INL/MIS-22-67289-Revision-0

Standardized Irradiation Testing and Multi-Modal Characterization for Advanced Nuclear Materials

May 2022

Changing the World's Energy Future

Geoffrey L Beausoleil

Idaho National Laboratory

INL is a U.S. Department of Energy National Laboratory operated by Battelle Energy Alliance, LLC

INL/MIS-22-67289-Revision-0

Standardized Irradiation Testing and Multi-Modal Characterization for Advanced Nuclear Materials

Geoffrey L Beausoleil

May 2022

Idaho National Laboratory
Idaho Falls, Idaho 83415

http://www.inl.gov

Prepared for the
U.S. Department of Energy
Under DOE Idaho Operations Office
Contract DE-AC07-05ID14517, DE-AC07-05ID14517

AMMT Environmental Effects Testing and Characterization Roadmap for FY22-FY27

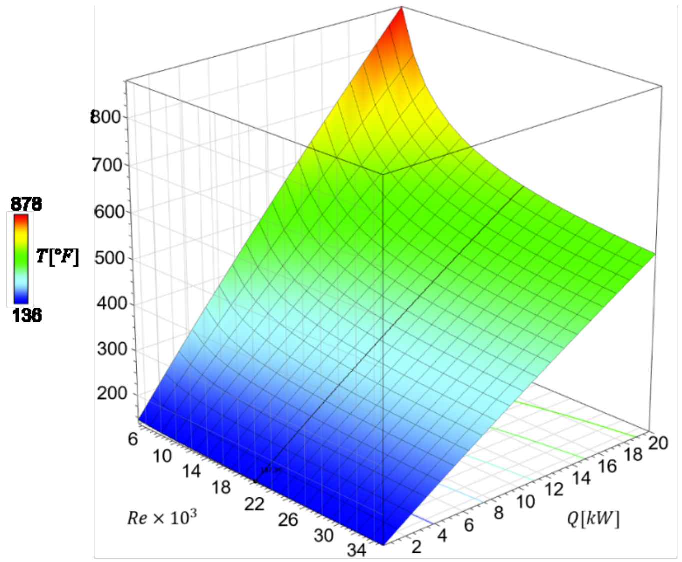
Idaho National Laboratory

INL Testing and Characterization Efforts

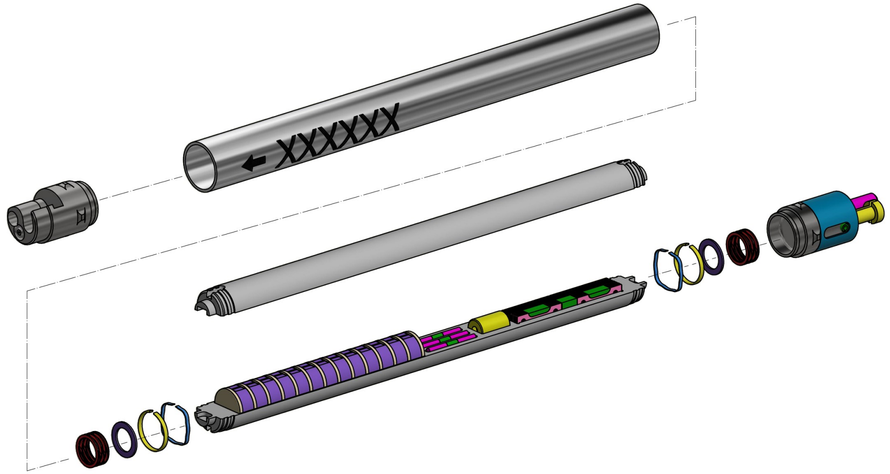
- Development of standard irradiation capsule ISHA-1
  - Using for SS316H and 16-8-2 Weld Metal
- In-Situ Property Measurement
  - Demonstration of single head thermal reflectance stage within G4 Hydra
  - Development of additional modalities using the feedthrough
- ANCERS, Application of Non-destructive Combinatorial Examination of Radioactive Samples
  - Design of Gamma-Ray Emission Tomography Assay (GRETA) table
  - Developing reconstruction methods using standard samples
- High temperature alloy characterization

FY21 - Universal Drop-in Capsule for ATR Testing

- Under NMDQi, we developed a semi-universal drop-in capsule for irradiation in the Advanced Test Reactor (ATR) called the Irradiation System for High-throughput Acquisition (ISHA-1). This utilizes a standard outer capsule that can facilitate both fuel experiments or structural material specimens in a variety of ATR Positions.
  - Structural material testing can support compact tension, bend bar, and tensile specimen geometries for temperatures up to 800 ˚C.
  - Utilizes concentric ASME pressure boundaries in order to support more reactive material irradiation testing within ATR, such as molten salts and other reactive materials.

