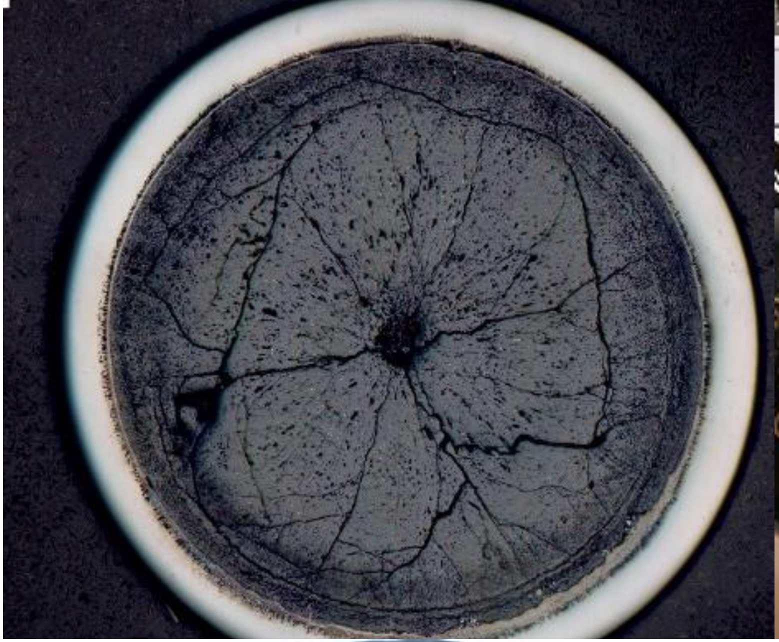
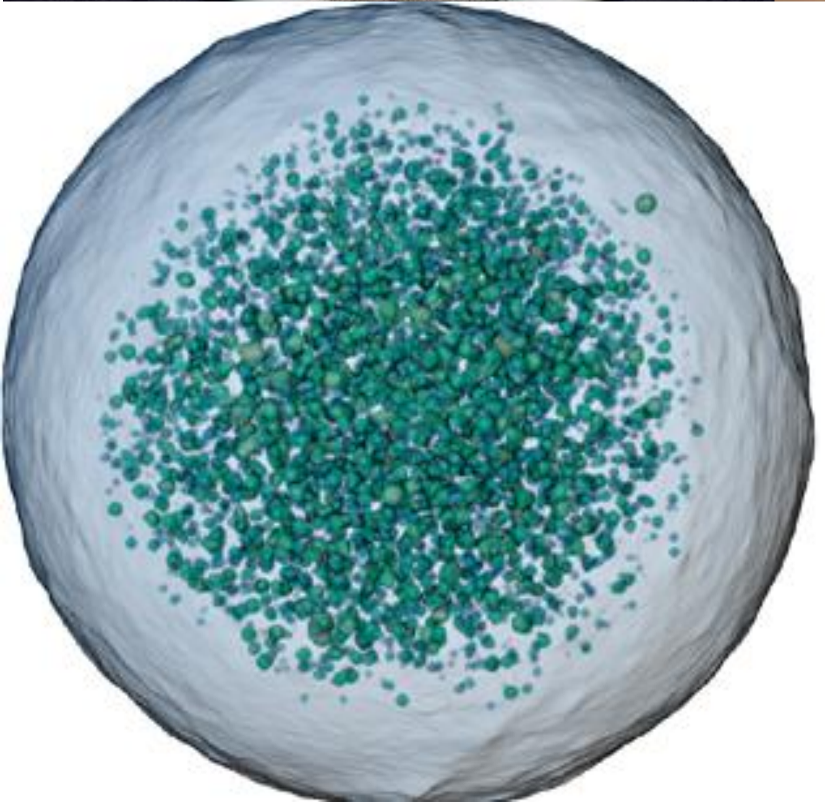


Daniel Murray

Characterization Department Manager



The Irradiated Materials Characterization Lab

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

INL Idaho National Laboratory

INL multiscale approach PIE to support US NE mission

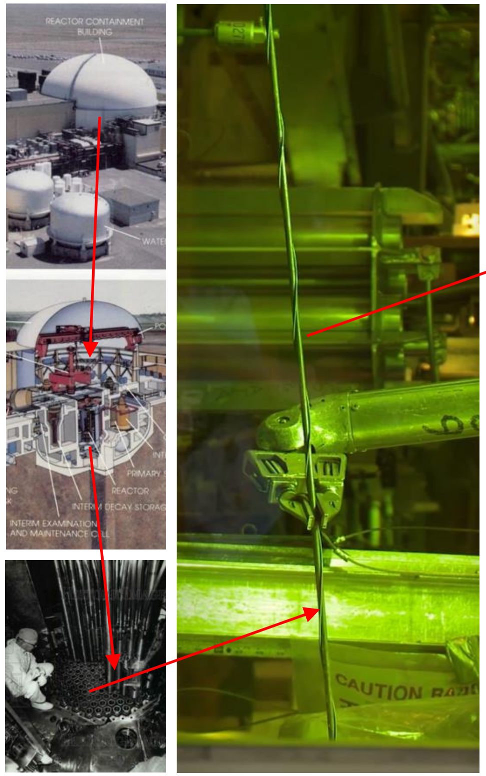
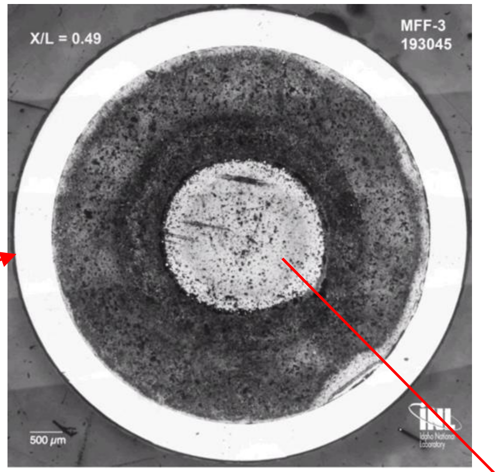
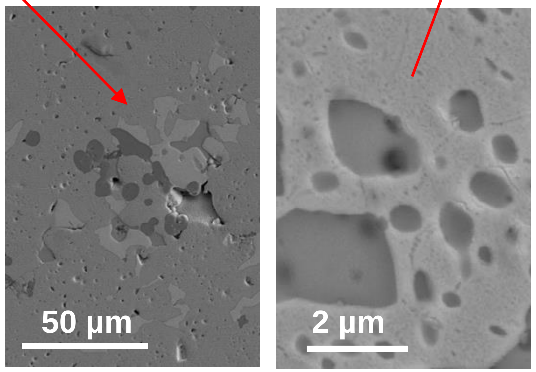


