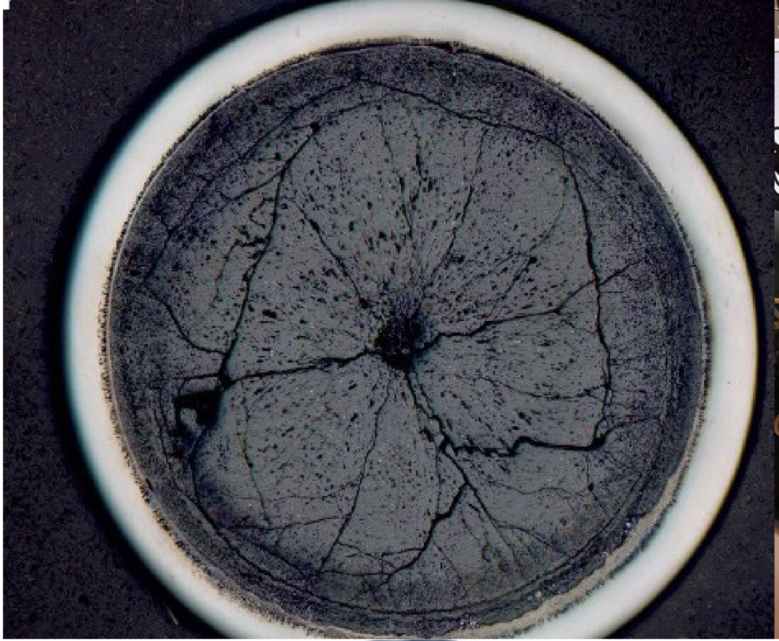
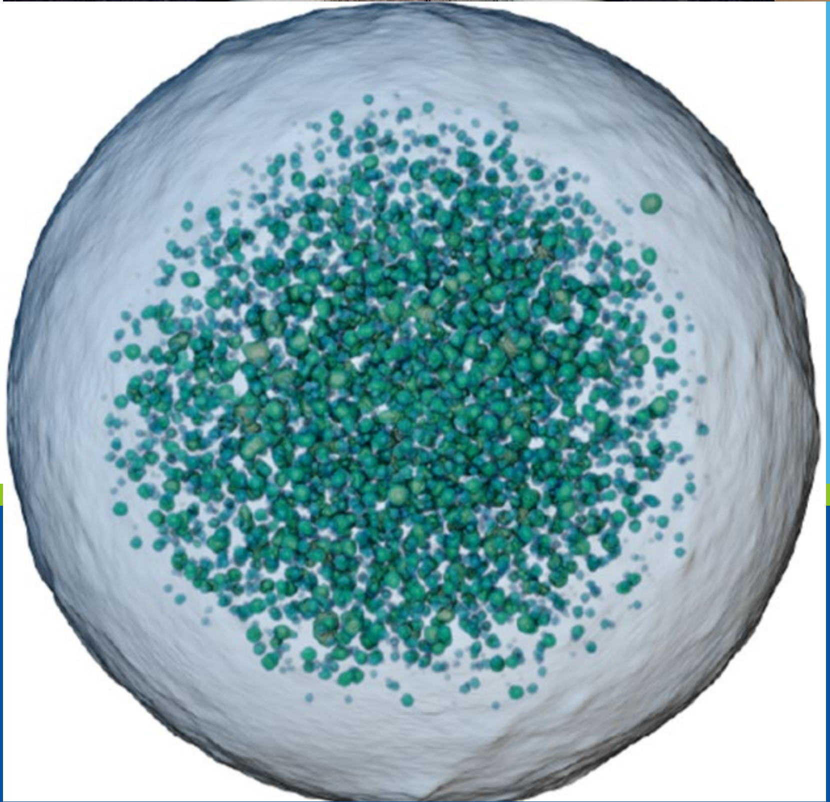


Daniel Murray

Sr. Manager

Materials Characterization Department



Fundamentals of Post-Irradiation Examination

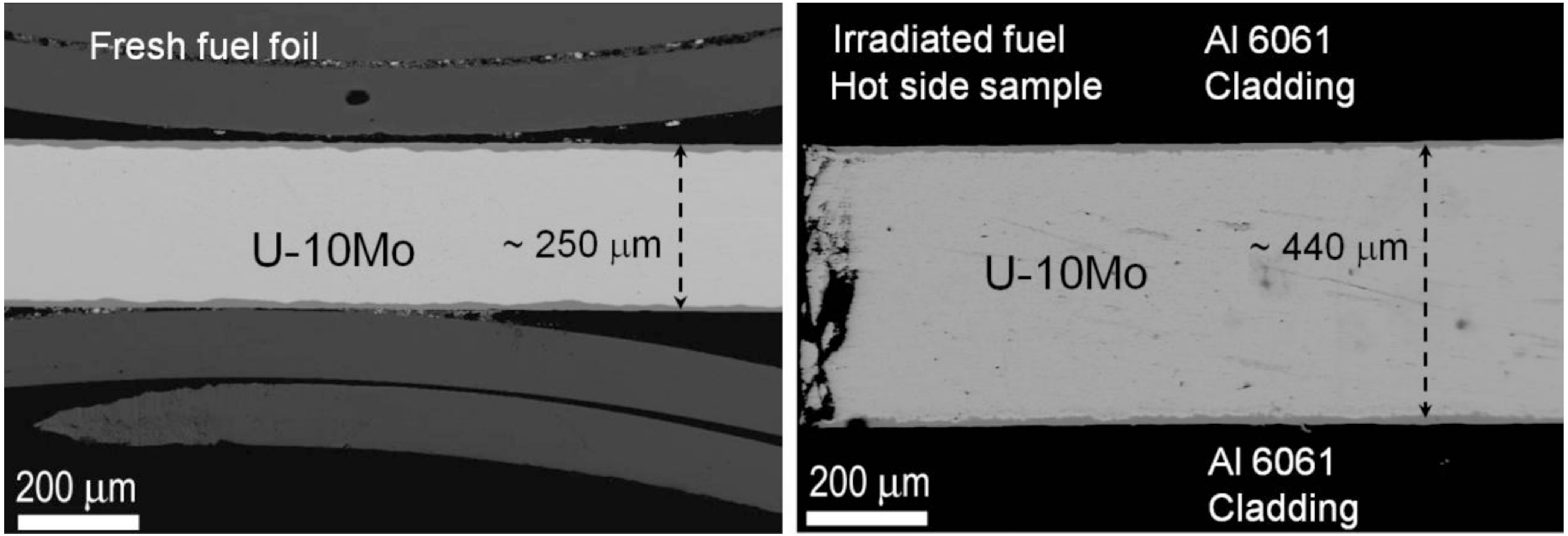
Battelle Energy Alliance manages INL for the U.S. Department of Energy's Office of Nuclear Energy

Idaho National Laboratory

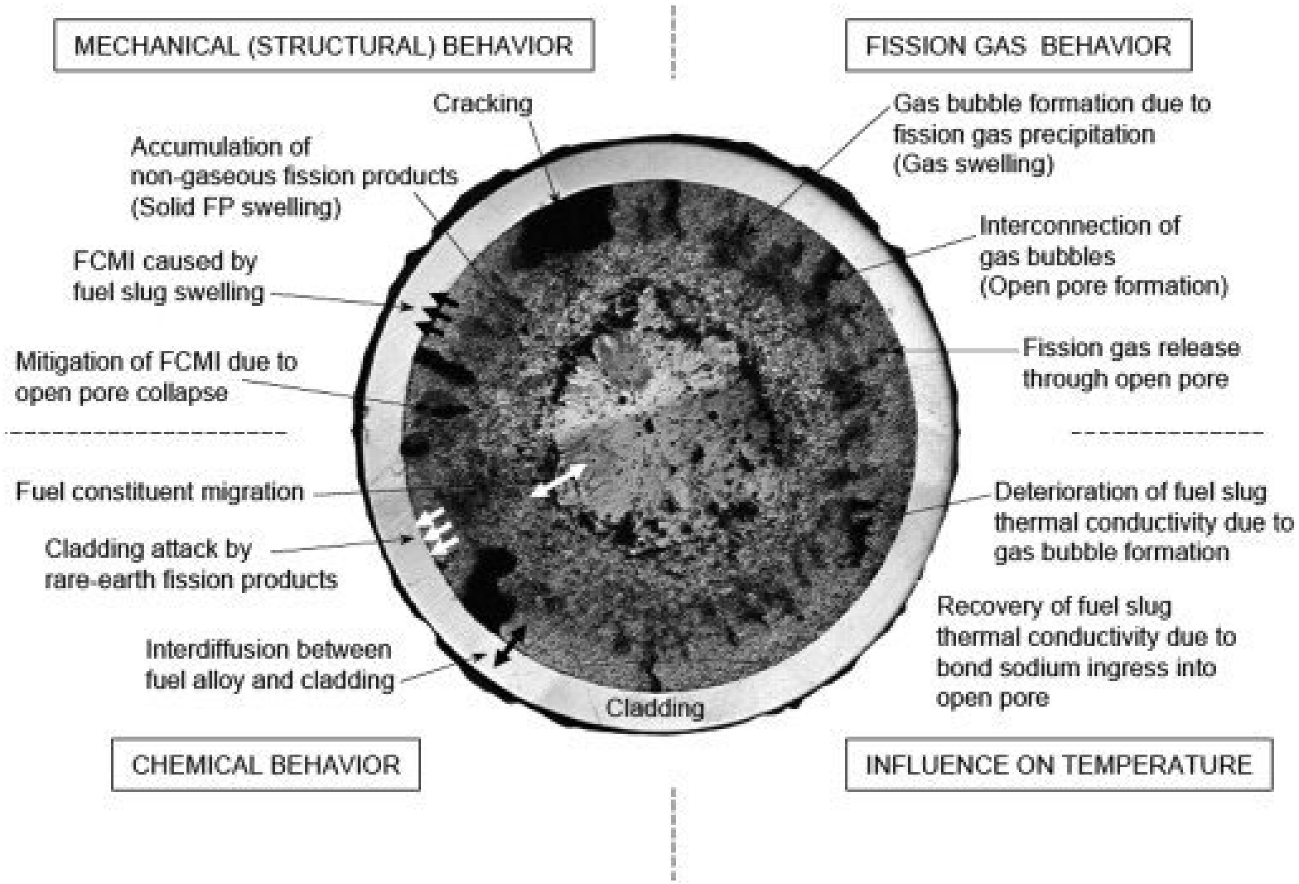
INL/MIS-25-86017

What is Post-Irradiation Examination (PIE)?

