

# Postirradiation Capabilities at the Idaho National Laboratory

Robert D. Mariani  
Rory Kennedy  
Doug Toomer

August 2011



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Idaho Falls, Idaho 83415**

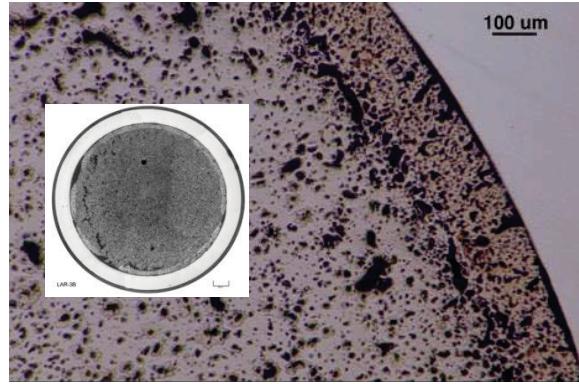
**<http://www.inl.gov>**

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August 2011

Idaho National Laboratory  
Idaho Falls, Idaho, USA



## For Additional Information

Robert D. Mariani  
Manager Fuel Performance and  
Design Department  
[robert.mariani@inl.gov](mailto:robert.mariani@inl.gov)

Rory Kennedy  
Manager Fundamental Fuel  
Properties and Characterization  
Department  
[rory.kennedy@inl.gov](mailto:rory.kennedy@inl.gov)

Doug Toomer  
Manager Industry Programs  
Nuclear Fuels and Materials  
Division  
[douglas.toomer@inl.gov](mailto:douglas.toomer@inl.gov)

## 1. Introduction [1, 2, 3]

The U.S. Department of Energy (DOE) Office of Nuclear Energy (NE) oversees the research, development, and demonstration activities that ensure nuclear energy remains a viable energy option for the United States. Fuel and material development through fabrication, irradiation, and characterization play a significant role in accomplishing the research needed to support nuclear energy. All fuel and material development requires the understanding of irradiation effects on the fuel performance and relies on irradiation experiments ranging from tests aimed at targeted scientific questions to integral effects under representative and prototypic conditions.

The DOE recently emphasized a solution-driven, goal-oriented, science-based approach to nuclear energy development. Nuclear power systems and materials were initially developed during the latter half of the 20<sup>th</sup> century and greatly facilitated by the United States' ability and willingness to conduct large-scale experiments. Fifty-two research and test reactors with

associated facilities for performing fabrication, characterization and postirradiation examinations were constructed at what is now Idaho National Laboratory (INL), another 14 at Oak Ridge National Laboratory (ORNL), and a few more at other national laboratory sites. Building on the scientific advances of the last several decades, our understanding of fundamental nuclear science, improvements in computational platforms, and other tools now enable technological advancements with less reliance on large-scale experimentation.

Advanced tools and techniques support the science-based approach by enabling an understanding of the behavior of fuels and materials in a nuclear reactor irradiation environment at the nano-scale and smaller. Understanding of nuclear fuel and material performance in the nuclear environment at these scales is critical to the development of the innovative fuels and materials required for tomorrow's nuclear energy systems since these are the scales at which irradiation damage occurs. This drives the need for PIE capability for highly activated fuels and materials at ever increasing levels of sophistication.

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## 2. Current core capabilities [2, 4, 9]

Idaho National Laboratory is the United States' only DOE laboratory with a core mission of supporting the development of nuclear energy. INL has a long history of performing PIE to support various DOE programs and has an existing trained workforce, with considerable expertise in fuels and materials technology. This expertise forms a foundation to build upon as new capabilities are implemented and techniques are developed to support the research interests of customers.

At INL, the core nuclear and radiological facilities needed to support PIE R&D capabilities include:

- (a) Hot Fuel Examination Facility (HFEF) with neutron radiography capability, a large inert environment hot cell facility with the ability to receive and process large material and fuel components,
- (b) Analytical Laboratory (AL), conducts analysis of irradiated and radioactive materials
- (c) Electron Microscopy Laboratory (EML), radiological facility contains optical, scanning, and analytical microscopes
- (d) Fuels and Applied Science Building (FASB), conducts fuel development, materials characterization and irradiated materials testing
- (e) Carbon Characterization Laboratory (CCL) conducts both pre-irradiation and post-irradiation material property

- (f) measurements of carbon/graphite materials. **Center for Advanced Energy Studies (CAES)**, a user facility by design, the CAES research facility and Microscopy and Characterization Suite (MaCS) operates in the same manner as universities do; in the case of low risk radiological research, this approach provides a cost-effective, innovative, and productive environment for exploring fundamental science questions and executing basic research complementary to research at INL Site facilities.