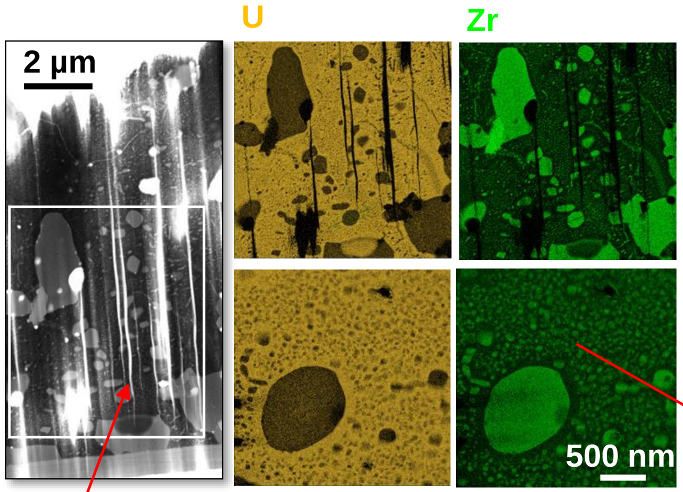
Photo observation of U-10Zr fuel pins irradiated in Fast Flux Test Facility (~1 m)



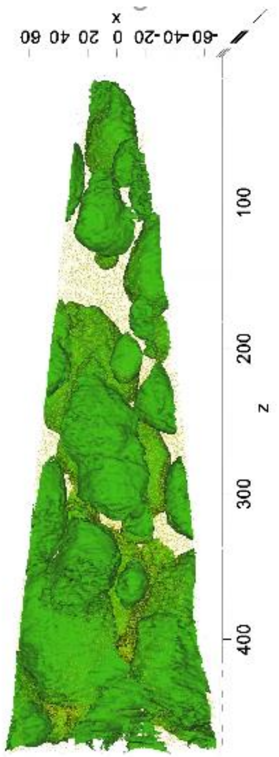
Optical microscopic examination (U-10Zr fuel) (1mm, 10^{-3} m)



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



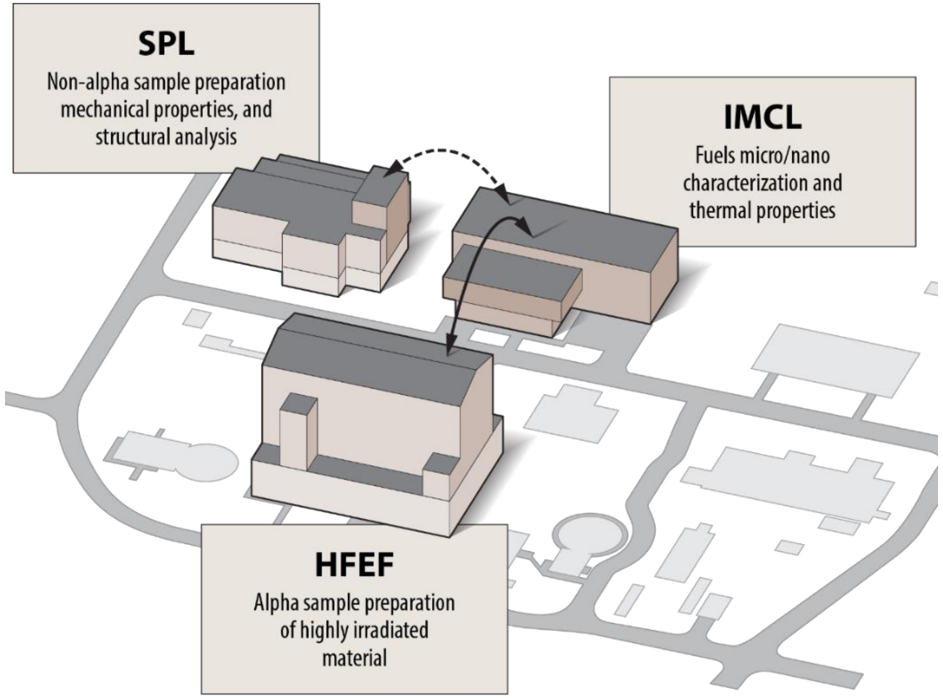
Transmission electron microscopy identification of Zr nano precipitate (2-5 nm, 10^{-9} m)