Examinations conducted on irradiated material to understand physio-chemical changes that can be related back to in-reactor performance

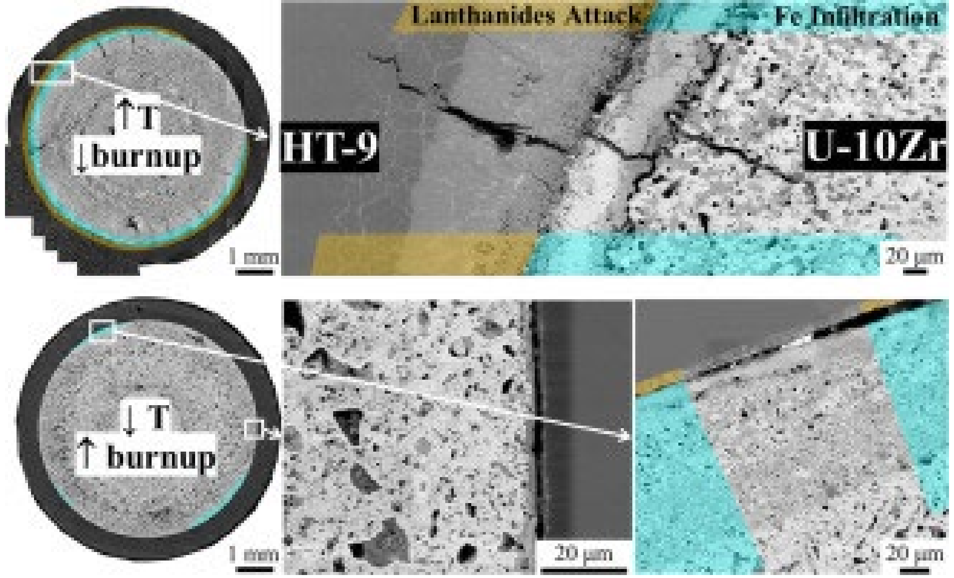


Nuclear fuel performance

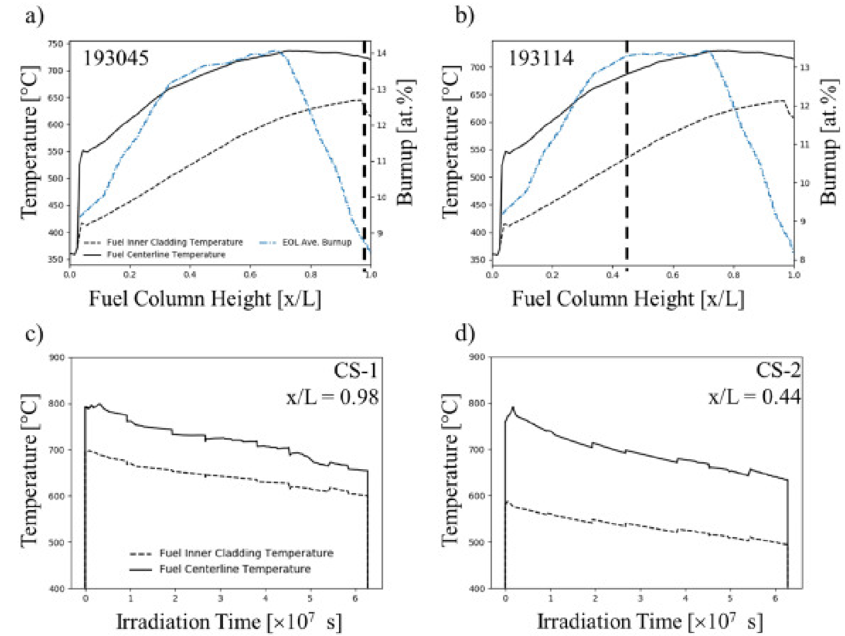


T. Sofu, Nuclear Engineering and Technology (2015), doi:10.1016/j.net.2015.03.004

Experimental



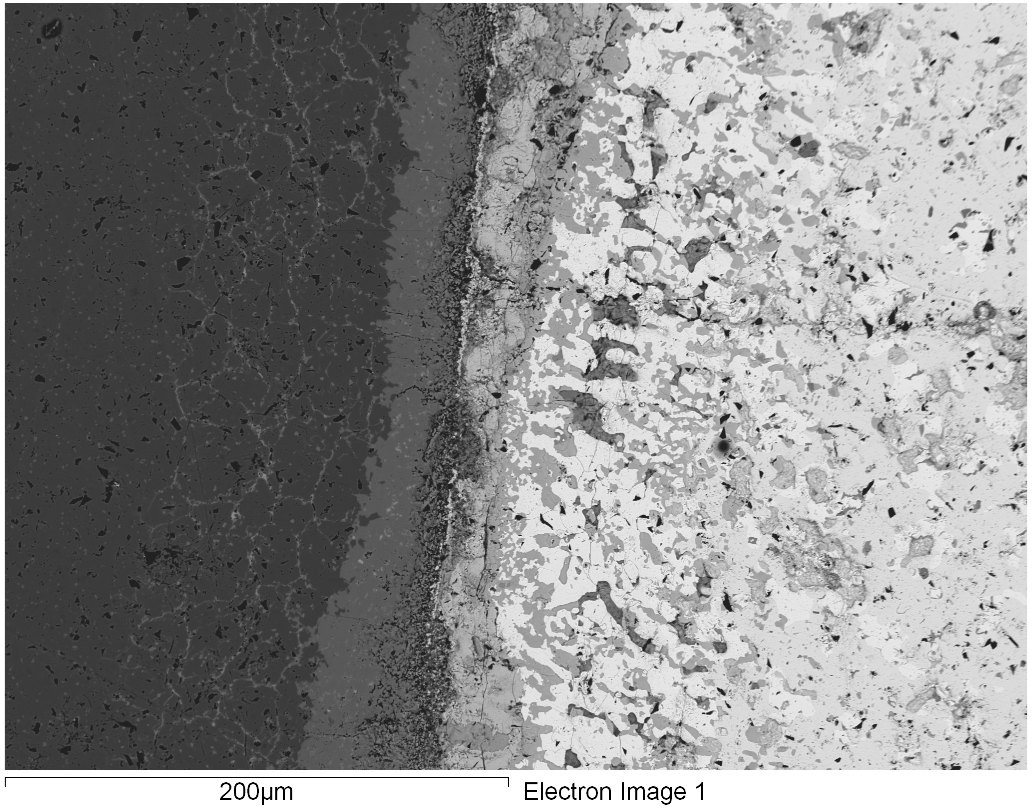
Modelling



D. Salvato et al., Journal of Nuclear Materials (2024), doi:10.1016/j.jnucmat.2024.154928.

Why is PIE Important?

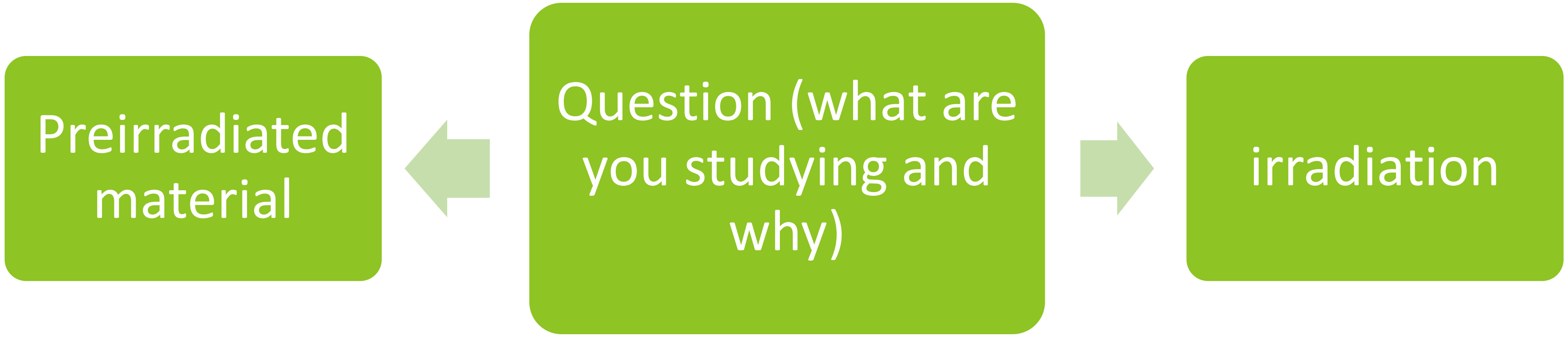
PIE provides feedback to the fuel/materials designers, fabricators, reactor operators and fuel model developers on the performance of fuel and structural components.



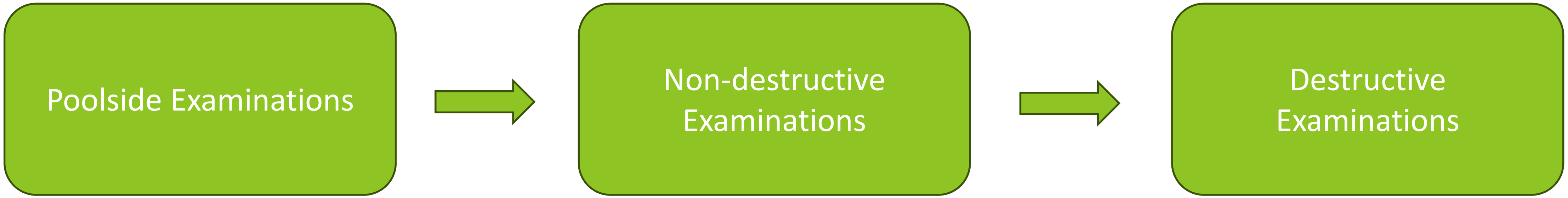
Fuel Cladding Chemical Interaction between HT-9 and U-Zr fuel

Approaches to PIE

- Starts with scientific question (like any research)



Process Flow



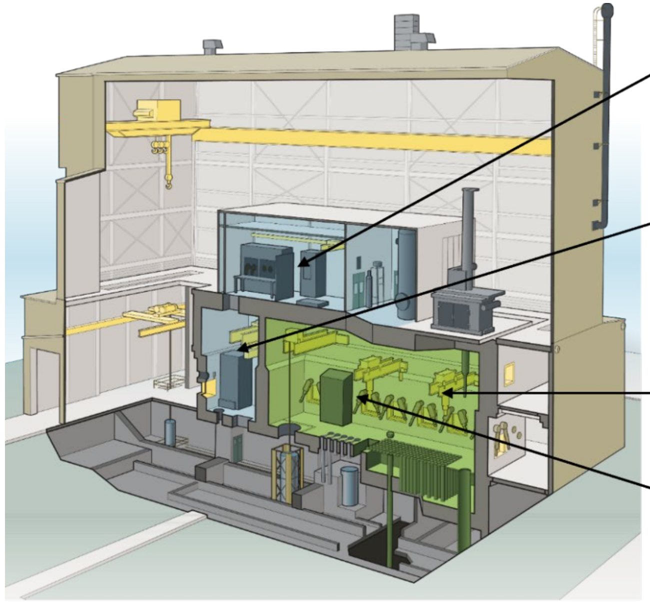
Only if performing an irradiation

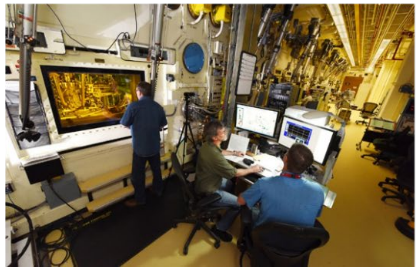
Traditional and Advanced

PIE Facilities

Facilities must be designed to handle radioactive materials (and contamination)