The majority of these facilities exist at MFC while CCL and CAES are located at the INL Research and Education Campus (REC). INL also has close proximity to irradiation



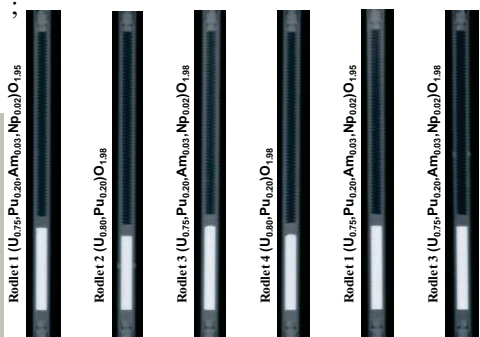
facilities like the Advanced Test Reactor (ATR) and the Transient Reactor Experiment and Test (TREAT) facility. INL's current capabilities are outlined in more specific detail in the following Tables. Within this detailed information are geometry limits for equipment; however larger items can often be accommodated. Recently many of these current capabilities, particularly the macroscopic examination capabilities, have undergone or been scheduled for refurbishment. The combined capabilities at INL now provide the most comprehensive PIE capability in the United States. Because of the comprehensive PIE capability INL can perform a multitude of analysis to learn about the properties of a material including highly irradiated and activated materials. This includes performing analysis on metal fuels, ceramic fuels, and many other highly activated materials.

CAES Website:

[https://inlportal.inl.gov/portal/server.pt/community/caes\\_home/281/macs\\_home](https://inlportal.inl.gov/portal/server.pt/community/caes_home/281/macs_home)

## Non-Destructive Examination Capabilities

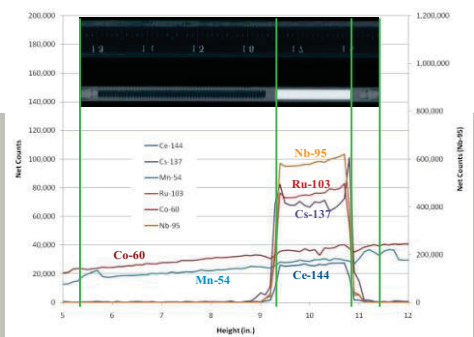
Capability / Equipment	Application	Description
Neutron Radiography - HFEF  (Upgrade to the control console currently underway)	Examine fuel elements to identify areas of interest: locate fuel for disassembly, identify cracking, density variations, hydrides in cladding	250 kW TRIGA reactor, two beam tubes and two separate radiography stations. Max Length: 152 in. Max Diameter: 6.5 in (round) or 4.5 X 8.5 (rectangular) Max Weight: 600 lbs
Precision Gamma Scanning - HFEF	Determine relative burnup profile; investigate constituent migration in irradiated fuels, pellet-pellet interfaces, and relative distribution of various isotopes of interest in fuel. Measures fission and activation-product activity distribution.	<ul style="list-style-type: none"> <li>• 4 Degrees of freedom: x, y, z, 180°</li> <li>• Multi-channel Analyzer w/ software Library</li> <li>• Vertical Step Travel Limit 0.01 in./step (0.05 - 0.1 in. typical)</li> </ul>
Dimensional Inspection -HFEF  (Currently undergoing upgrade to allow rotation of pin while scanning diameter. The spiral capability will support wire-wrap pins.)	<ul style="list-style-type: none"> <li>• Measure diameter/plate thickness</li> <li>• Cladding creep down in-reactor service and creep out during dry storage</li> <li>• Irradiation induced swelling</li> <li>• Fuel rod growth</li> </ul>	<ul style="list-style-type: none"> <li>• Continuous contact profilometer. ----- Measures diameter profiles.</li> <li>• Element contact profilometer: diam. <math>\pm 0.0002</math> in. (<math>\pm 0.0051</math> mm)</li> <li>• Max Length: 96 in. (Additional length is possible)</li> </ul>
Element/Capsule Bow and Length examination - HFEF	Measures distortion (bow) and length of fuel elements.	<ul style="list-style-type: none"> <li>• Bow and Length: <math>\pm 0.02</math> in. (<math>\pm 0.51</math> mm)</li> <li>• Max Length: 96 in. (Additional length is possible)</li> </ul>
Visual Exam - HFEF	Digital Still/Video Camera Photography, full color with grey balance for gross surface anomaly identification and documentation.	Dedicated workstation and in-cell exam stage, thru hot cell window, Thru-cell wall periscope 2X, 10X, 25X.
Eddy current examination - HFEF  (Upgrade currently underway to improve sensor geometry / resolution and to improve the element handling)	<ul style="list-style-type: none"> <li>• Non destructively evaluate cladding performance, measure oxide formation on cladding</li> <li>• Detect and characterize material surface defects</li> <li>• Non-destructively measure oxide layer thickness</li> <li>• Measures electrical current induced when a conductor is placed in a region of shifting magnetic flux</li> </ul>	<ul style="list-style-type: none"> <li>• Measures surface and material defects, ability to set stop points for repeatable results</li> <li>• Max size: 1 in. D x 154 in.</li> <li>• Oxide thickness uncertainty: <math>\pm 5</math> <math>\mu</math>m</li> </ul>