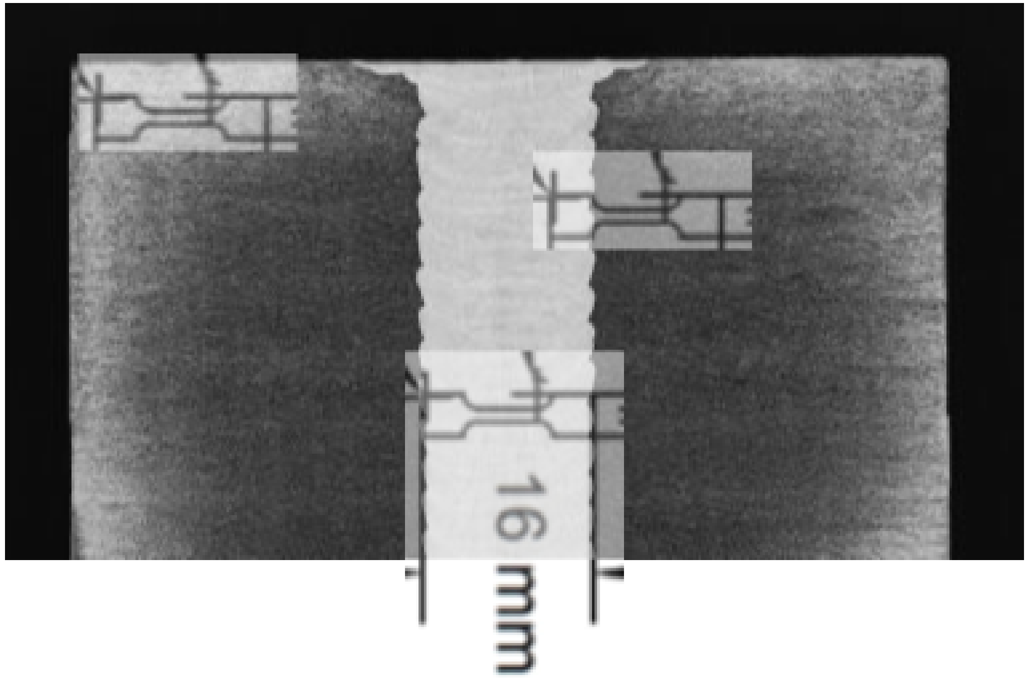


ISHA is a semi-universal drop-in capsule for ATR with an extensive, bounding safety case



SS316 Irradiation Test

- Partnership with Kairos Power ARDP award and NMDQi efforts
  - Irradiating welded SS316H with AWS 16-8-2 weld metal
  - Temperatures of 550 ˚C and 650 ˚C
  - Target doses of <1 dpa, 1-2 dpa, 10-15 dpa

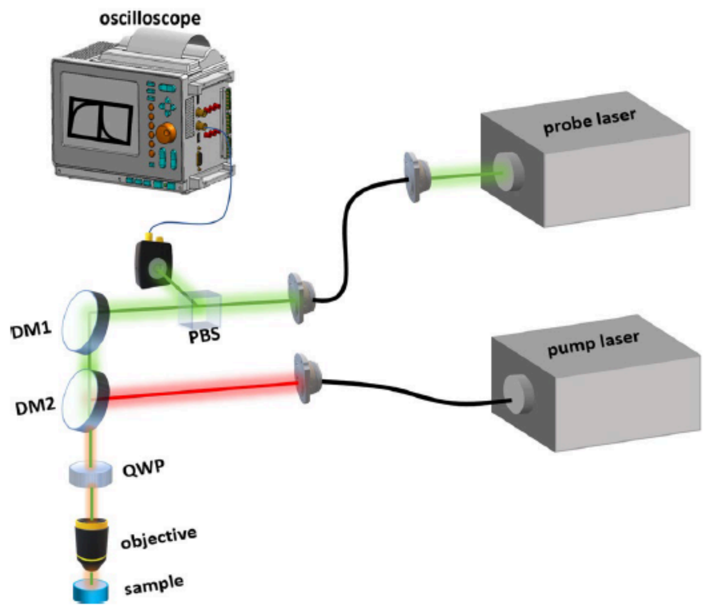


- Objectives will be to mechanically characterize difference between the base metal, weld metal, and heat affected zone
- National Reactor Innovation Center (NRIC) is funding the design, assembly, and deployment of three in-cell load frames to perform the mechanical tests
- Multiple creep rupture lifetimes being investigated
- FY22-FY27 effort
  - Irradiation and PIE funded through ARDP award

High temperature creep testing of 316 and small scale specimens

Thermal Reflectance Method and the SPTR

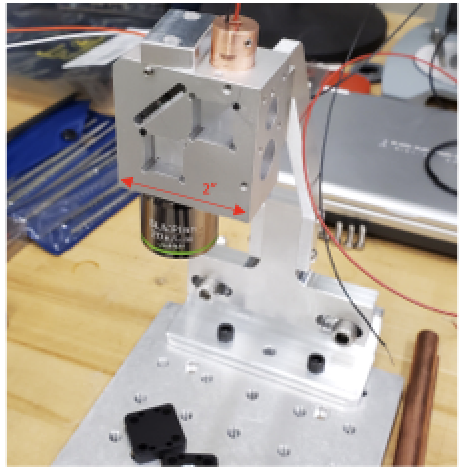
- Thermal reflectance methods utilize the small, local change in optical reflectance of a material based upon its temperature
  - Can compare temperature vs frequency (frequency domain) or temperature vs time (time domain)
  - SPTR utilizes a time domain interpretation
- Thermal reflectance is the basis for the Thermal Conductivity Microscope (TCM) at the Irradiated Materials Characterization Laboratory (IMCL)
- The square pulse thermal reflectance (SPTR) method was developed to support in-situ testing within the G4 Hydra PFIB/SEM
  - Spot sizes on the order of 2 μm on the specimen surface