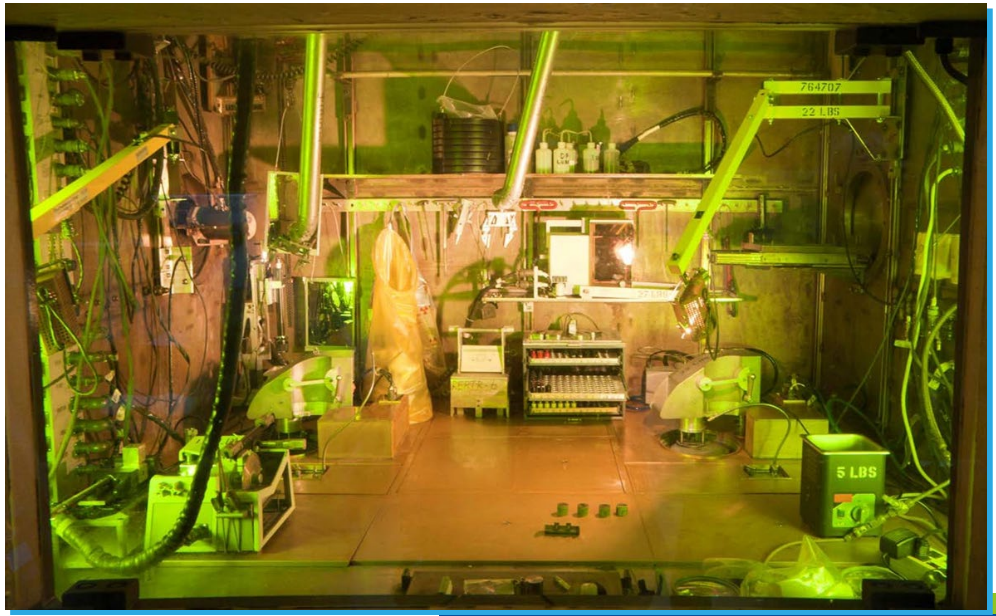
- Remote handled
  - Hot-Cells
  - Shielded enclosures
- Contact handled





Hotcells

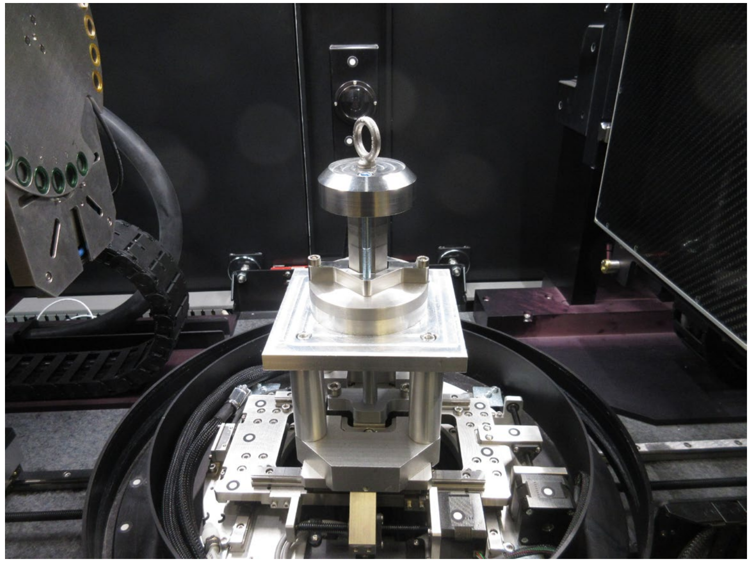
- Thick concrete walls
- Lead glass windows for shielding and visual
- Use manipulators to handle materials (up to 3 meters away from actual samples)
  - Limited feel feedback
  - Dexterity
  - Challenges with view potentially
- Inert vs Air Environments
- Increased training time to become proficient

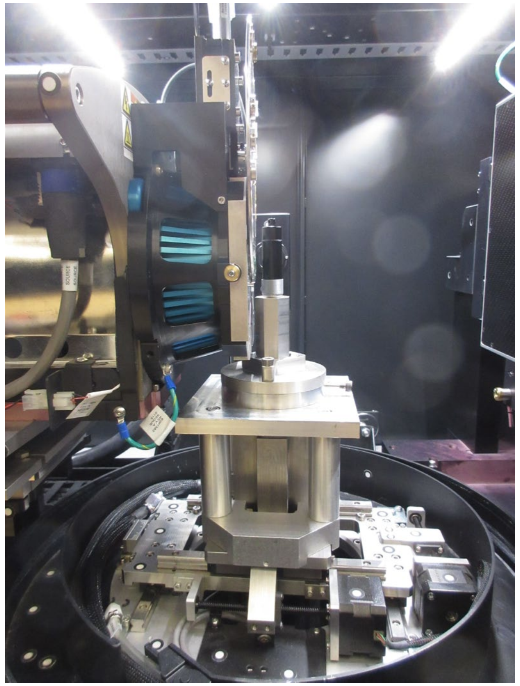


Shielded Enclosures

Gloveboxes/Hoods

- Glovebox
  - Full contamination control with potential for some radiation shielding
  - Limited dose rates for contact handling
  - Limited dexterity and feel
  - Use of shielding items to handle samples
- Hoods
  - Limited contamination and radiation protection
  - Samples of sample dose rates as gloveboxes
  - Better dexterity and feel
  - Increased contamination

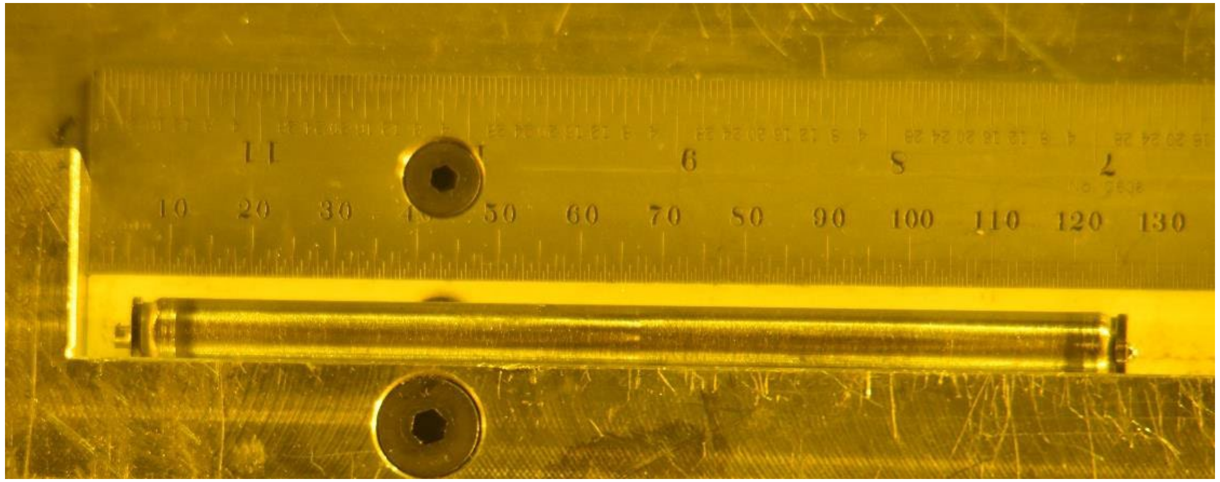




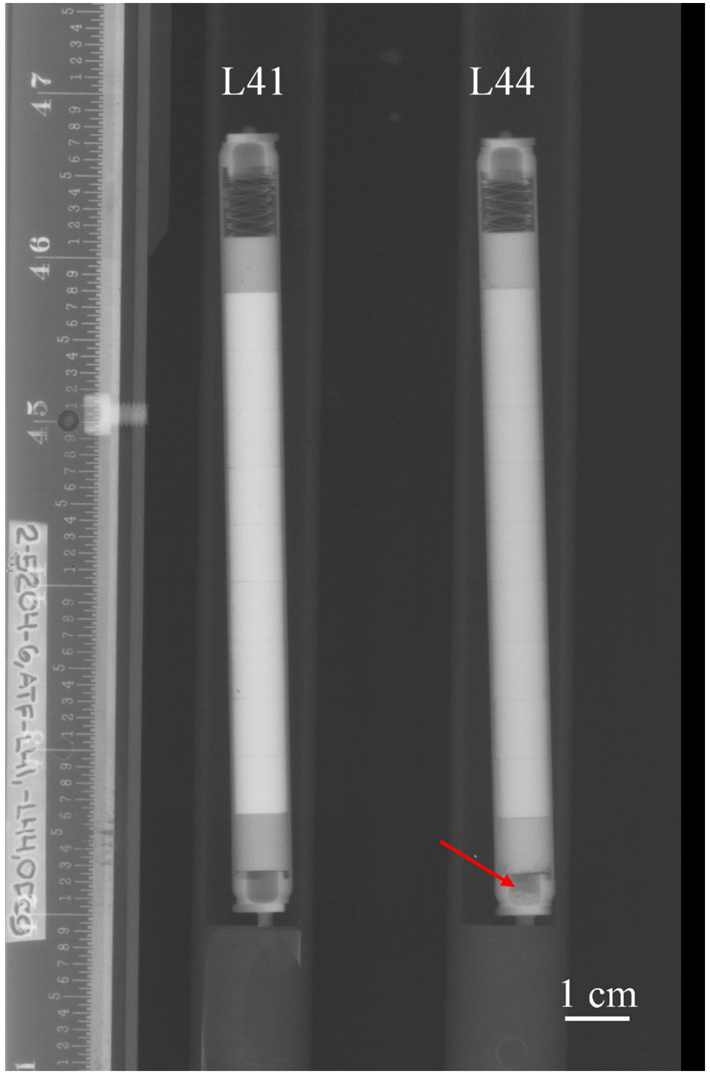
Push pop shielding for X-ray microscope testing