Left: Neutron Radiography

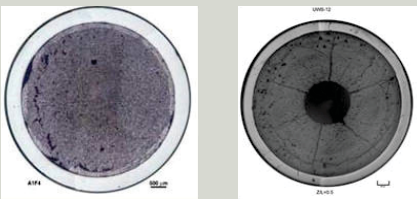
Right: Gamma Scanning

Neutron radiography and gamma scan analysis are primarily used to study gross fuel structural behavior, burnup, constituent redistribution. The examples shown here are for minor actinide bearing MOX fuel designed for fast reactors.





## Sample Preparation and Physical Properties Capabilities

Capability / Equipment	Application	Description
Gas Sampling (GASR) - HFEF	Capture sample to determine fission gas and helium release	<ul style="list-style-type: none"> <li>• Laser puncture and gas collection</li> <li>• Fuel rod internal void volume and gas pressure, +/- 5%</li> <li>• Plenum gas batch sample chemical analysis</li> </ul>
Test disassembly, sample retrieval, sample machining - HFEF  (New Electric Discharge Machine (EDM) planned for installation in IMCL)	<ul style="list-style-type: none"> <li>• Disassemble irradiation experiments and retrieve samples for testing.</li> <li>• Disassemble irradiation experiment capsules</li> <li>• Retrieve reduced size samples for SEM / TEM analyses in EML</li> <li>• Evaluate irradiated plate fuel residual bond strength</li> <li>• Finite element analysis for deriving properties from specialized geometries</li> <li>• Machine complex specimen geometries from irradiated materials and cladding from LWR and advanced reactors</li> </ul>	<ul style="list-style-type: none"> <li>• Table top Mill: 7 x 5 x 5 in.</li> <li>• SEM Punch</li> <li>• Furnace temp: 1200°C</li> <li>• Furnace cavity: 6" D x 24" H</li> <li>• RAM Electric Discharge Machining (EDM)</li> </ul>
Physical properties testing	<ul style="list-style-type: none"> <li>• Characterize effects of irradiation on basic physical properties.</li> <li>• Provide data necessary to identify fundamental mechanisms and develop predictive models</li> <li>• Immersion density measurements using Archimedes Principle</li> <li>• Density to quantify irradiation swelling</li> <li>• Density as input to derive thermal conductivity</li> <li>• Hydrogen measurement by fusion of sample</li> <li>• Separate released gas and quantify by thermal conductivity</li> <li>• Hydrogen absorption and hydrogen embrittlement</li> </ul>	<ul style="list-style-type: none"> <li>• Immersion density: <math>\geq 0.25</math> g sample, 1RSD: <math>\leq \pm 1\%</math></li> <li>• Gas pycnometry: <math>\geq 1</math> cm<sup>3</sup> sample, 1RSD: <math>\leq \pm 1\%</math></li> <li>• LECO RH-400 H<sub>2</sub> Determinator modified for remote operation</li> <li>• Hydrogen content: 0.2 g sample, 1RSD: <math>\pm 2.5\%</math></li> </ul>
Metallography / Ceramography  	<p>Characterize irradiated fuel grain size and morphology, porosity, phase, fuel-cladding interaction</p> <p>Measure cladding oxide thickness, hydride distribution</p> <p>Measure hardness profiles</p>	<ul style="list-style-type: none"> <li>• Leitz MM5 RT Metallograph (80X to 800X) (up to 1500X at EML)</li> <li>• Microindenter Hardness Tester; LECO AMH43 – HFEF, EML</li> <li>• Automatic stage control</li> <li>• Integrated data collection and analysis</li> <li>• Digital Still Photographic Image</li> <li>• Common etchants include: Hydrofluoric acid, Nitric acid, Hydrochloric acid, and Ammonium hydroxide</li> </ul>