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

PIE capabilities span 10 orders of magnitude

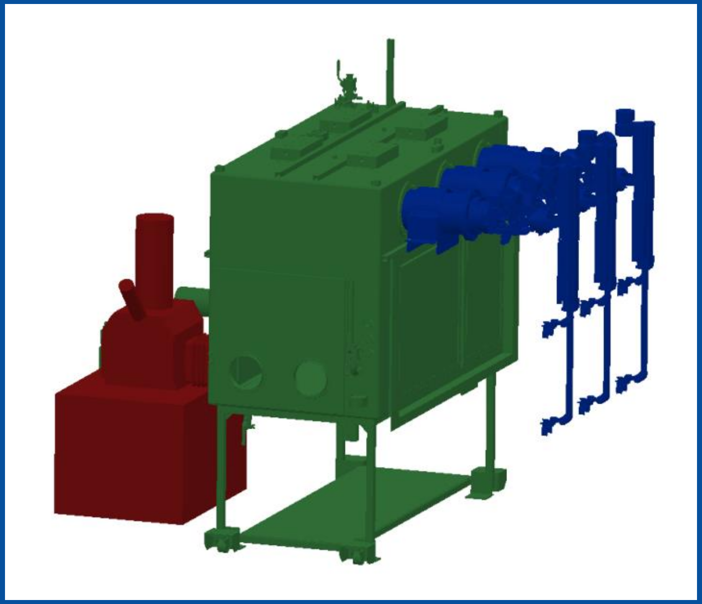
Current PIE capability at INL

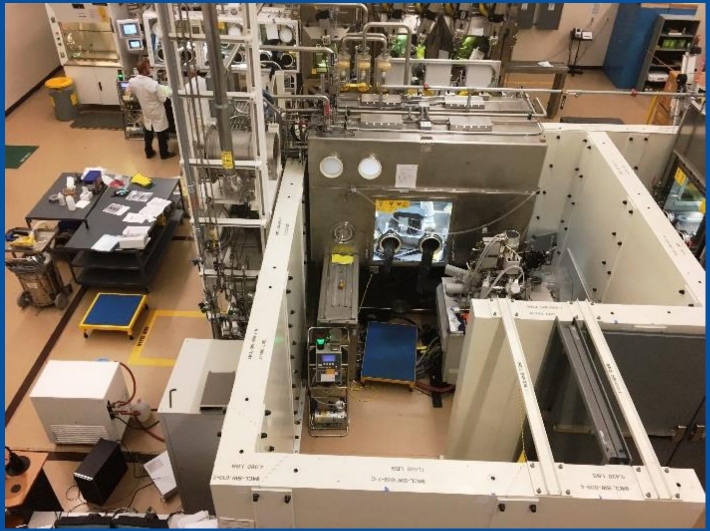


- IMCL provides high end microscopy, thermal testing, and small-scale mechanical testing of irradiated materials including sample preparation of commercial fuel pellet sized experiments
- IMCL, SPL, and HFEF are broadly available to the nuclear research community through the Nuclear Science User Facilities, University partnerships, DOE programs, and Strategic Partnership programs

Design Features of IMCL

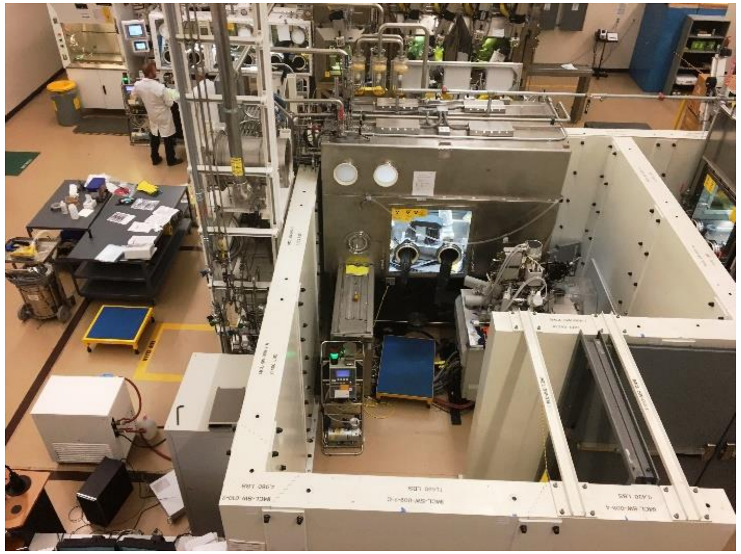
- Haz Cat 2 Nuclear facility
- “Accessible” Shielded instrumentation
  - Steel walls provide the shielding
  - Glove box controls contamination
  - Inert atmosphere in glovebox prevents sample oxidation
- Remote or contact equipment loading/unloading and operation



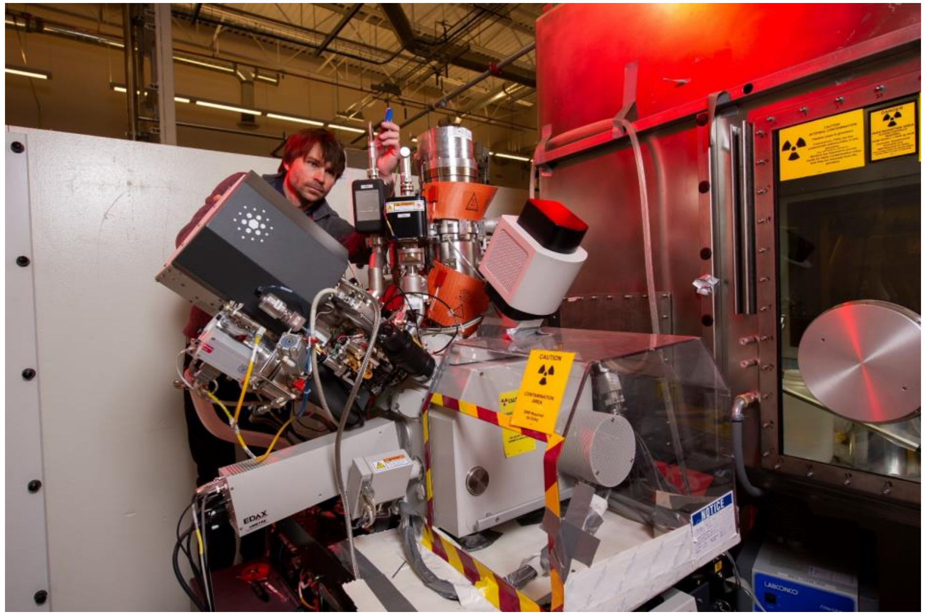


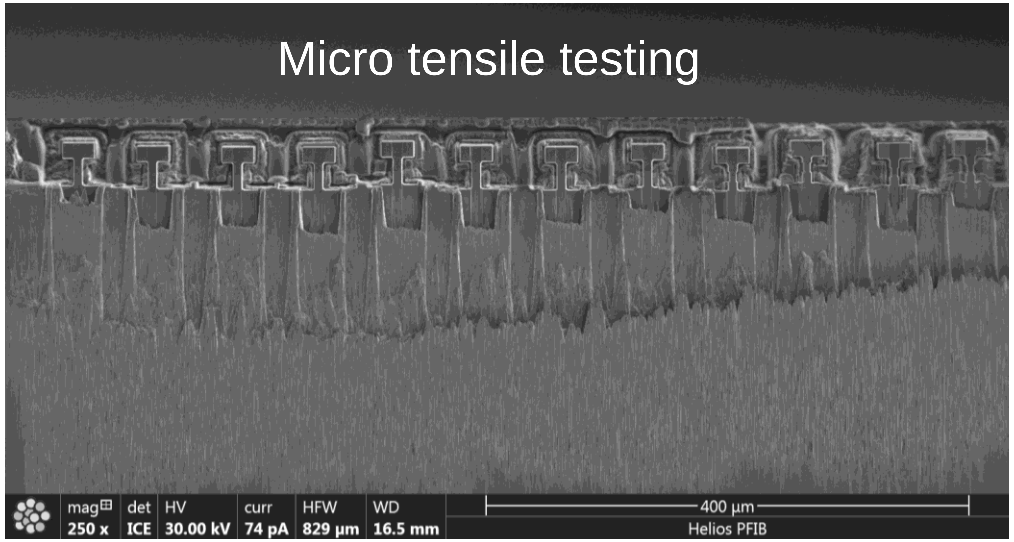
IMCL Overview

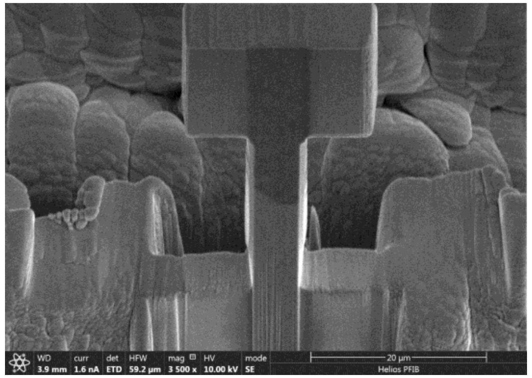
<u>No similar shielded instrument capability exists in the United States</u>