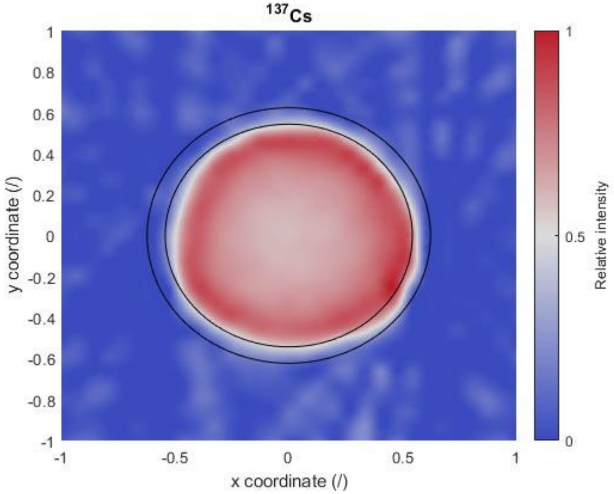
Non-Destructive examination

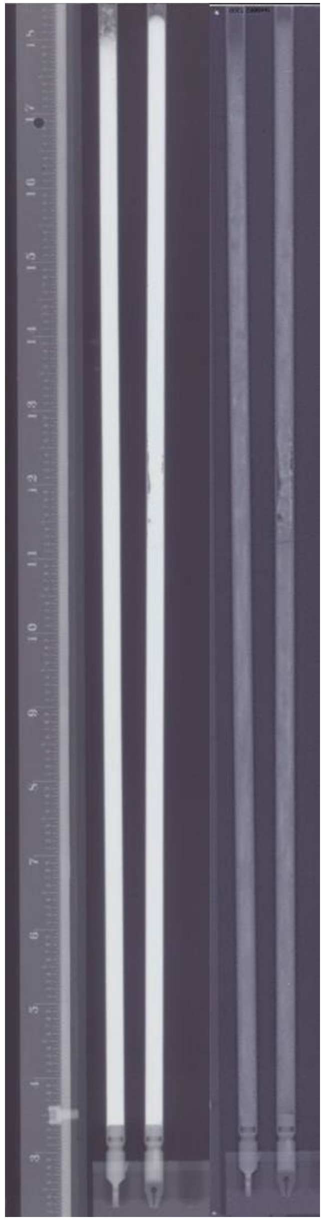
- First step in the process
  - Visual examination
  - Thickness, diameter and oxide thickness measurements
  - Neutron radiography
  - Gamma scanning
  - Density
  - Fission gas analysis
  - X-Ray Micro-CT*



Non-Destructive examination



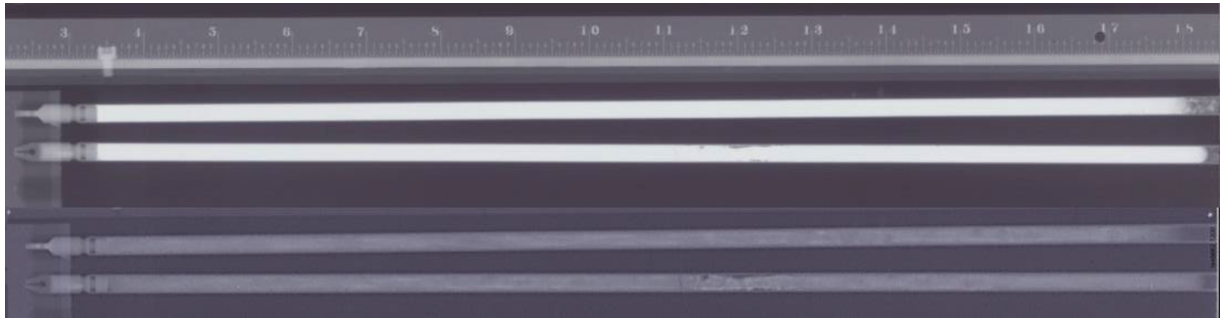




INL EXT-20-59619

Destructive Examinations

- NDE data must be examined before DE can start
- NDE data is used to determine sampling locations

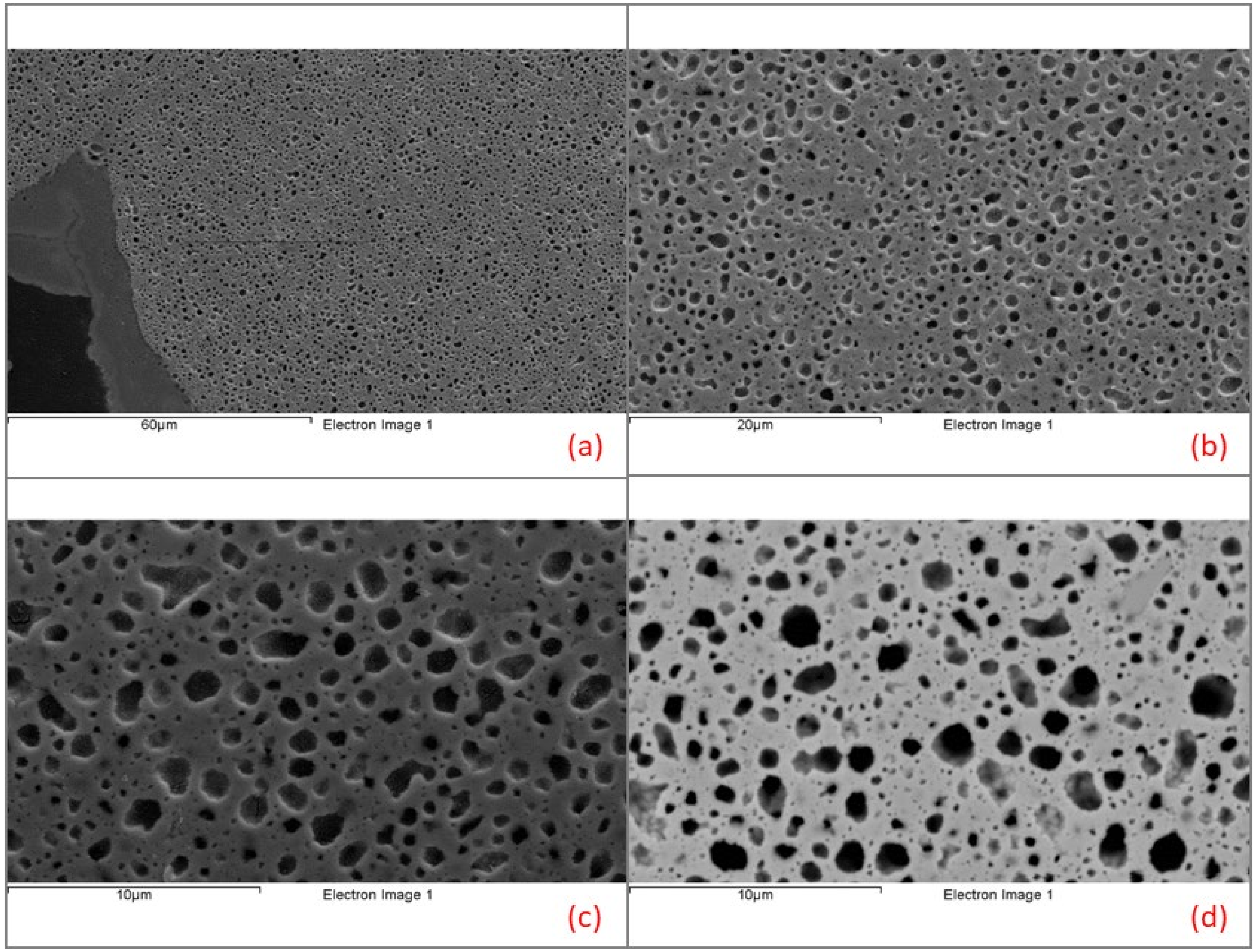


Destructive Examinations

- Sampling and sample preparation
- Optical metallography
- Scanning electron microscopy
- Electron probe microanalysis
- Transmission electron microscopy
- X-ray diffraction
- Radiochemical burnup determination
- Thermophysical properties measurements
- Secondary ion mass spectrometry
- Neutron diffraction
- Atom probe tomography
- X-Ray Micro-CT*

Sample Preparation

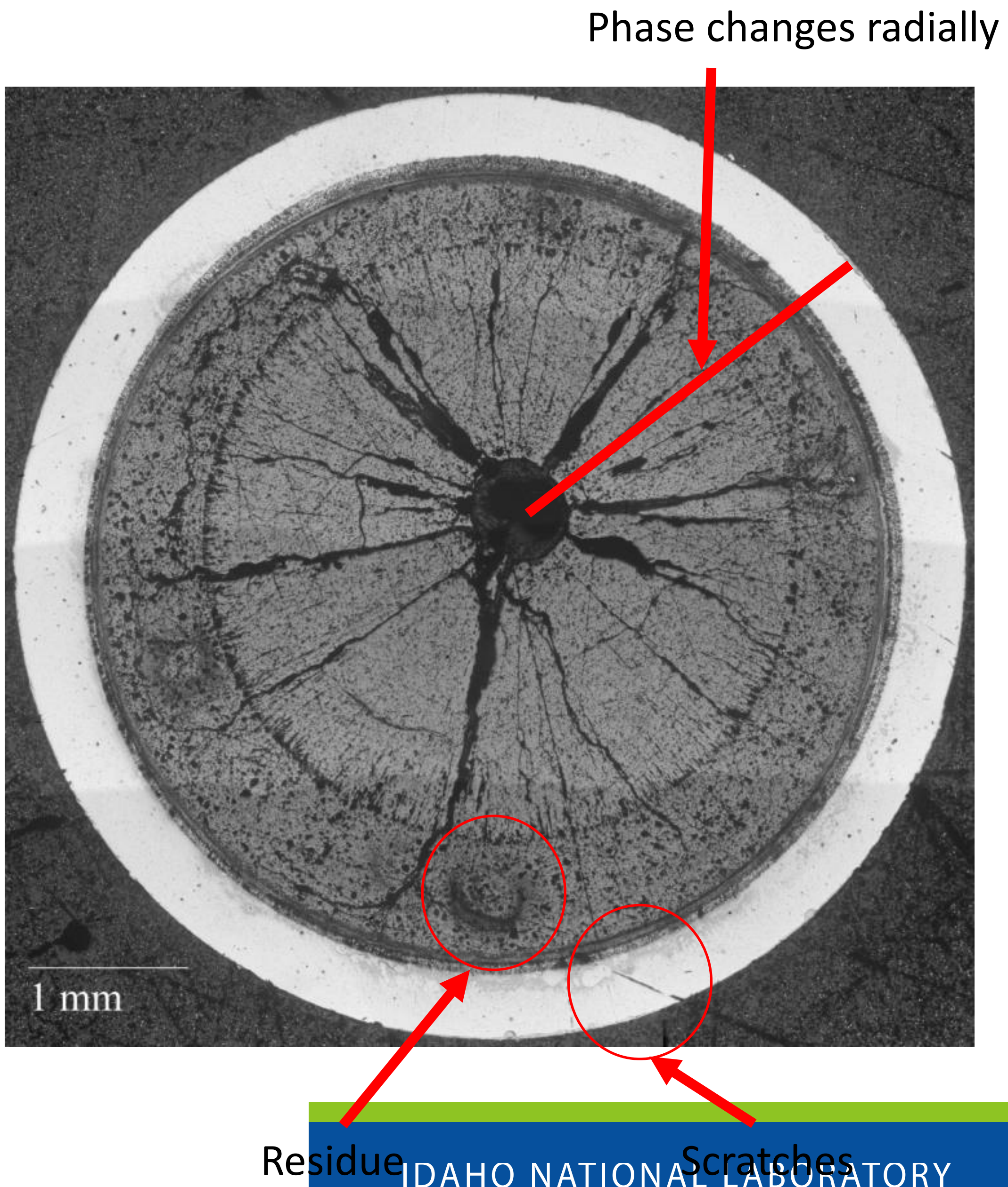
- High quality prep challenging with non-radioactive samples
- Provides accurate and reliable results for advanced characterization results
  - Best practices provide the best results
- Compounded significantly with radiation and contamination fields added in with radiological samples