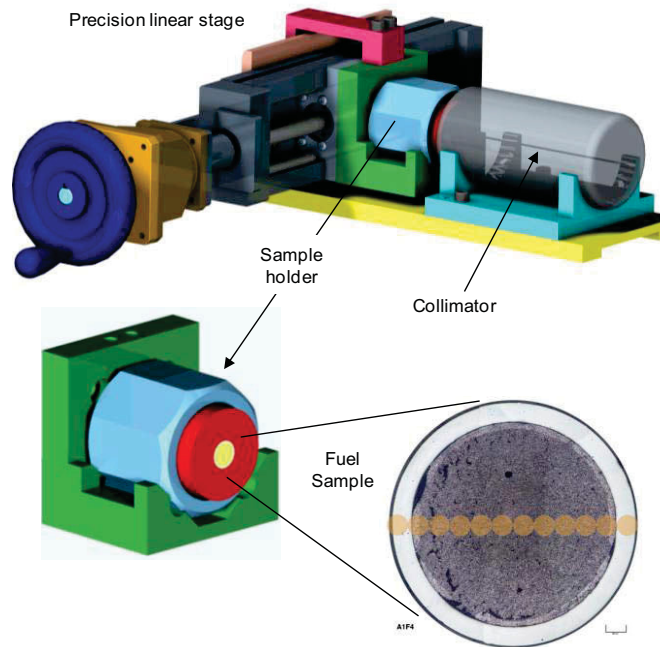
*Left: Metallography samples*

## Chemical, Isotope, and Radiological Analysis Capabilities

Capability / Equipment	Application	Description
Inductively Coupled Plasma Mass Spectrometry(ICP-MS, also Multi-Collector ICP-MS (MC-ICP-MS)) with Dynamic Reaction Cell (DRC), - AL	To mitigate isobaric interferences and obviate chemical separation	U, Pu isotopics: $<\pm 1.0\%$ Fission product isotopes: $\pm 2\%$ Elemental analysis: $\pm 2-5\%$ NIST traceable standards
Inductively Coupled Plasma Atomic Emission Spectroscopy(ICP-AES)	Measure bulk isotopic and chemical composition of actinide fuel samples as-fabricated and post-irradiation	U, Pu isotopics: $<\pm 1.0\%$ Fission product isotopes: $\pm 2\%$ Elemental analysis: $\pm 2-5\%$ NIST traceable standards
Carbon, Oxygen, Nitrogen, Hydrogen and Sulpher Analysis; – AL  Non high rad materials	This multi-element analyzer measures the common elements: hydrogen, nitrogen, oxygen, sulfur, carbon	Provides elemental Analysis  ONH-2000 by Eltra Analyzers Accuracy: $\pm 1\%$  CS-800 by Eltra Analyzers Accuracy: $\pm 0.5\%$
Thermal Ionization Mass Spectrometry (TIMS) - AL	Perform nuclear material accountability measurements by Thermal Ionization Mass Spectrometry (TIMS) isotope dilution, can do isotopic analysis on a single element.	U Typical error $\pm 1.9\%$ Pu Typical error $\pm 1.9\%$
Gas Mass Spectrometry  (Gas mass spectrometer is currently undergoing upgrade. At this time, gas samples are being transported to PNNL for analysis.)	Gas mass spectrometry (GMS) is a method that combines the features of gas-liquid chromatography and mass spectrometry to identify different substances within a test sample. Additionally, it can identify trace elements in materials that were previously thought to have disintegrated beyond identification.	Determines fission product gasses i.e. Xenon, Krypton
Isotope Mass Separator	Produces isotopically pure compounds	i.e. Ba-134
Alpha Spectroscopy Analysis	An Alpha Particle X-Ray Spectrometer (APXS) is a device that analyses the chemical element composition of a sample from the scattered alpha particles, emitted protons, and fluorescent X-rays after the sample is irradiated with alpha particles and X-rays from radioactive sources.	
Beta Spectroscopy Analysis	An instrument used to determine the energy distribution of beta particles and secondary electrons. Also known as beta spectrometer.	