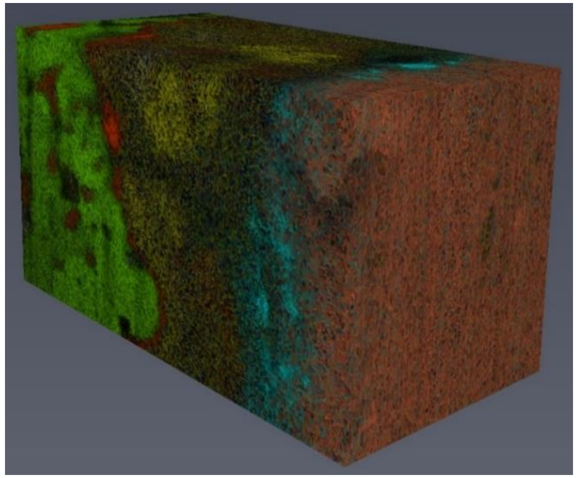
Shielded Dual Beam FIB’s



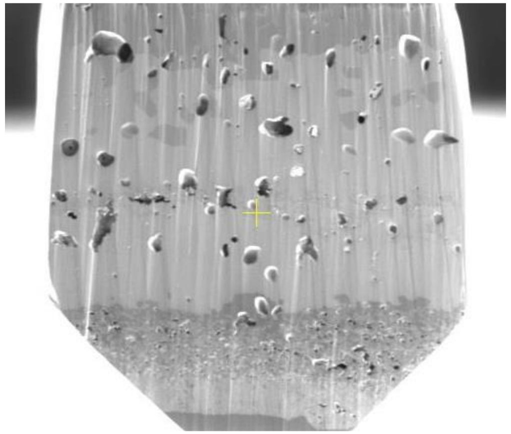


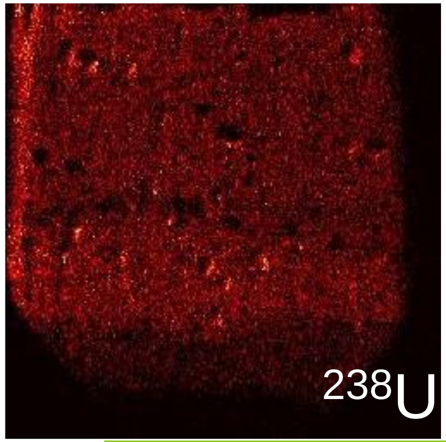


Irradiated U10Zr EDS FIB tomography



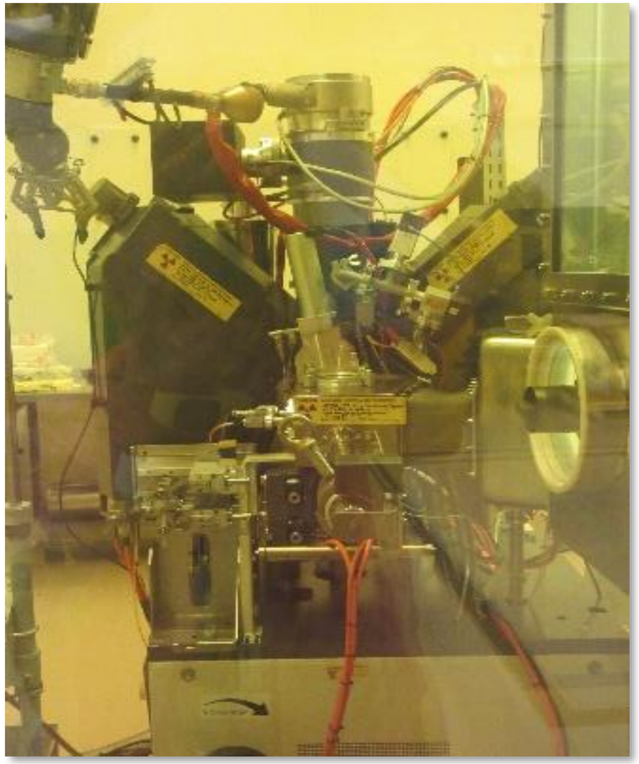
FIB-TOF-SIMS





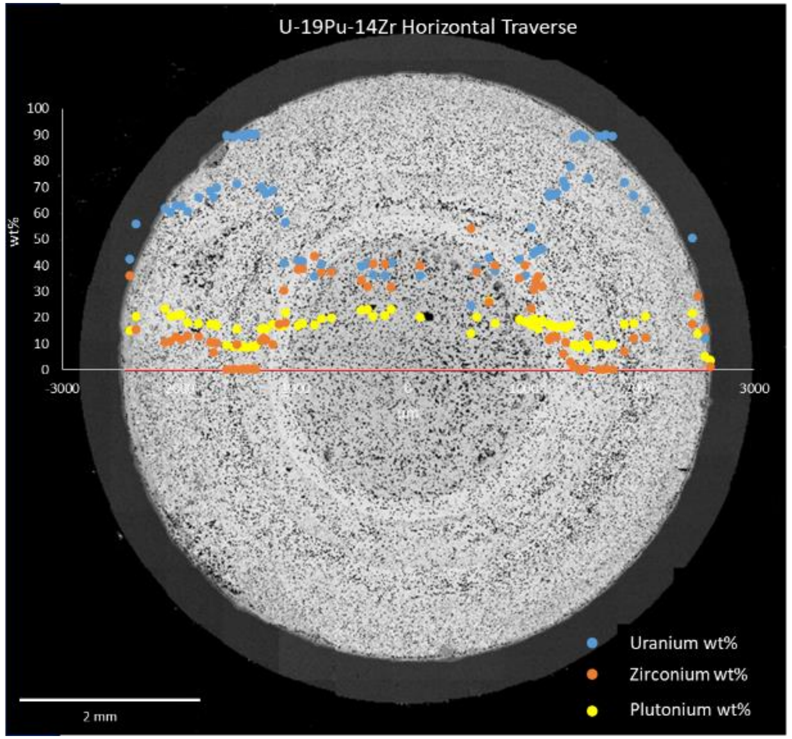
Electron Probe Microanalyzer (EPMA)