Porosity of U-Mo fuel

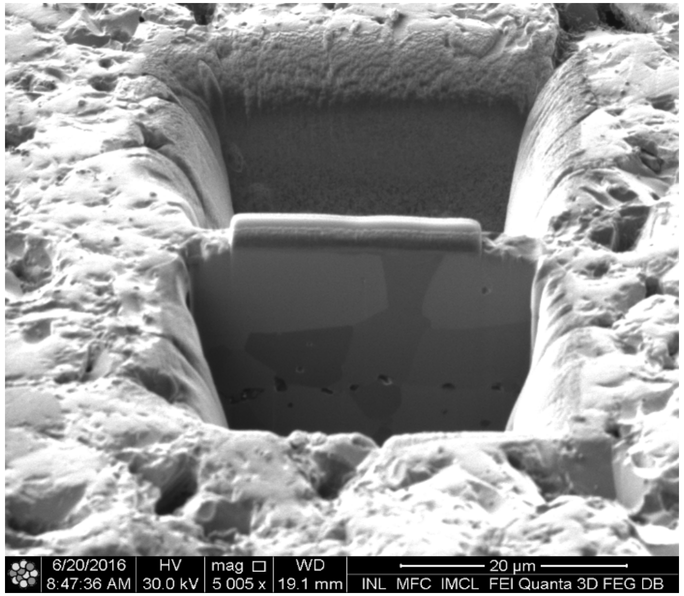
Example: U-Zr Based Fuel Cross-Section

- U-Zr based fuel in a HT-9 cladding
- Multi-phases in fuel
  - Polish different rates (rounding)
  - Holds liquid and could lead to residues
  - Increased polish times of different phases
- Porosity
  - Buildup of polishing debris in pores which lead to scratching later
- Residue staining in areas from cleaning
- Removal of Cs-bearing precipitates due to use of water
- Radiolysis of the epoxy due to radiation fields
- Decontamination of the material leads to increased handling
- Oxidation of the surface if perform in air vs inert
- Many more!

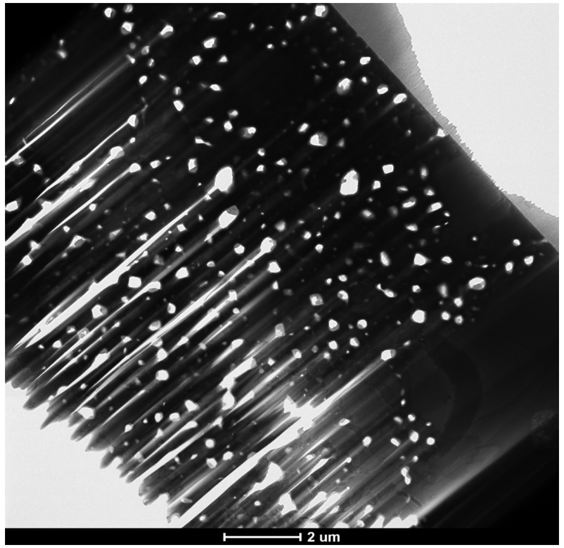


Focused Ion Beam Microscopy

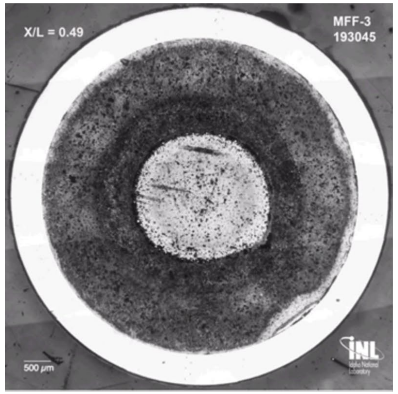
- Site Specific TEM and APT sample prep
- Utilize FIB to remove defects on the surface of the material
  - Mechanical damage from polishing
  - Oxidation
  - Can get electron backscatter diffraction quality samples with some materials
- Drawbacks
  - Curtaining effects
  - Sputtering of radioactive materials into the instrument



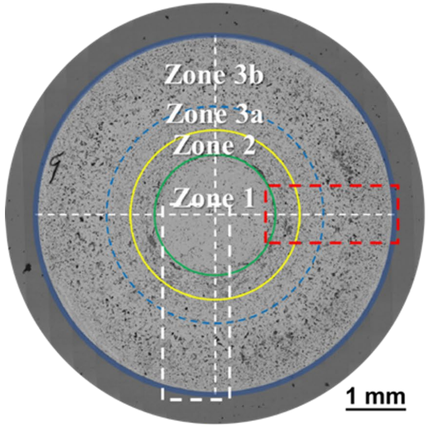
Cross section of $(U,Pu)O_2$ Fuel

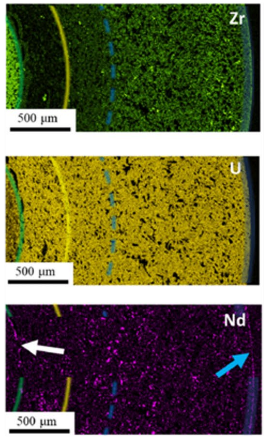


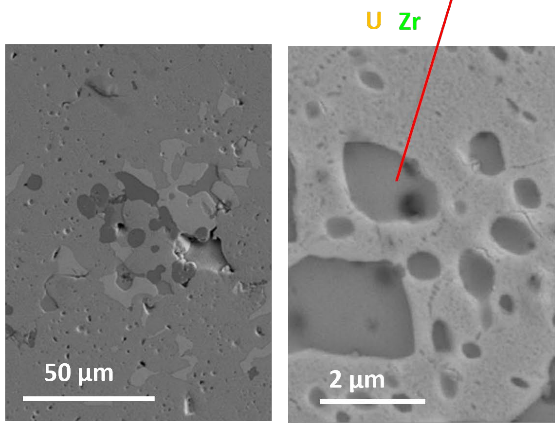
PIE Example Workflow



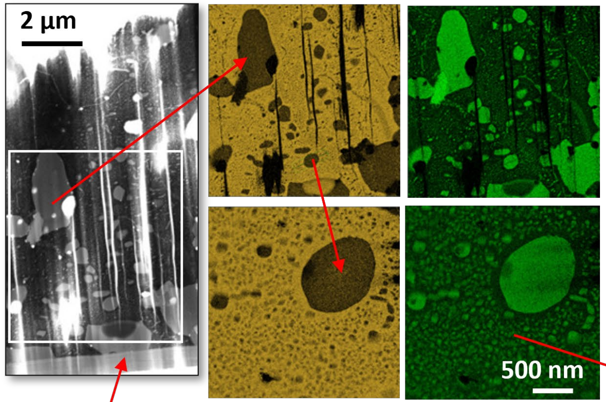
Optical microscopic examination
(U-10Zr fuel)
(1mm, 10^{-3} m)



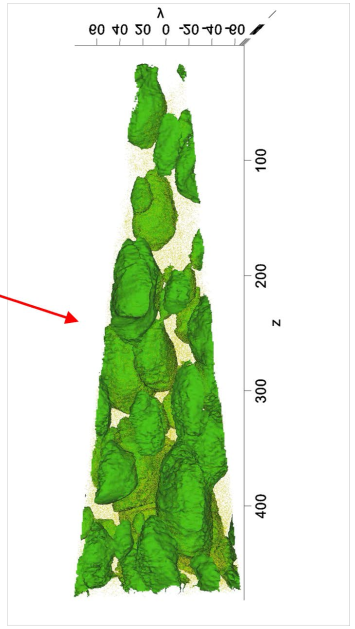




Scanning electron microscopy characterization
of U-10Zr grain and precipitate (1 μm, 10^{-6} m)



Transmission electron
microscopy identification of
Zr nano precipitate
(2-5 nm, 10^{-9} m)

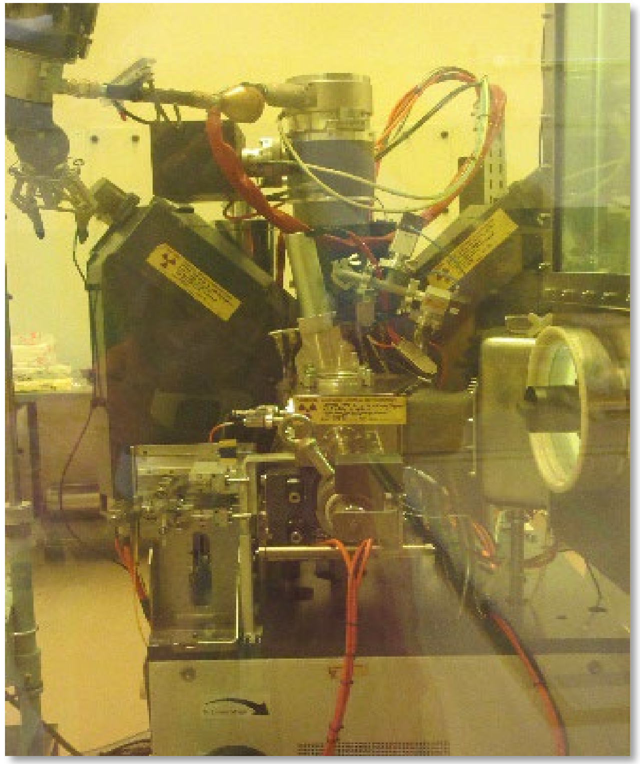


Atom probe tomography study of
Zr atom distribution in 3D
(3 Å, 10^{-10} m)