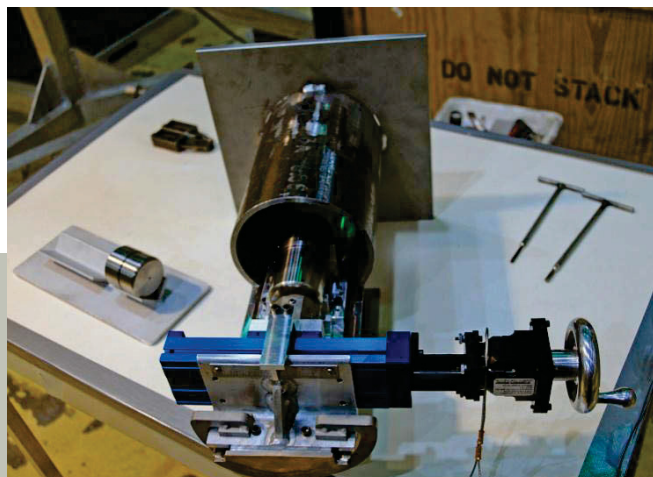
## Chemical, Isotope, and Radiological Analysis Capabilities

Capability / Equipment	Application	Description
Gamma Spectroscopy Analysis	Determine gamma emitting species in PIE samples	i.e. Cs-137, Ag-110
Micro-Gamma Scan Analysis	Determine radial burnup profile and investigate constituent migration in irradiated fuels	Enhancement of Analytical Laboratory Gamma Scan System with a precision linear stage and integral sample holder with a Tungsten Collimator Spot Size: 0.020 in. (working on an upgrade for Spot Size of 0.010 in.)



*The INL Analytical Laboratory provides services for analytical chemistry of fission products, actinides, and other radionuclides in various matrices.*

