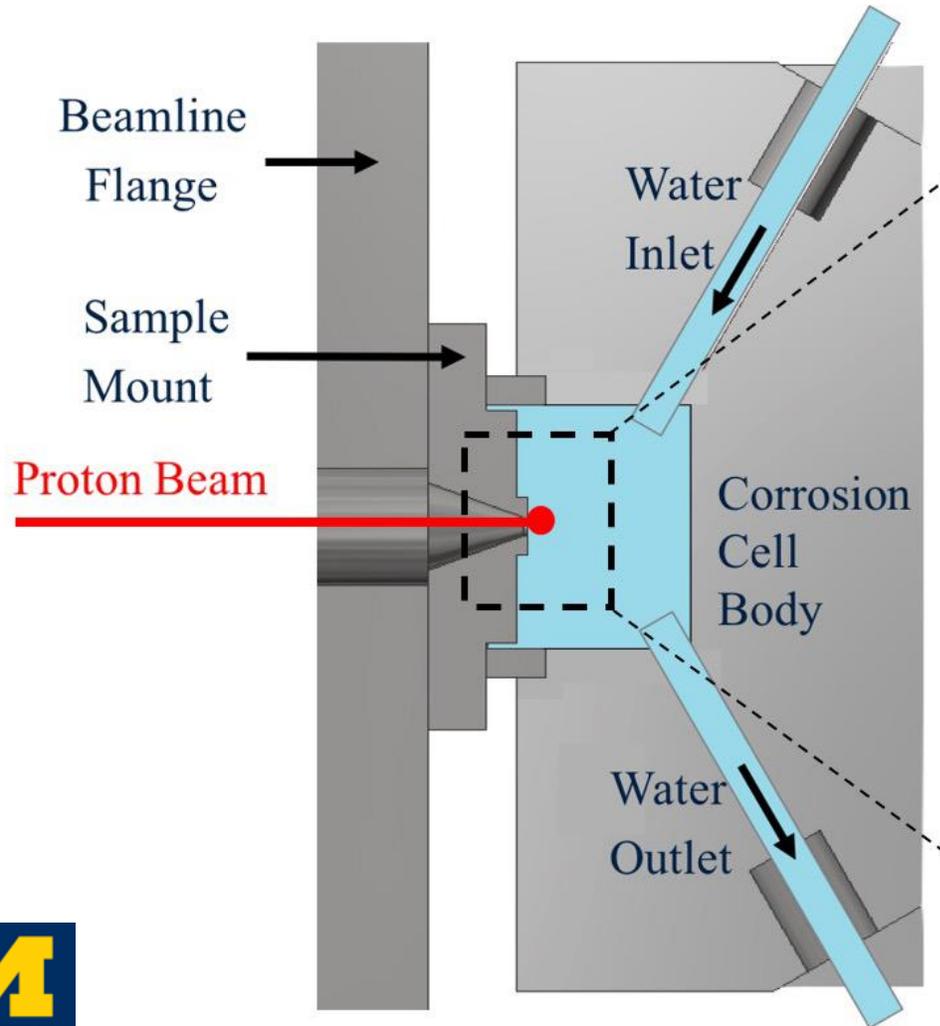
# In Situ Ion Irradiation + Molten Salt Corrosion