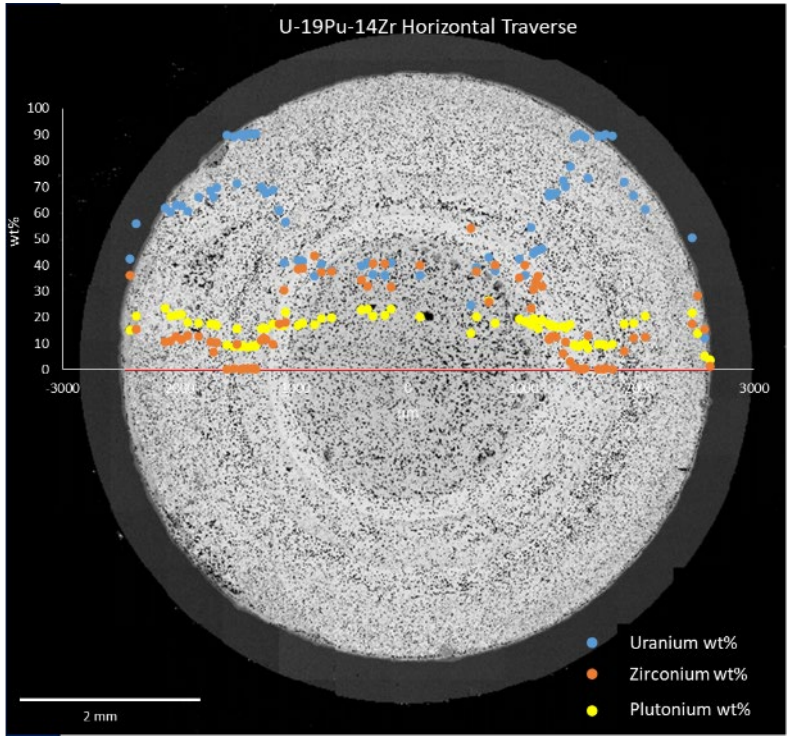
PIE microstructure characterization covers 7 orders of magnitude

Electron Probe Microanalyzer (EPMA)

- Shielded to 3 curies of Cs-137 radiation energy allowing analysis of irradiated fuel pin cross sections
- Capable of quantitative analysis of solid specimens on a micrometer spatial scale
- Can detect elements from B-Cm+ (including gasses trapped in bubbles)

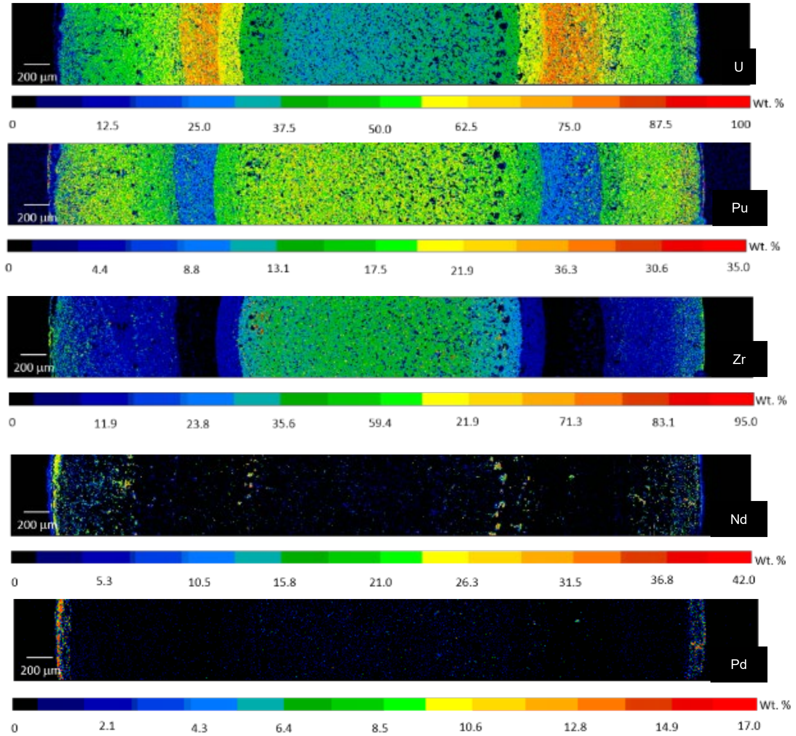


Quantitative analytical diameter traverse of mapped diameter region of U-19Pu-14Zr



Quantitative X-ray maps of irradiated U-19Pu-14Zr

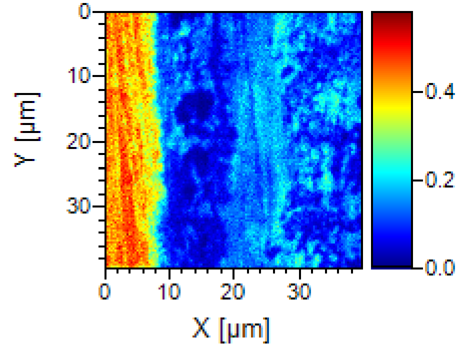
- Enhanced center pin Zr concentration
- Depleted center pin U concentration
- Rare earth element phases near center
- Asymmetrical fuel-cladding chemical interaction