*Above and Right: Micro Gamma Scan Analysis*

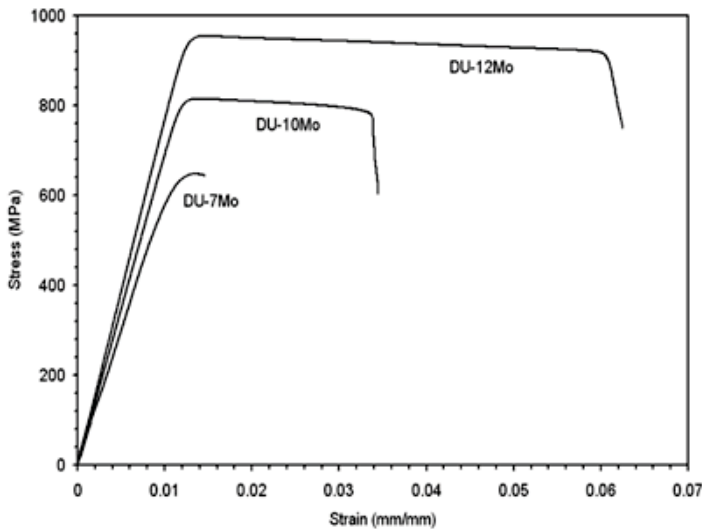




## Mechanical Property Examination Capabilities

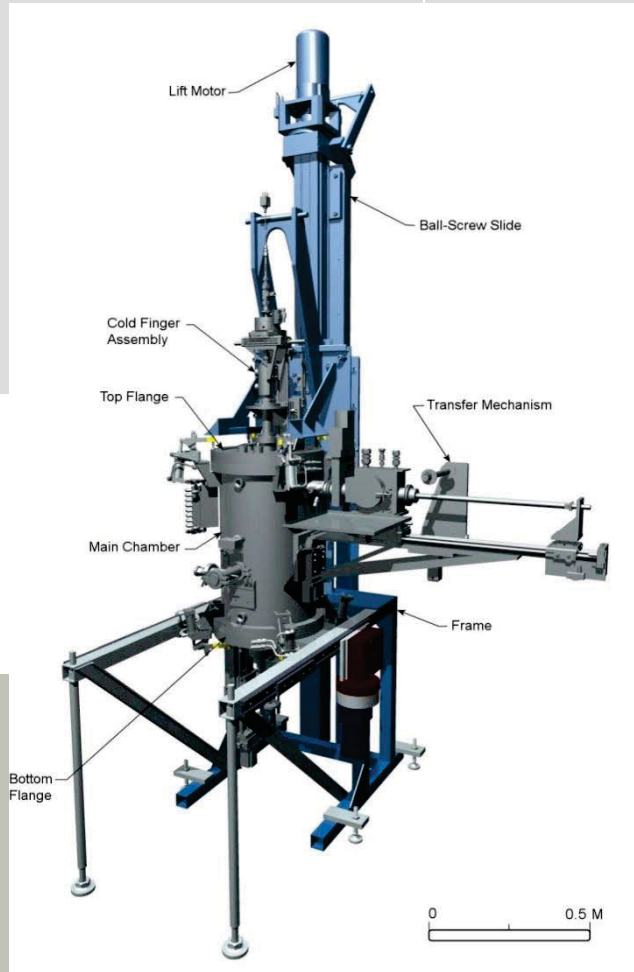
Capability / Equipment	Application	Description
Tensile testing and Shear punch testing / FASB or HFEF Instron tensile tester w/ furnace (1600°C)	<p>Provides engineering mechanical data on ASTM standard specimens</p> <p>Determine tensile properties/ductility for axial and circumferential directions at room temperature and elevated temperature prototypic of reactor operation or storage</p> <p>Ring (hoop) tensile tests Ring (hoop) compression tests Uni-axial tensile tests Uni-axial tensile tests at temperatures Tensile tests at high and low strain rates Compression tests</p>	<ul style="list-style-type: none"> <li>50 kN Instron tensile testing system installed in HFEF and qualified</li> <li>Instron Model 5869: 50 kN, 0.001-500 mm/min Furnace: 1200°C, Inert atmosphere Inspection tolerance <math>\pm 0.0005</math> in</li> </ul>

### Tensile Testing



## Furnaces Capabilities

Capability / Equipment	Application	Description
Fuel Accident Conditions Simulation Furnace (FACS) - HFEF	<p>Fuel annealing furnace for fission gas release studies and high temperature accident testing of HTGR fuel</p> <p>Heat irradiated fuel in helium sweep gas (<math>T \leq 2000^{\circ}\text{C}</math>)</p> <p>Condense fission products during annealing on water-cooled cold plate for subsequent measurement</p> <p>Collect and measure released fission gases (Kr, Xe) in cryo traps</p>	<ul style="list-style-type: none"> <li>• Max temp: <math>2000^{\circ}\text{C}</math></li> <li>• Graphite heating element</li> <li>• Helium atmosphere (@ ambient pressure)</li> <li>• Hot zone: <math>\sim 3.25''</math> diameter x <math>\sim 6''</math> high</li> <li>• Computer controlled operation</li> <li>• Automated cold plate exchange</li> </ul>
Blister Annealing Furnace	<ul style="list-style-type: none"> <li>• Anneal plates until blister forms on surface</li> <li>• Test of clad/fuel bonding</li> <li>• Combined with GASR to determine failure mode</li> </ul>	<ul style="list-style-type: none"> <li>• Furnace capacity: 6 inch diameter and 24 inch depth</li> <li>• <math>1200^{\circ}\text{C}</math></li> </ul>
<p>Mechanistic Fuel Failure Examination System (MFFES) – HFEF</p> <p>Planned for future installation</p>	Blister annealing and melting of fuel plates with a Argon sweep gas to carry fission products past detection systems to determine mechanistic failure modes	<p>Main Furnace:</p> <ul style="list-style-type: none"> <li>• High Vacuum</li> <li>• Graphite Resistance Elements</li> <li>• T Range <math>0-2000^{\circ}\text{C}</math></li> <li>• Argon Sweep Gas</li> <li>• Water Cooled</li> <li>• <math>10 \times 30.5</math> cm Heat Zone</li> <li>• Clam Shell Access</li> <li>• Light and Camera Ports.</li> </ul> <p>Thermal Gradient Furnace:</p> <ul style="list-style-type: none"> <li>• Connects To Main Furnace</li> <li>• Six Discreet Heat Zones</li> <li>• Clam Shell Design</li> <li>• Heat Zone <math>2.5 \times 76</math> cm</li> <li>• T Range <math>0-900</math> C.</li> </ul>