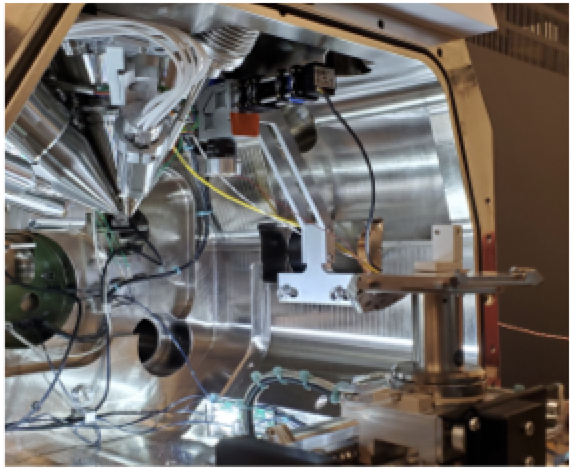


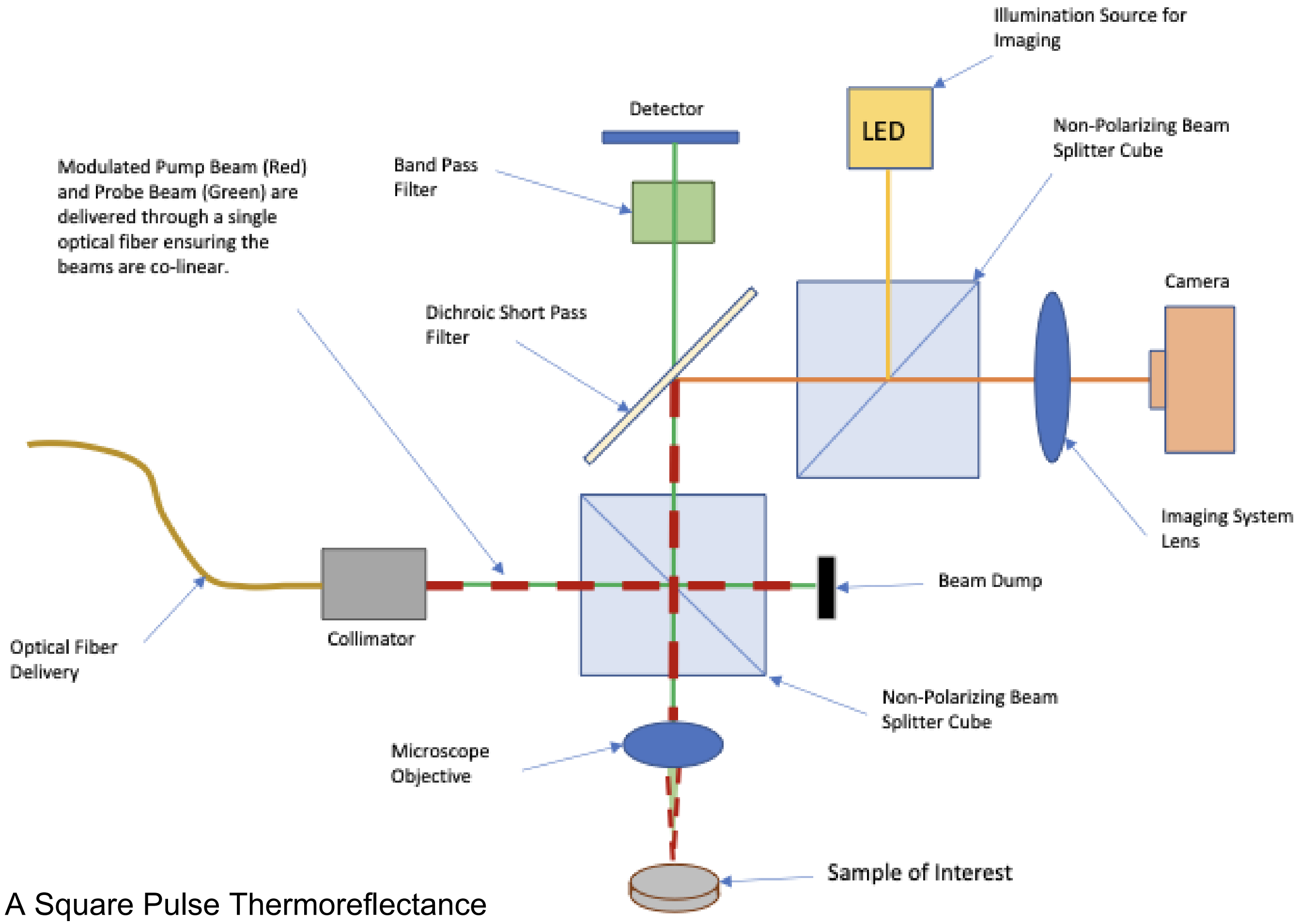
Explicitly correlating thermal/mechanical properties to microstructural features during PIE

Design of Thermal Reflectance Head in G4 Hydra

- In-Situ testing property testing during microscopy
- Stage compresses both the probe and pump lasers into a single optic that can support laser change outs from outside the SEM
- Feed through a vacuum port on the SEM chamber