Cameca SX100R EPMA viewed through shielded window



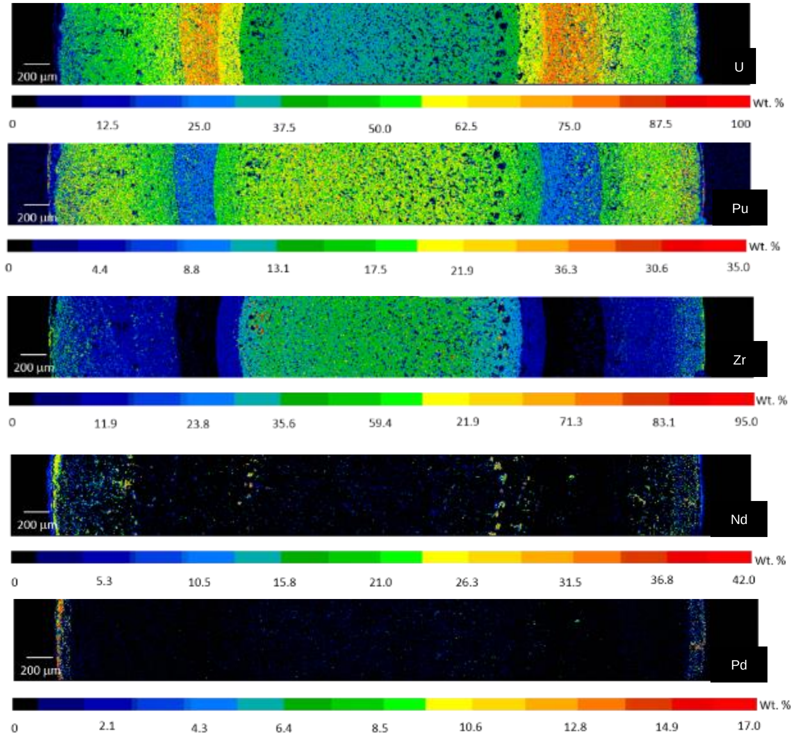
- Shielded to 3 curies of Cs-137 radiation energy allowing analysis of irradiated fuel pin cross sections
- Capable of quantitative analysis of solid specimens on a micrometer spatial scale
- Can detect elements from B-Cm+ (including gasses trapped in bubbles)

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



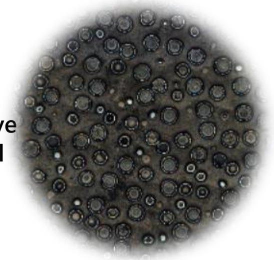
Contact: Karen.Wright@inl.gov

3D X-ray Imaging of Irradiated TRISO Fuel Compacts & Particles

Nikolaus L. Cordes, Brian J. Gross, William C. Chuirazzi, Joshua J. Kane, & John D. Stempien

Introduction

To qualify TRISO fuel, neutron irradiation tests have been performed on compact fuel elements using INL's Advanced Test Reactor



Traditional post-irradiation examinations rely on destructive sample preparations for optical or electron microscopy

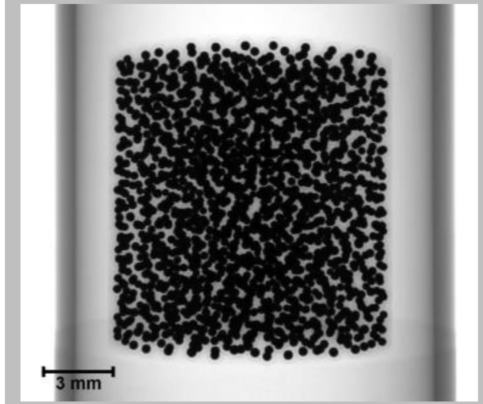
A commercial X-ray microscope has been adapted to image fuel in 3D *nondestructively*

Materials

TRISO compacts from AGR-3/4 (burnup ~5% - 15% FIMA) and TRISO particles from deconsolidated AGR-2 compacts (burnup ~8.8% FIMA) have been imaged

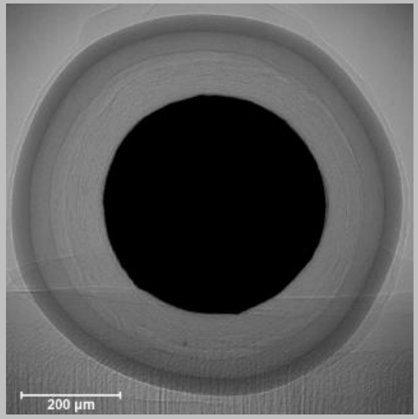
Methods

A stainless steel container was designed to shield personnel from gamma radiation and protect the microscope's components. It seamlessly interfaces with the X-ray microscope



TRISO Compact

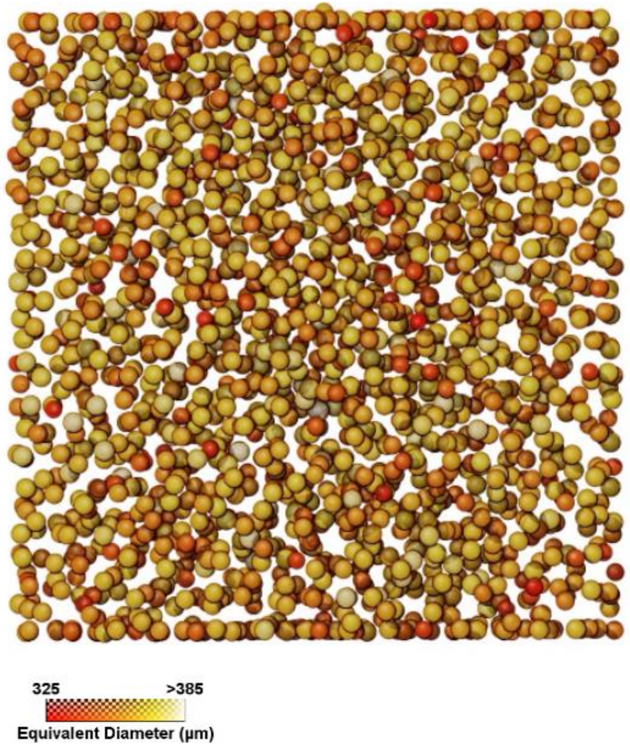
5,000 radiographs were collected over 360° sample rotation in ~28 h. Each pixel is 11 µm



TRISO Particle

1,600 radiographs were collected over 360° sample rotation in ~8 h. Each pixel is 1 µm

Results



This 3D rendering shows the size and location of each of the 1,918 UCO fuel kernels in the TRISO compact

This work was sponsored by the U.S. Department of Energy, Office of Nuclear Energy, through the Advanced Reactor Technologies Advanced Gas Reactor Fuel Development and Qualification Program.