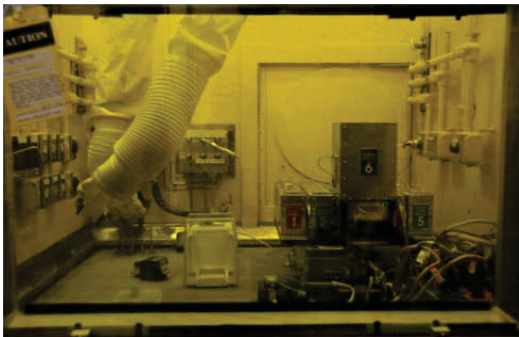
*Fuel Accident Conditions Simulation Furnace*

## Thermal Property Examination Capabilities

Capability / Equipment	Application	Description
<p>Thermal diffusivity (Laser Flash method (LFD), Scanning Thermal Diffusivity Microscope (STDM), Laser Resonance (LRUS))</p> <p>LFD is in a glovebox in AL for alpha producing materials, and in FASB for low-field beta/gamma materials. A remote system will be installed in IMCL for high hazard materials</p> <p>STDM, a first generation system, developed by INL, is currently installed in a hot cell in AL, a second system will be installed in IMCL</p>	<p>Reliable measurement of thermal diffusivity in many cases can be obtained through the laser flash technique. In this method, a short pulse (less than 1 millisecond) of heat is applied to the front face of a specimen using a laser flash, and the temperature change of the rear face is measured with an infrared (IR) detector. LFTD is designed to acquire data automatically, from cryogenic temperatures up to 2500°C. Data analysis algorithms interpret the rear face temperature response and provide instrumental and specimen dependent corrections. LFTD is used in the study of microstructural factors affecting the thermal transport properties of materials.</p> <p>Thermal diffusivity to quantify conductivity changes with irradiation</p>	<ul style="list-style-type: none"> <li>• STDM Spot Size 50µm</li> </ul>
<p>Differential Scanning Calorimetry (DSC)</p> <p>AL – for alpha producing materials FASB – for low-field beta/gamma materials</p> <p>New DSC planned for AL Fresh Fuels Glovebox</p> <p>A future system for remote use will be developed and installed in the IMCL for high radiation/hazard materials</p>	<p>Differential scanning calorimetry or DSC is a thermoanalytical technique in which the difference in the amount of heat required to increase the temperature of a sample and reference are measured as a function of temperature. Both the sample and reference are maintained at nearly the same temperature throughout the experiment. Generally, the temperature program for a DSC analysis is designed such that the sample holder temperature increases linearly as a function of time. The reference sample should have a well-defined heat capacity over the range of temperatures to be scanned. The main application of DSC is in studying phase transitions, such as melting, glass transitions, or exothermic decompositions. These transitions involve energy changes or heat capacity changes that can be detected by DSC with great sensitivity.</p>	

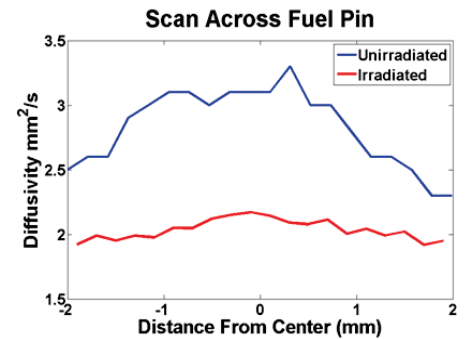
## Thermal Property Examination Capabilities

Capability / Equipment	Application	Description
Thermogravimetric Analysis (TGA) / Thermomechanical Analyzer (TMA) – FMF, AL  New TGA/TMA planned for AL Fresh Fuels Glovebox and in IMCL for higher radiation field materials	Thermogravimetric Analysis or TGA is a type of testing that is performed on samples to determine changes in weight in relation to change in temperature. Such analysis relies on a high degree of precision in three measurements: weight, temperature, and temperature change. As many weight loss curves look similar, the weight loss curve may require transformation before results may be interpreted. A derivative weight loss curve can be used to tell the point at which weight loss is most apparent. Again, interpretation is limited without further modifications and deconvolution of the overlapping peaks may be required.	