- 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

# In Situ Proton Irradiation + Corrosion in MIBL



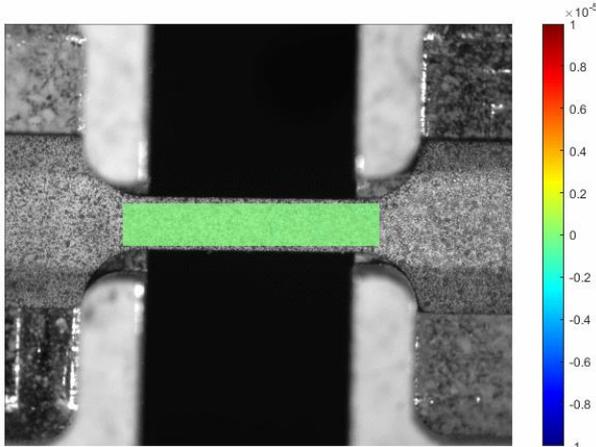
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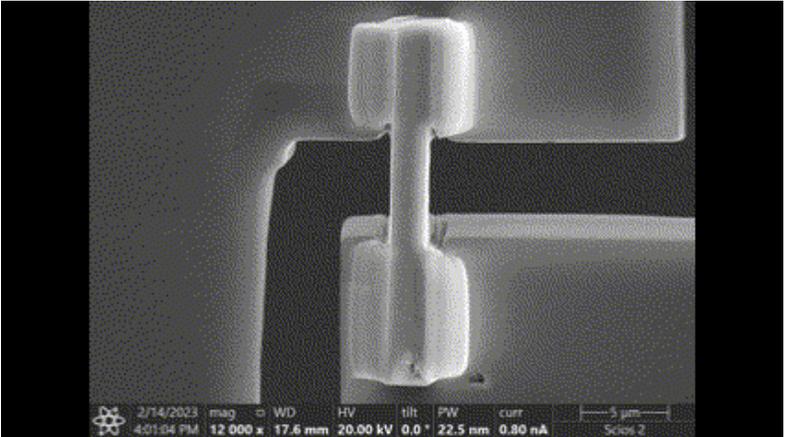
# In situ Mechanical Testing + Microstructure Characterization



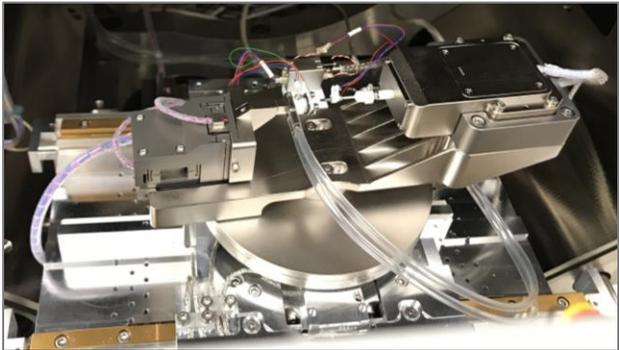
Argonne NATIONAL LABORATORY



INL Idaho National Laboratory

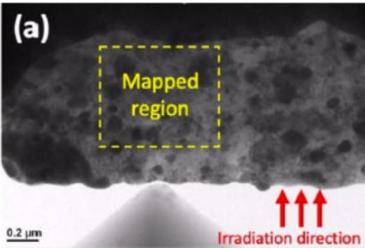
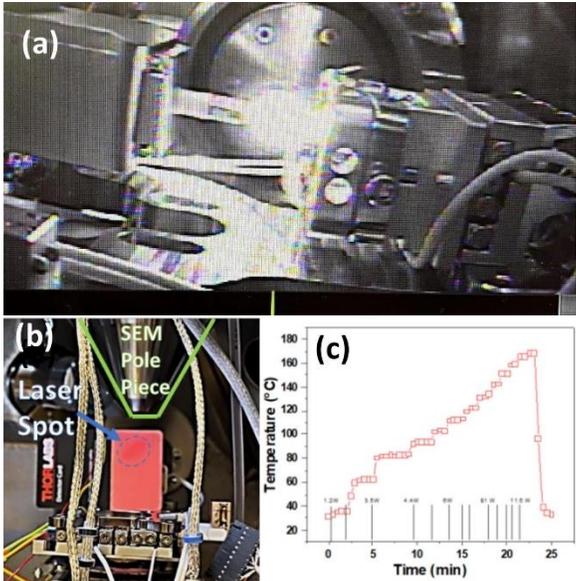


Berkeley UNIVERSITY OF CALIFORNIA

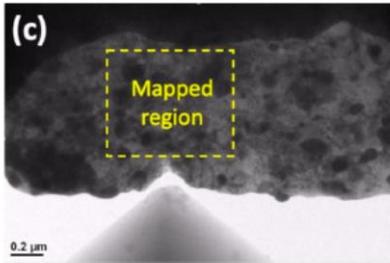
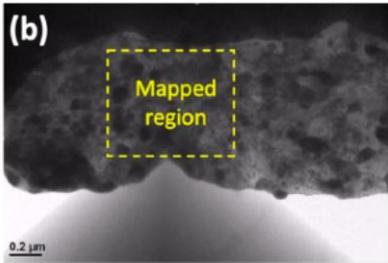


UNIVERSITY OF FLORIDA

Sandia National Laboratories



CAES Center for Advanced Energy Studies



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# *In situ* Stress + Corrosion: IASCC

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



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- 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.



**NSUF**

Nuclear Science  
User Facilities

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