IDAHO NATIONAL LABORATORY

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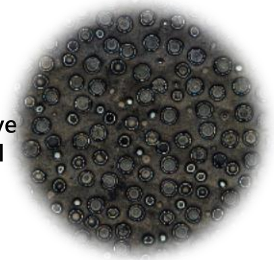

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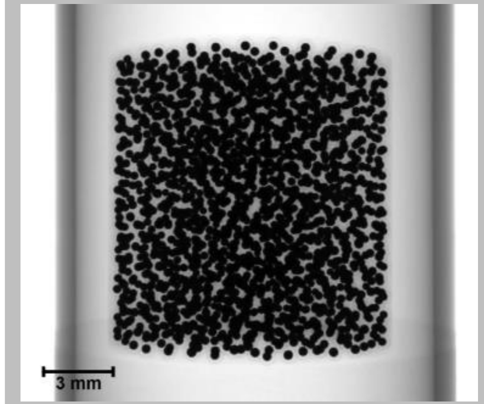

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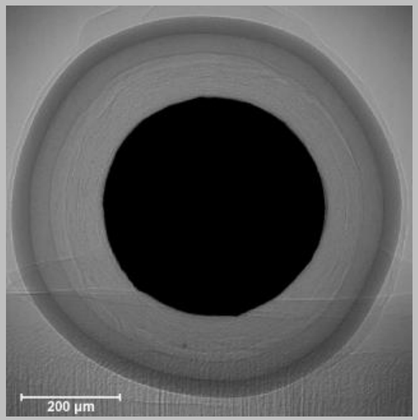

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Acknowledgements

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INL Radiological Controls
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Ben Tolman
Chad Coles
Quintin Harris
Chester Perry
Joe Price
Ed Wiscaver

INL's Irradiated Materials Characterization Laboratory
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Jim Madden
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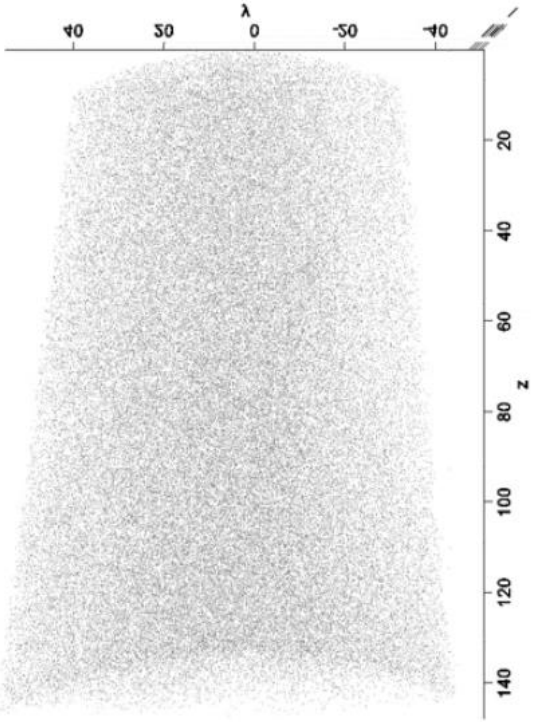
Jeff Bailey
Jayson Bush
Miles Cook
Keth Galloway
Britton Schow
Nickolas Owen
Courtney King

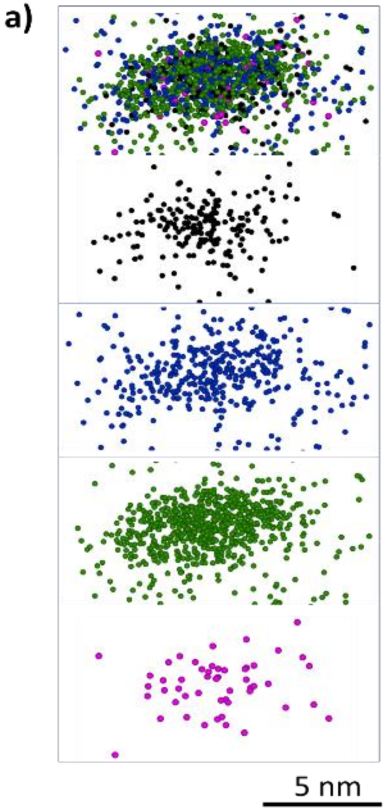
This work was sponsored by the U.S. Department of Energy, Offic
Nuclear Energy, through the Advanced Reactor Technologies
Advanced Gas Reactor Fuel Development and Qualification Progr

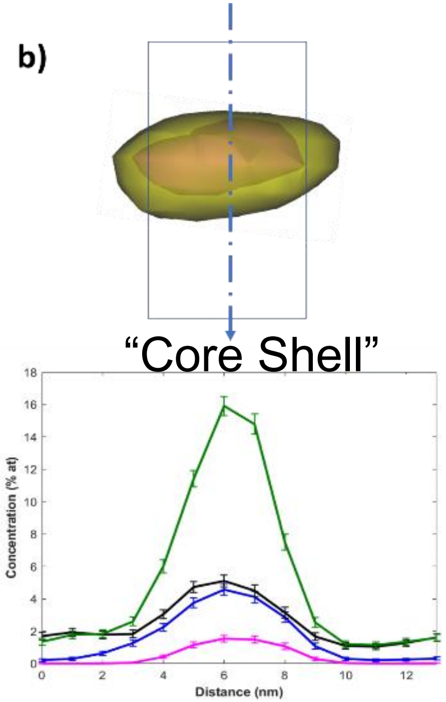
Understanding precipitation mechanism in neutron irradiated RPV steel

Atom Probe Tomography (APT)