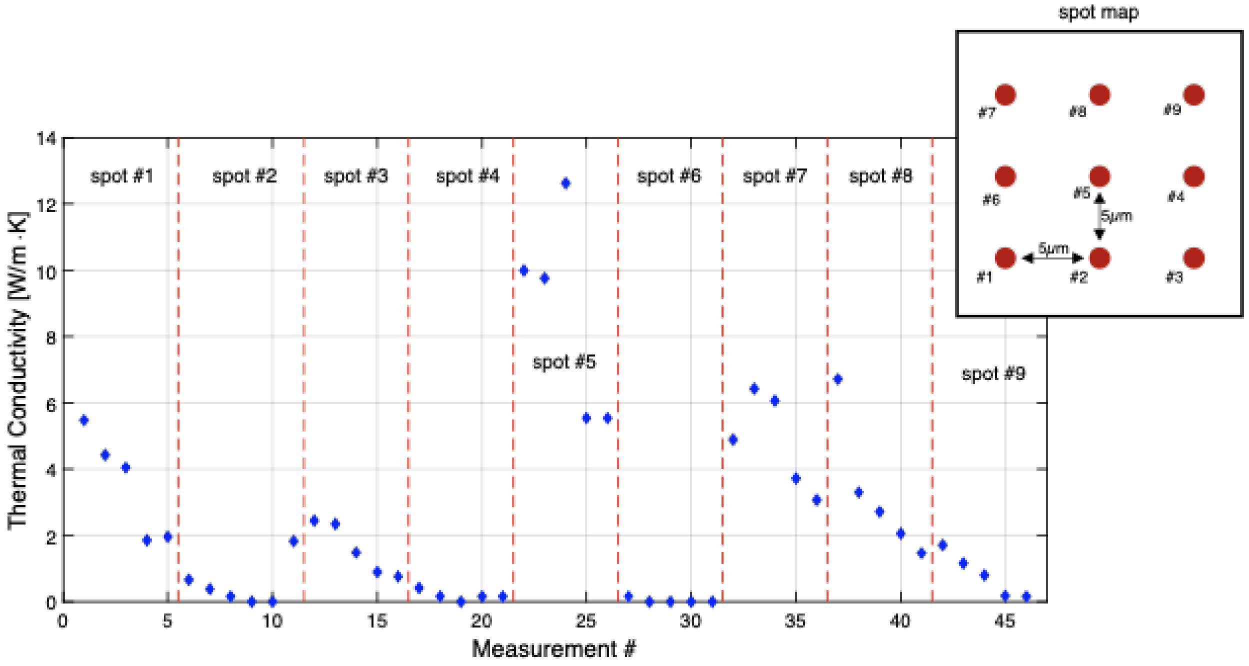


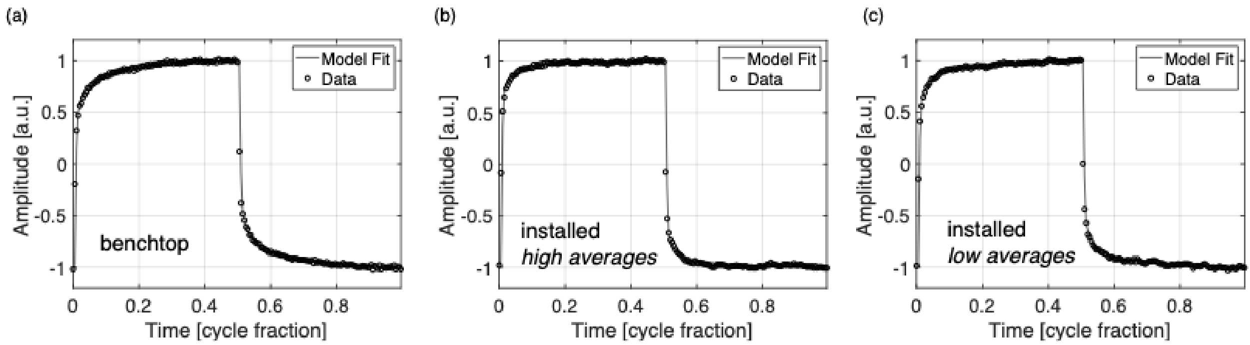


A Square Pulse Thermoreflectance Technique for the Measurement of Thermal Properties
Y Wang, V Chauhan, Z Hua, R Schley, CA Dennett… - International Journal of Thermophysics, 2022

Square Pulse Thermal Reflectance (SPTR) Results

- Investigation of the method focused on identifying sensitivities and proving the model
  - Spot size, film (transducer) size, and model variables
- Emphasis was placed on relationships between material conductivity and film thickness
- Validation with BK7 glass and gold films
  - Benchtop was within 3% of known values for BK7 (~ 1 W/m/K)
  - Installed values were initially not good (~3-4 W/m/K)



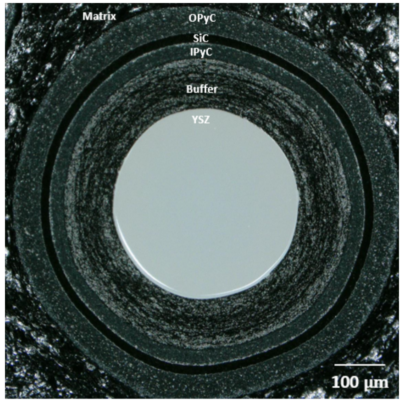


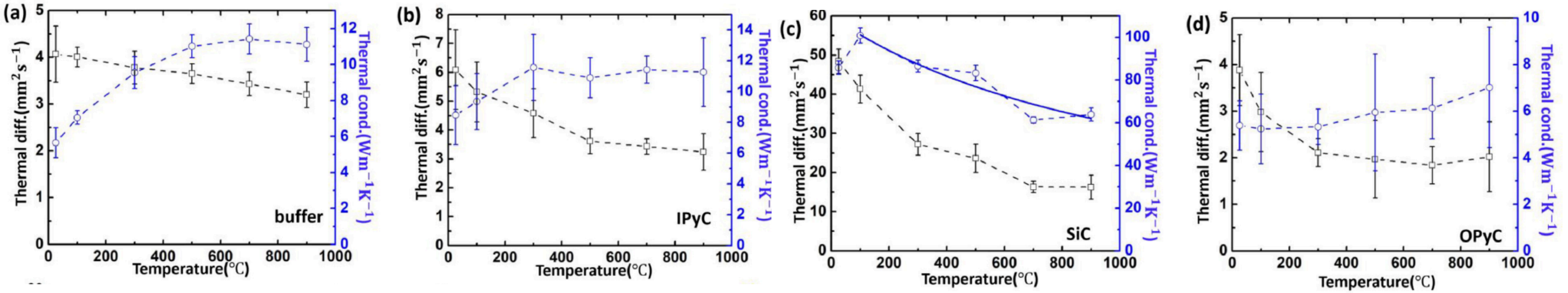
Investigation of TRISO Particle in G4

- A tri-isotructure (TRISO) fuel particle was used as a basis for demonstrating the SPTR instrument within the G4
  - Well established material properties for comparison
  - Small, well defined layers to distinguish the results from
- Results were also compared to a similar instrument, the TCM at IMCL