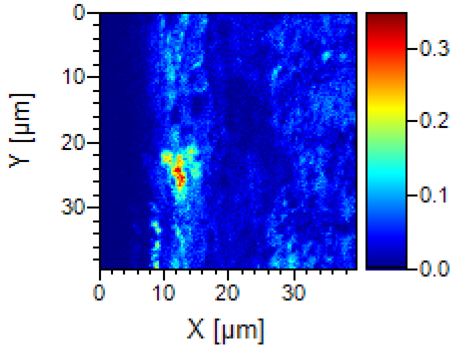
Contact: Karen.Wright@inl.gov

Secondary Ion Mass Spectrometry

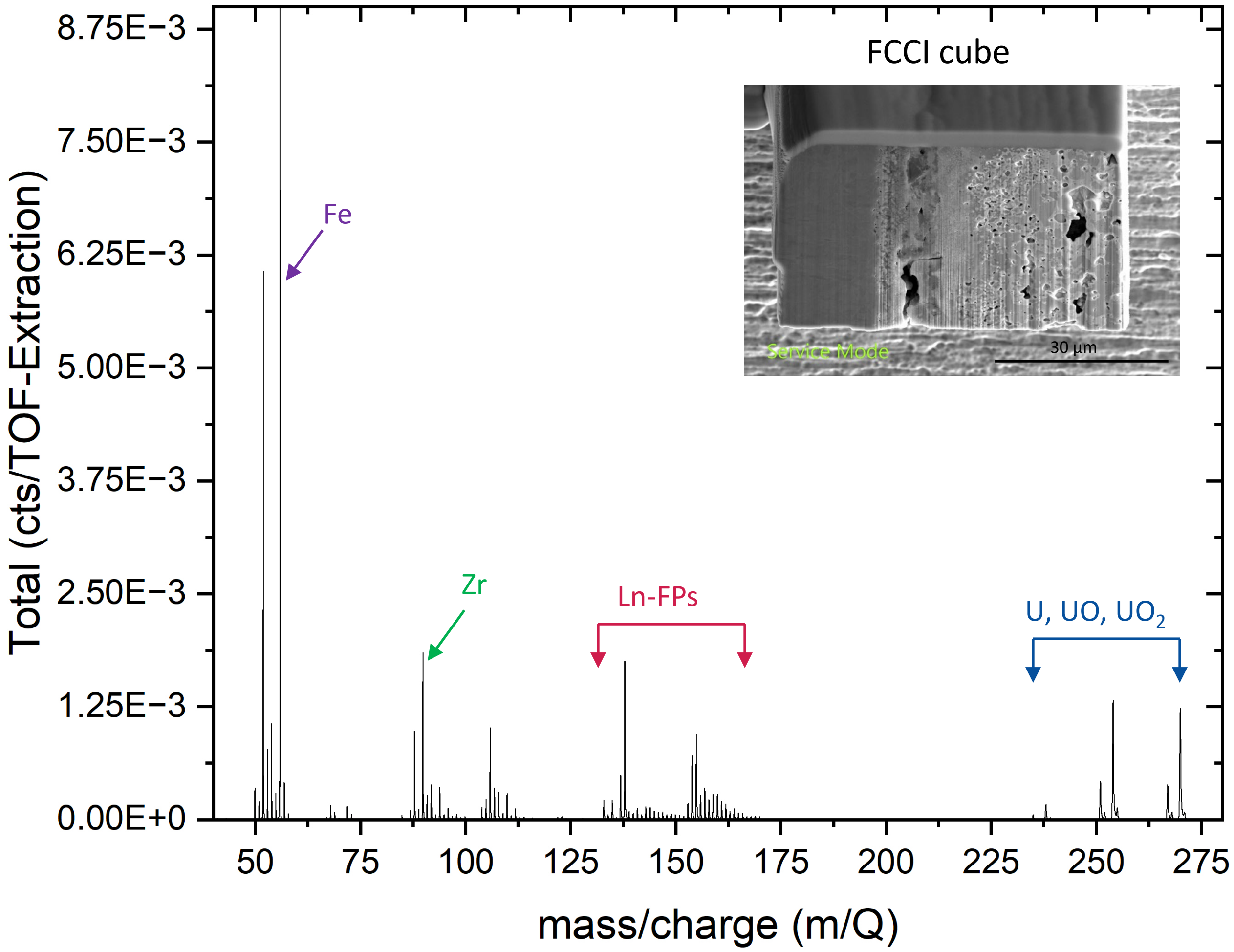
Fe^{56}



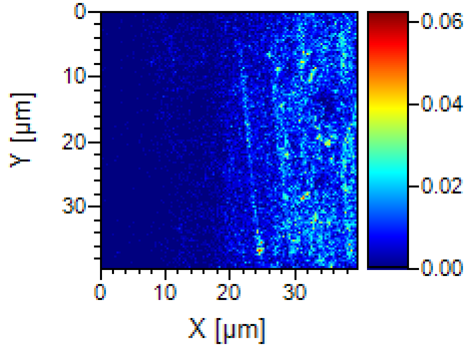
Zr^{90}



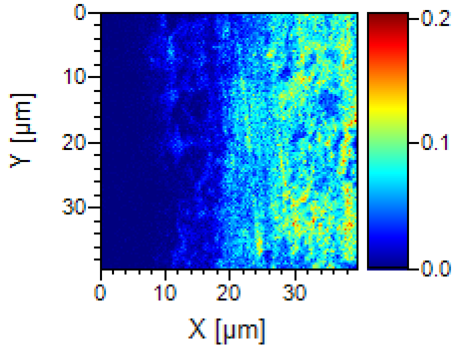
Mass spectrum



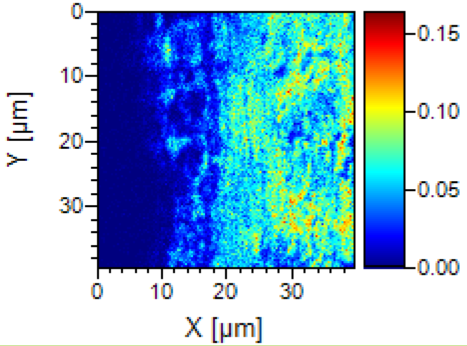
U^{238}



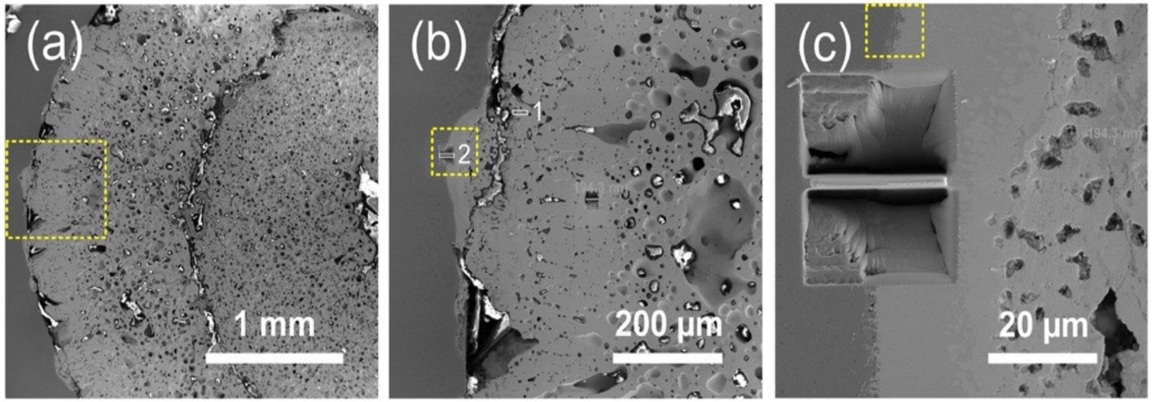
UO

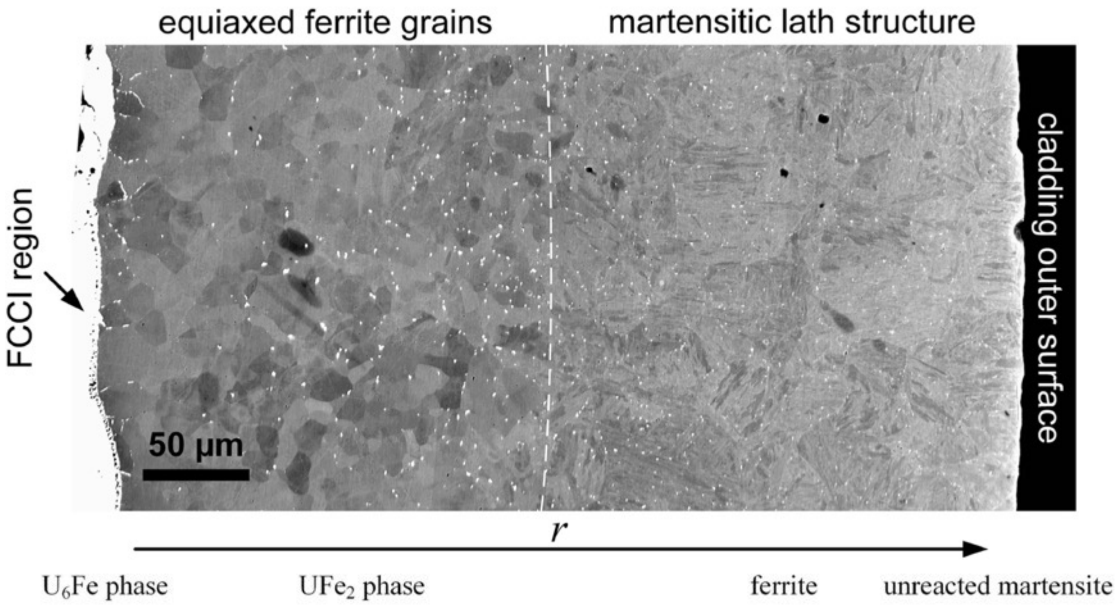


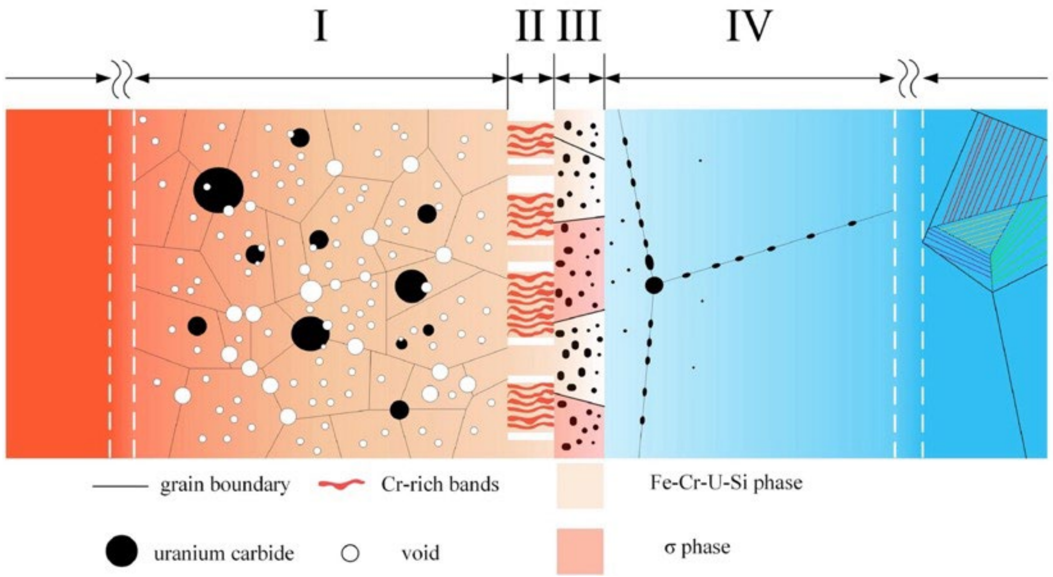
UO_2

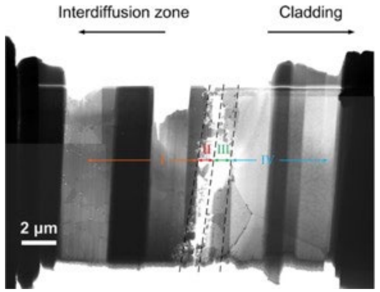


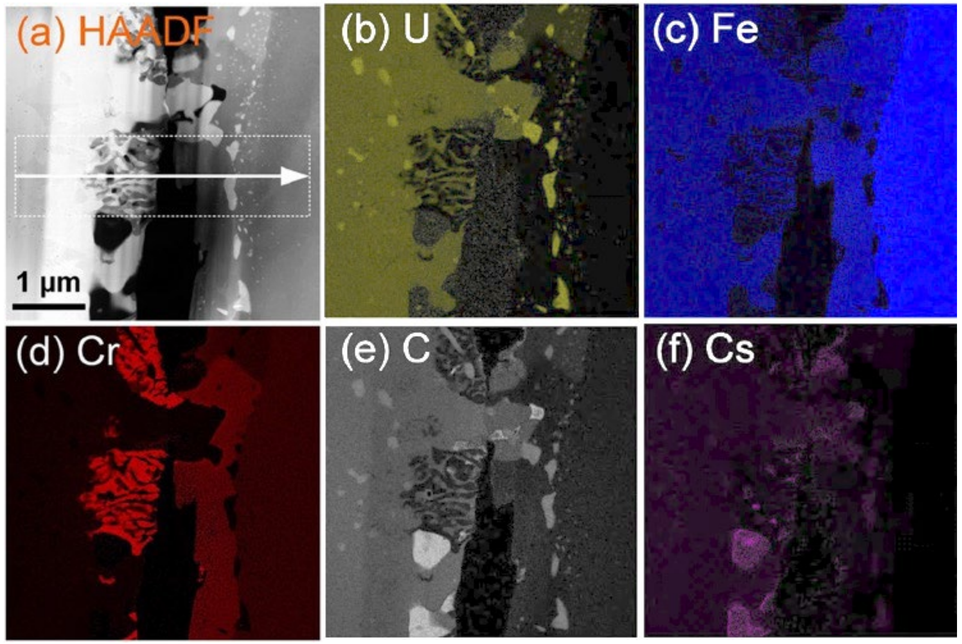
TEM analysis











EELS and EDS

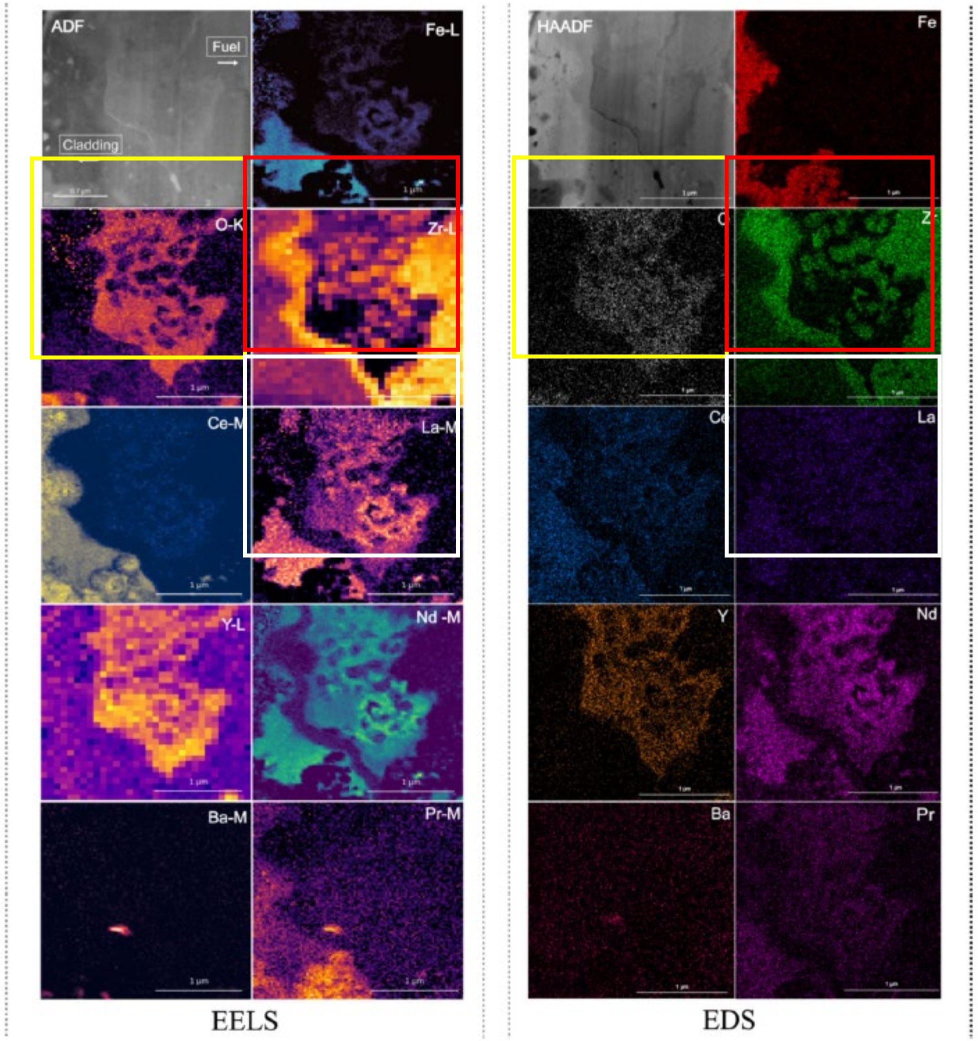
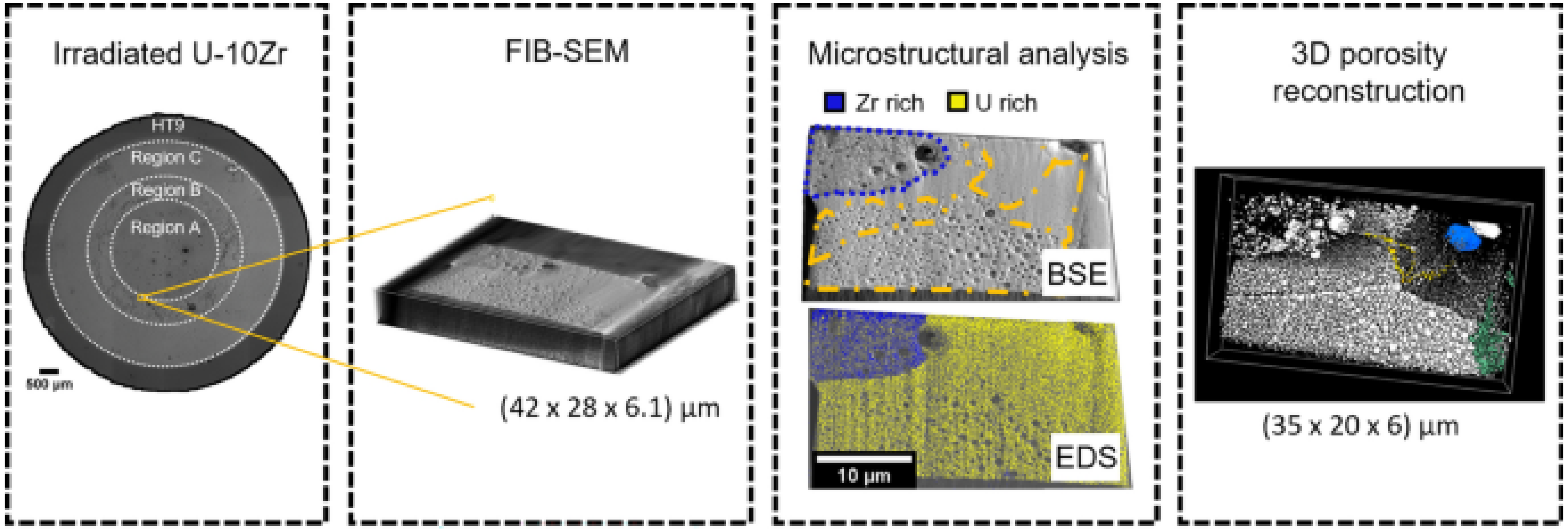
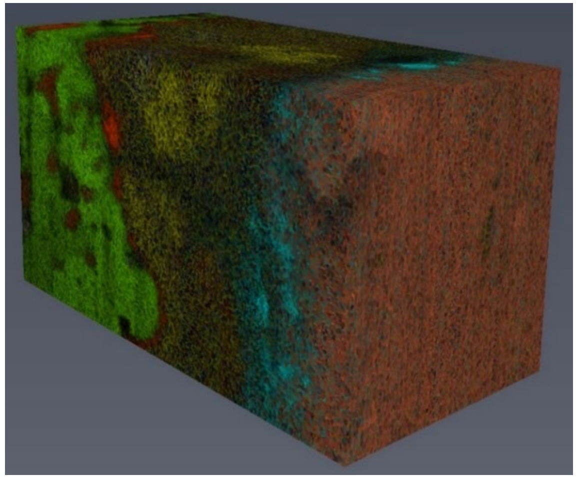