●Mn●Ni●Si●P

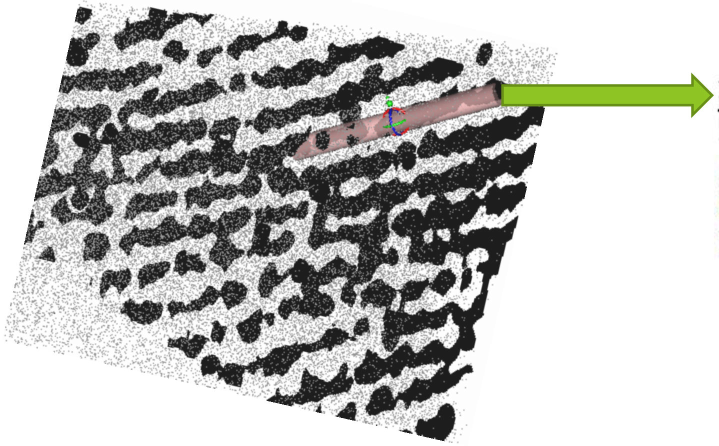


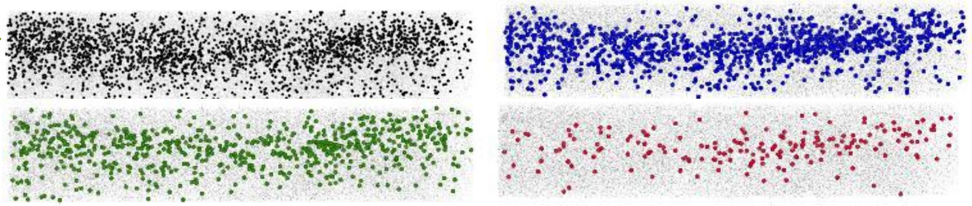




“Core Shell”

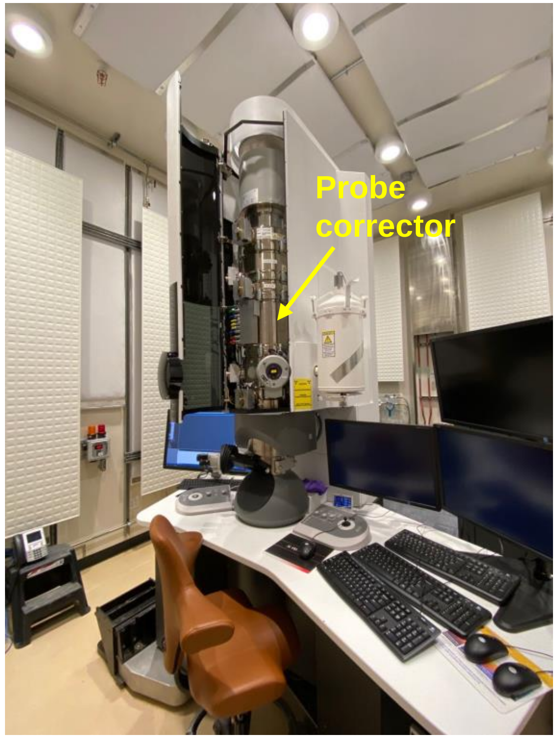
Network of dislocations at grain boundary