		SPTR (W/mK)*	TCM	
			Therm cond.(W/mK)	Therm diff.(mm^2/s)
RT	YSZ	2.22	2.65	0.9
	Buffer	3.64	3.25	3.25
	IPyC	4.75	5.16	5.56
	SiC	74.1	84.3	38.3
	OPyC	4.45	3.96	3.52

*assume heat capacity from literature

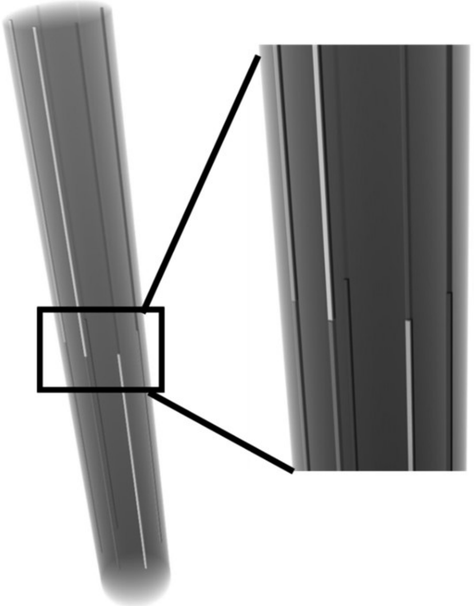




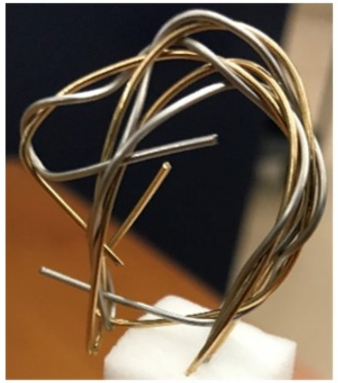
Thermal properties measurement of TRISO particle coatings from room temperature to 900 °C using laser-based thermoreflectance methods
Y Wang, Z Hua, R Schley, G Beausoleil II, DH Hurley - Journal of Nuclear Materials, 2022

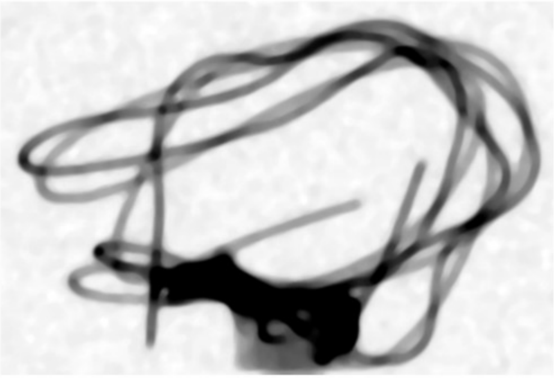
FY21: Began Development of Integrated Tomographic Methods for Characterization for Irradiated Materials

- To increase the throughput of materials testing and characterization, combinatorial tomographic methods are under development with the goal of increasing the scientific impact of analysis but combining the data output of a single sample characterized with multiple investigative methods.
  - Application of Non-destructive Combinatorial Examination of Radioactive Samples (ANCERS)
  - Provide isotopic and spatial resolution <10 μm on large, dense irradiated specimens



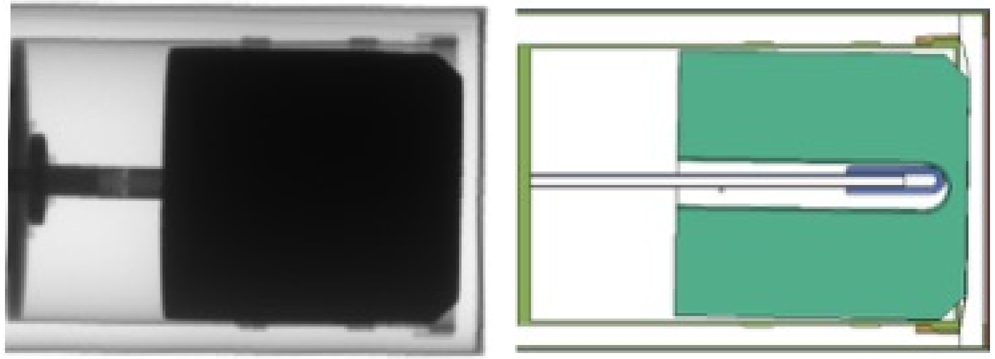
Combining tomographic methods allows each to make up for the others' shortcomings, thus improving the value proposition of tomography on irradiated materials