Fig. 5. Elemental maps of a FCCI region from a high burnup fuel (13.2 at%) along with their microstructure image through STEM-EELS (left) and EDS (right) (Pradhan *et. al*).

A. Pradhan, F. Xu, D. Salvato, I. Charit, C. Judge, L. Capriotti, T. Yao, Characterization of Fuel Cladding Chemical Interaction on a High Burnup U-10Zr Metallic Fuel via Electron Energy Loss Spectroscopy Enhanced by Machine Learning, Materials Characterization 218 (2024) 114524.

3D FIB/SEM reconstructions





Thermal diffusivity measurements

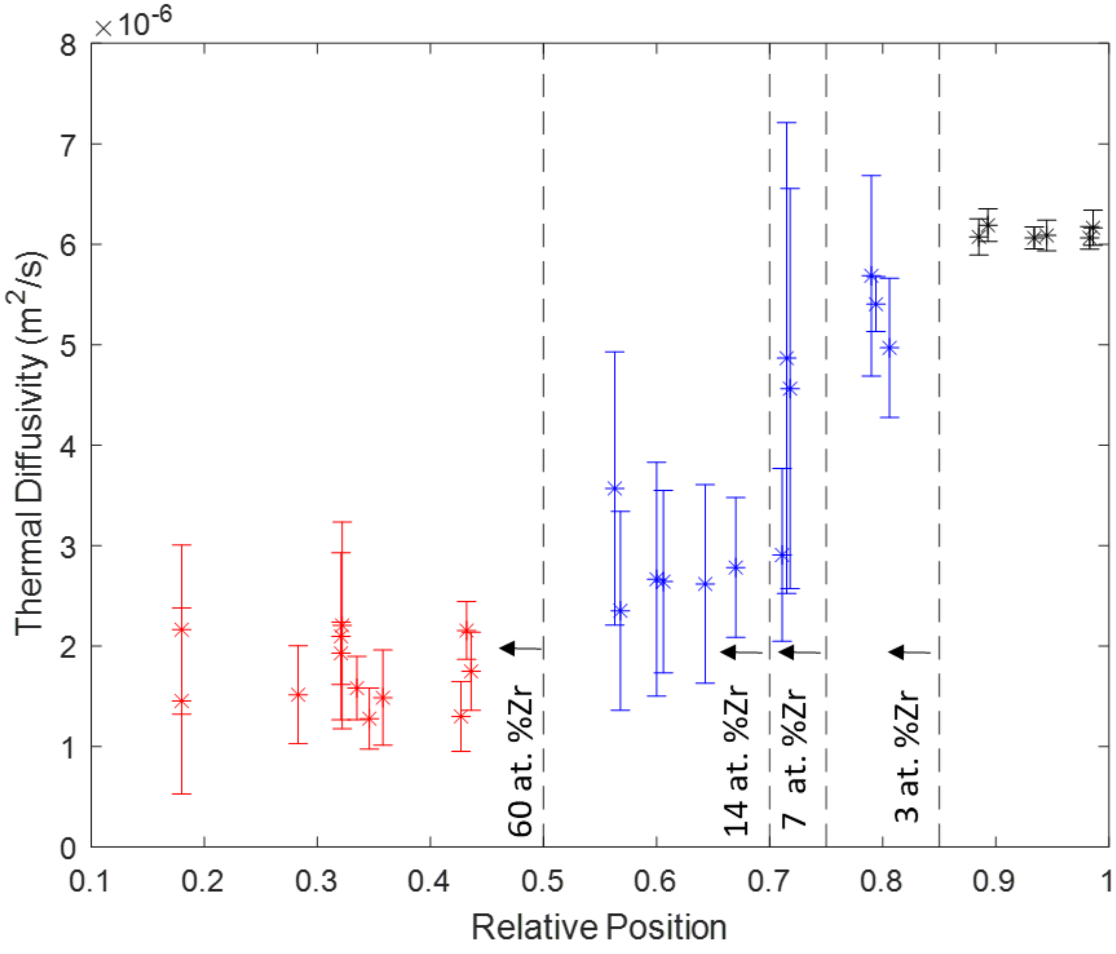
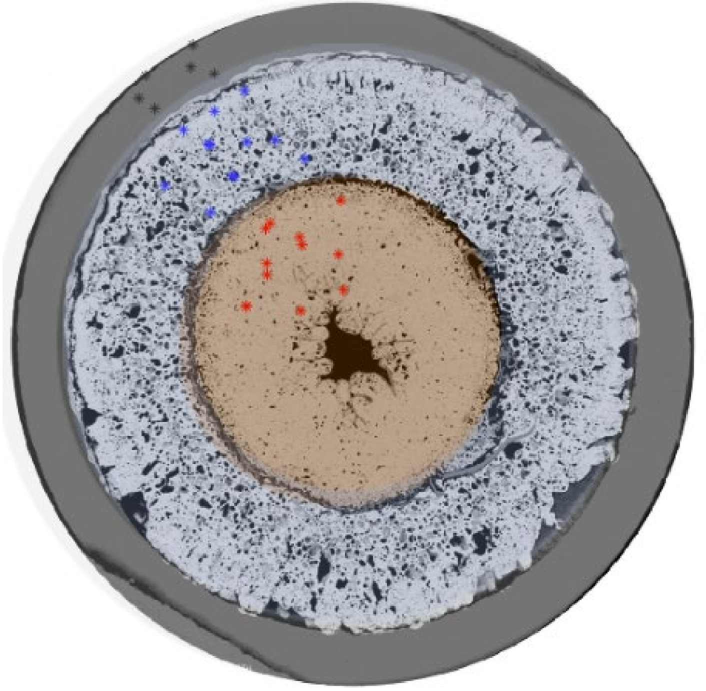


Figure 2 Thermal diffusivity values from TCM measurements of the inner fuel region (red), annular fuel region (blue), and cladding region (black).



Idaho National Laboratory

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