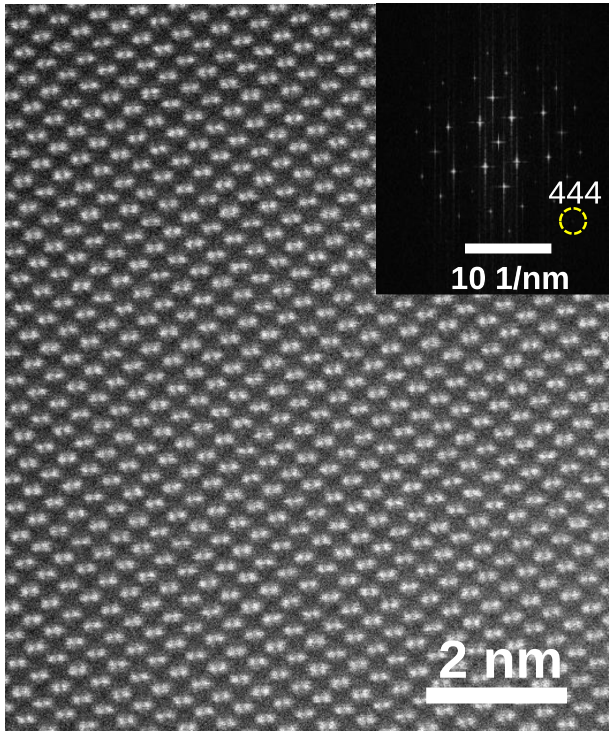




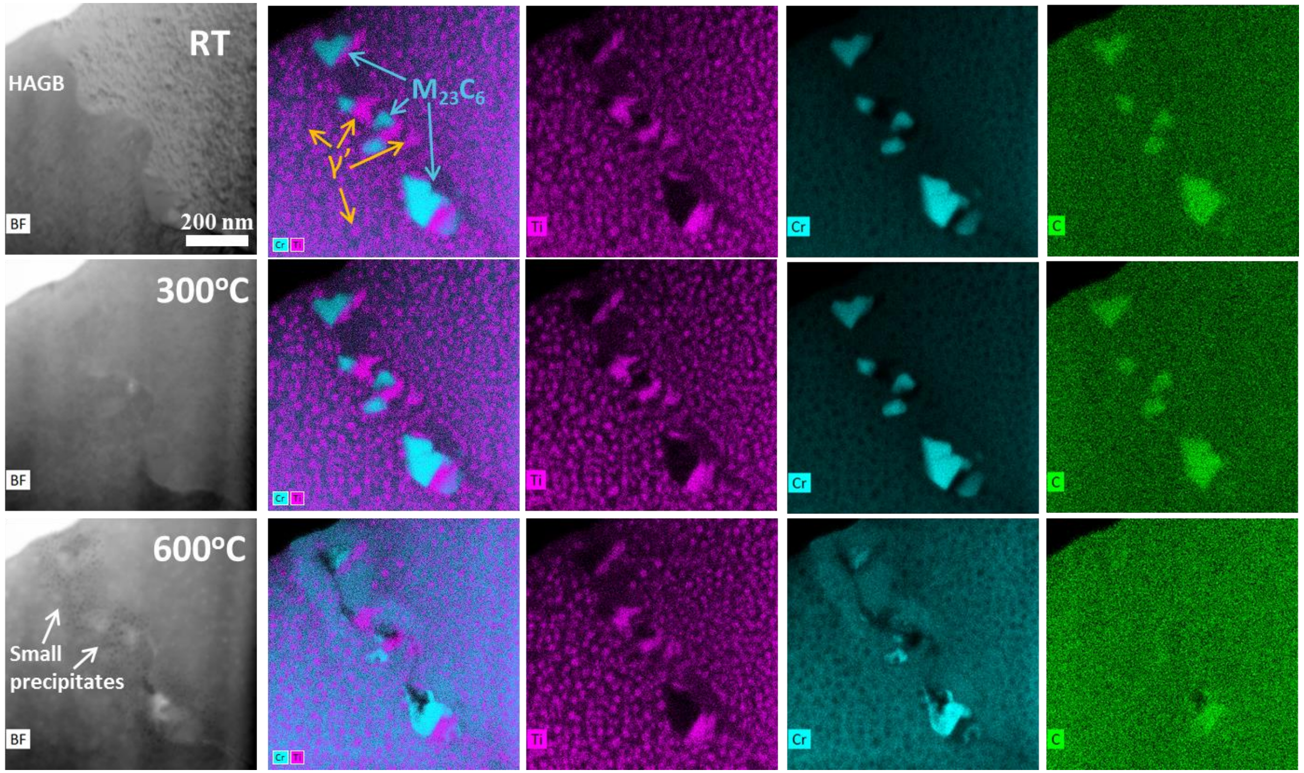
Cylinder: 50 nm X 10 nm X 10 nm

Transmission Electron Microscope: Probe Corrected Titan Themis 200





In Situ Heating Studies



X-750 alloy; the heating rate is 100°C/min.

- Titan Themis 200 (IMCL) with super-X EDS system

Continuum Duel Electron Energy Loss Spectroscopy (DuelEELS): light elements, chemical bonding, bubble pressure, etc.

C_S Probe Corrector: STEM resolution of 0.08 nm

- In Situ Holder

DENSSolutions D9 heating holder up to 1300°C

PI-95 PicoIndenter

Fischione tomography holders

Vacuum transfer holder

Thermal property cell (TPC) – high burnup fuel example

Thermophysical properties are critical for fuel performance (margin to melting, fuel rod pressurization, etc.)

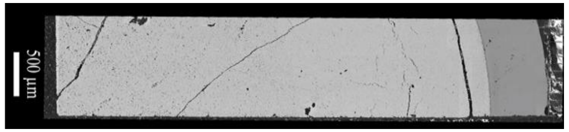
STA/MS – Simultaneous Thermal Analyzer/ Mass Spectrometer (RT to 2000°C)

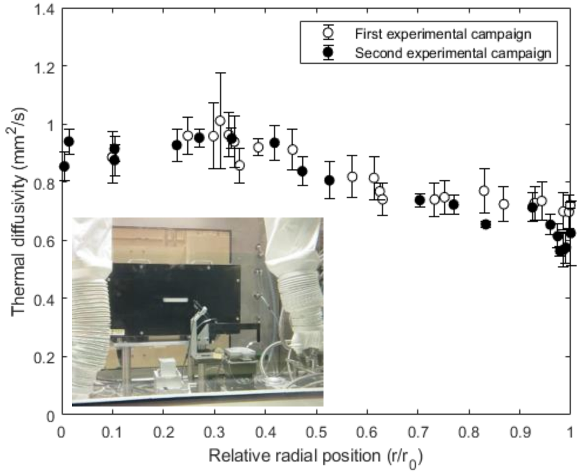
- Combination instrument that uses a thermal measurement furnace (DSC/DTA/TGA)
- Gaseous species and volatized materials released by temperature and/or pressure
- Post-irradiated materials for phase temperatures and enthalpy; specific heat; vapor pressure

TCM - Thermal conductivity microscope (RT)

- Thermal diffusivity, thermal conductivity at the meso-scale (resolution ≈ 50 μm)
- Example shows the degradation of thermal diffusivity in high burnup UO_2 from center to periphery

Radial section - 76 GWd/tHM UO_2

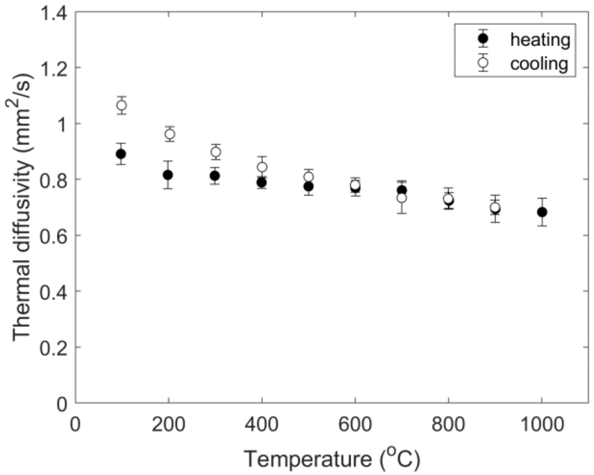




LFA – Laser Flash Analysis (RT to 2000°C)

- Thermal diffusivity, thermal conductivity
- Example below shows the evolution of thermal diffusivity as a function of temperature for high burnup UO_2.
- The annealing of defects at higher temperature leads to a recovery in thermal diffusivity and thermal conductivity upon cooling

Heating/ Cooling cycles of 67 GWd/tHM UO_2



Work supported by AFC campaign

Current IMCL Instrumentation

- Shielded FEI Quanta dual-beam focused ion beam (**FIB***)
- Shielded FEI Helios dual beam Xe plasma FIB (**P-FIB***)
- Shielded CAMECA SX-100R electron probe microanalyzer (**EPMA**)
- FEI Titan Themis 200 **TEM** with probe corrector and **EELS**
- Shielded Sample Preparation Area (**SSPA**)
- Shielded Thermal Property cell
  - **LFA**
  - **TGA**
  - **TCM**

- Unshielded JEOL 7600 **FEG-SEM**
- PANalytical XRD and Bruker **micro-XRD**
- Unshielded Zeiss Versa 520 **x-ray microscope**
- Quantum Design Physical Property Measurement System
- CAMECA LEAP 5000 **Atom Probe**
- Unshielded FEI Helios Hydra dual beam plasma **FIB/SIMS**
- Alemnis and PI-88 is-situ **nanoindenters**

Idaho National Laboratory

Battelle Energy Alliance manages INL for the U.S. Department of Energy's Office of Nuclear Energy. INL is the nation's center for nuclear energy research and development, and also performs research in each of DOE's strategic goal areas: energy, national security, science and the environment.