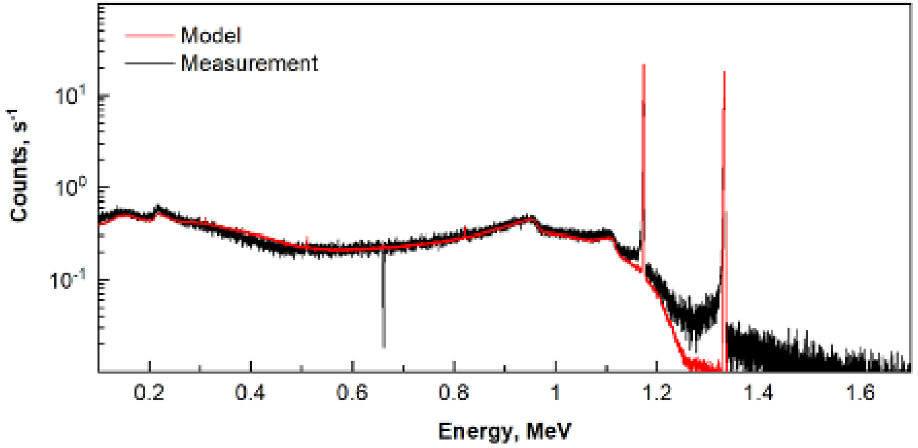


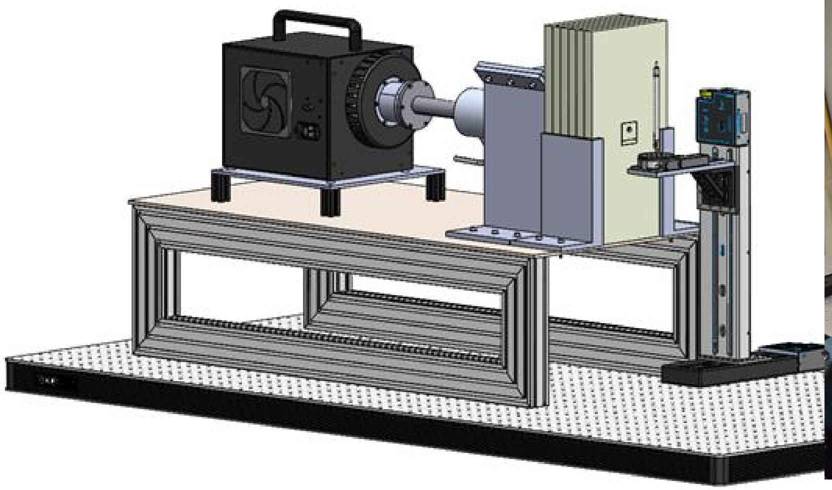


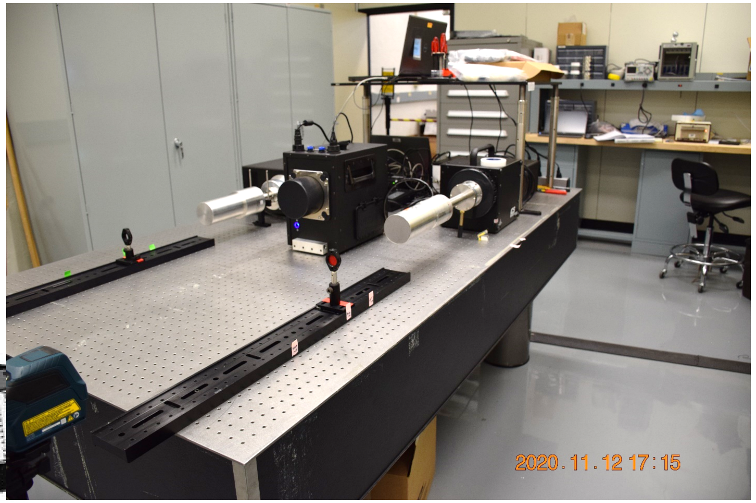
Gamma Ray Emission Tomography Assay (GRETA) System

- Gamma emission tomography (GET) instrument to support ANCERS
- A high purity Ge detector was received and characterized
- Unfortunately, there were some problems with the received detector and the vendor is in process of replacing it.

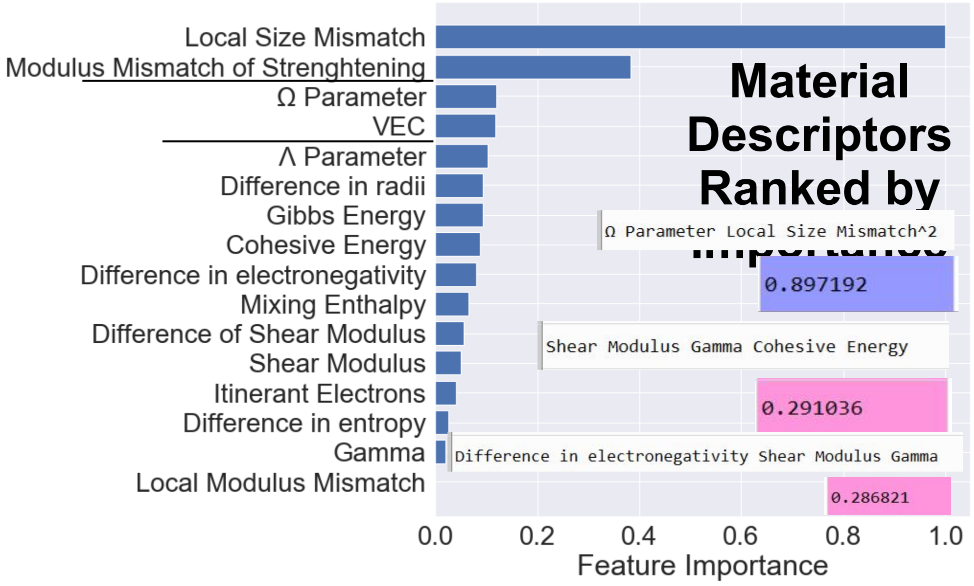








Multi-Principal Element Alloys for Nuclear Applications



B Group:

- FeCrNiCoCu
- FeCrNiCo
- FeCrNiMnCo

BB Group:

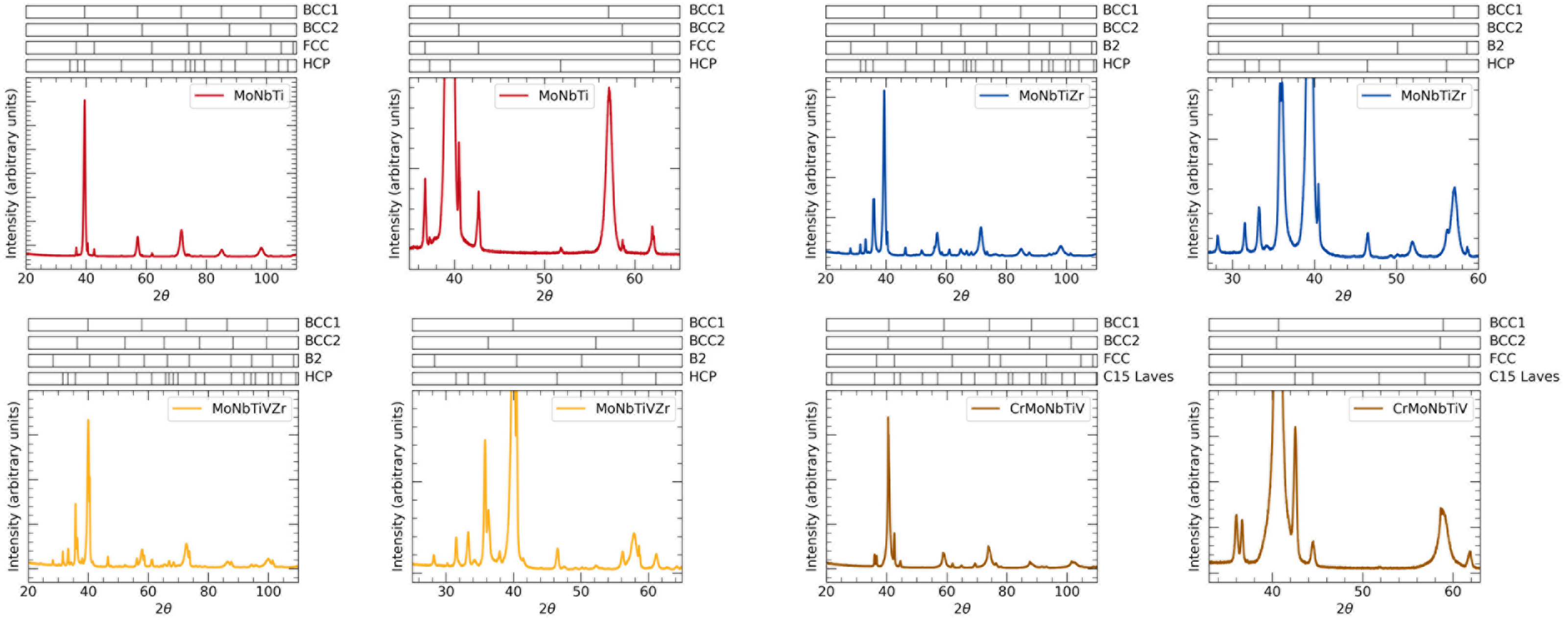
- FeCrNiMnSiC
- FeCrNiMnSiCMo
- FeCrNiMnAlCoSiCCuTi
- FeCrMn
- FeCrMnCo
- FeCrMn*

D Group:

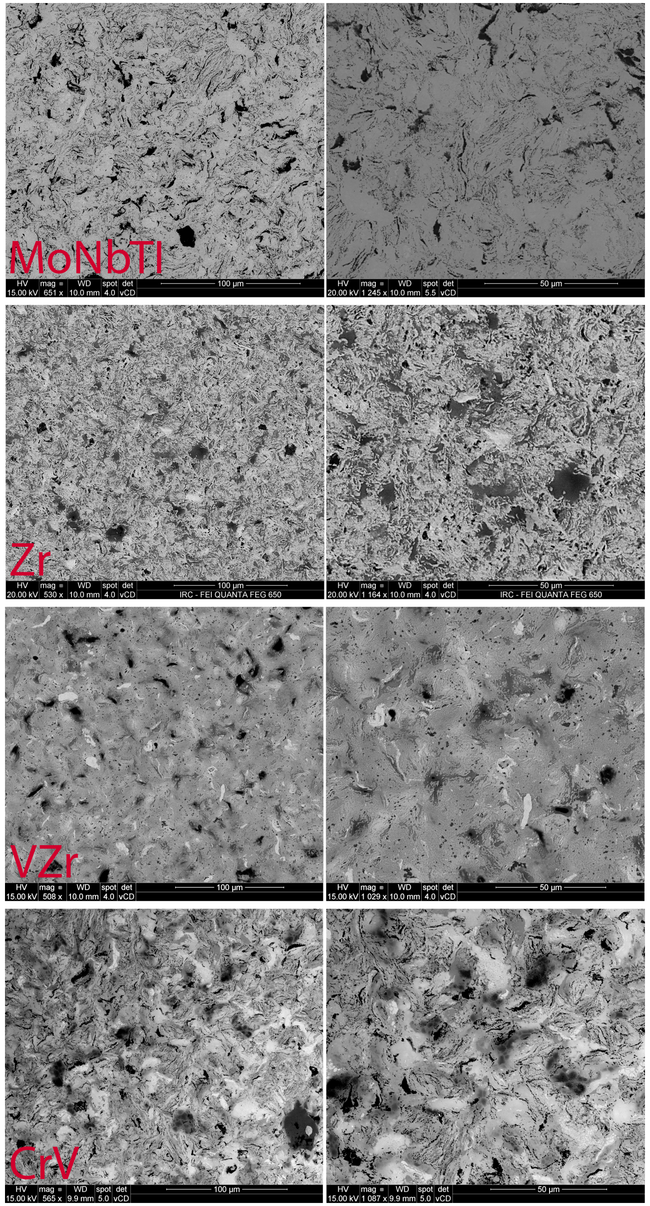
- CrAlZrMoNbTa
- CrAlZrMoNb
- CrAlZrMoNbN
- $CrAlZrMoNbNTa_2$
- TiZrVMoNb
- $Ti_2ZrHfV_{0.5}Mo_{0.2}$
- TiZrHfNb
- TiZrHfNbTa
- FeCrVTaW
- CrVTaW
- CrMnVTaW

ds

- sdfsd



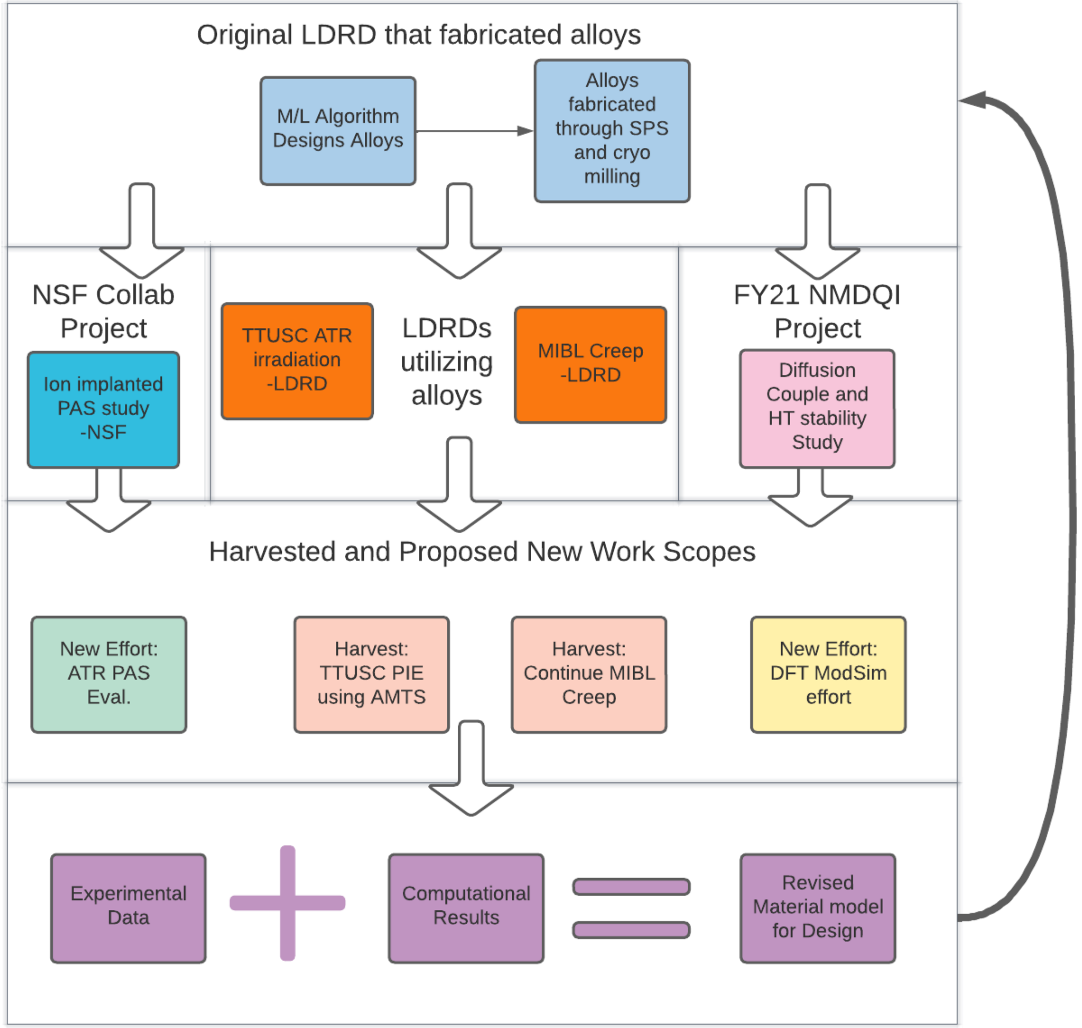
BCC1
BCC2
FCC
HCP
B2
C15 Laves
MoNbTi
MoNbTiZr
MoNbTiVZr
CrMoNbTiV
Intensity (arbitrary units)
2θ



MoNbTI
Zr
VZr
CrV

FY23-FY25 Harvested LDRDs

- Advanced Alloy Design
  - Machine learning algorithm to predict material mechanical properties
  - Cryogenic milled powders and SPS to fabricate alloys
  - Phase and mechanical testing
- High temperature phase stability and property assessment
- TTUSC – Neutron irradiation and mechanical testing
- MIBL Creep Rig – in-situ ion irradiation and creep testing
- NSF Positron Annihilation Spectrometry
- Addition of ModSim development for atomistic behaviors



Demonstrates experimental framework for testing new alloys

Utilizing ATF-2 Irradiation Opportunities

- Vendors often miss insertion dates and are replaced by dummy capsules
  - Conditions are within a flux trap typical of PWR core
  - 300 series SS
  - Simple cylinder for analysis credit
  - Typical minimum irradiation time of 2-3 cycles in the position
- The source of these cylinders is somewhat irrelevant and so the option of fabricating A/M SS316 cylinder
  - Fabricate a collection of A/M SS316 capsules with varying parameters and keep on hand to perform irradiations when available

Essentially a ‘free’ ATR irradiation in PWR Conditions


INL
Idaho National Laboratory
NORTH

High Temperature Multi-Principal Element Alloys