*Left: Scanning Thermal Diffusivity Microscope installed in hot cell for remote operation. Measurement of thermal diffusivity at 50 micron spatial resolution.*

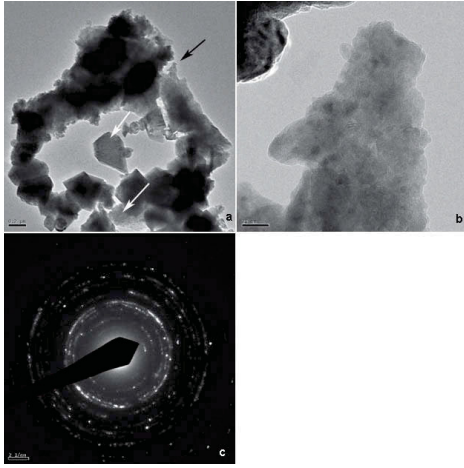
*Right: Radial profile of metallic alloy fuel specimens. Blue (Top) shows fresh fabricated fuel. Red (Bottom) shows irradiated fuel with a decrease in thermal diffusivity due to irradiation damage.*



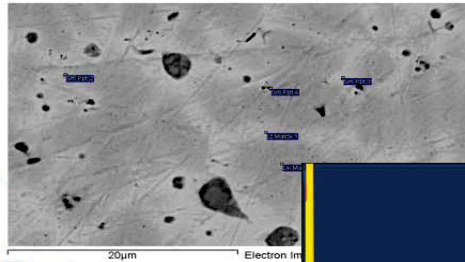
## Microstructure, Phase and Property Analysis Capabilities

Capability / Equipment	Application	Description
Scanning Transmission Electron Microscope (STEM) - EML	<p>Characterize small scale structural features: precipitates, dislocation loops</p> <p>Identify crystal structure information</p> <p>Determine composition by energy dispersive spectroscopy</p>	<p>Characterize nanometer-scale microstructure, phases and homogeneity of actinide fuel forms</p> <p>JEOL 2010, 200 kV, 2,000 X to 1,500,000 X, equipped with energy dispersive X-ray spectrometer</p>
<p>Scanning Electron Microscope (SEM) with energy dispersive (EDS) and wavelength dispersive X-ray spectrometers (WDS) and electron back scatter diffraction detector (EBSD);</p> <p>One SEM available in <b>CAES</b>, with others available at FASB and EML for Uranium and Transuranic materials</p>	<p>Determine composition by energy dispersive spectroscopy and wavelength dispersive spectroscopy</p> <p>Identify phases using crystal structure information by electron backscatter diffraction</p> <p>Study fuel cladding chemical interaction</p> <p>Capable of analyzing elements from Be through Cm with full matrix correction, including fission gases on samples</p>	<p>Characterize as-fabricated microstructure, phases and homogeneity of actinide fuel forms</p> <p>JEOL JSM 7000F FEG SEM, up to 30 kV and 600,000 X</p> <p>Zeiss DSM 960a SEM, up to 30 kV</p> <p>LEO 1455 VP SEM, up to 30 kV, operates at higher pressures</p> <p>JSM 6610 LV, 30KV and maximum 300,000X. It can be operate under vacuum or adjustable pressure from 10-270 Pa. It is equipped with EDS, EBSD and CL (Cathodoluminescence).</p>
<p>Microfocus X-ray Diffractometer (<math>\mu</math>-XRD) performs micro-scale phase identification, small-sample powder diffraction and texture determination</p> <p>XRD in FASB (non rad samples)</p> <p>(<math>\mu</math>-XRD in AL will be moved into a shielded cell in the IMCL for use on high activity / hazard materials.)</p> <p>Variable Temperature XRD – AL (high temperature)</p>	<p>Conventional X-Ray Diffractometers consist of an X-ray source, a sample stage, an X-ray detector and a goniometer on which the X-ray source and detector are mounted. When X-rays from the source strike crystals, which are ordered arrays of atoms, the atoms of the crystals scatter the X-rays. This scattering is a result of interactions between the incident X-rays and the electrons of the atoms making up the crystal. The scattered electromagnetic waves interfere with each other to produce “reflections” at discrete angles that are characteristic of the types of scattering atoms and their orderly arrangement in the crystal structure. Thus, the reflections can be used for identification of a material and its crystal structure. A microfocus X-Ray Diffractometer allows for the analysis of small samples or spatial analysis of small areas of large samples. Microfocus XRD analysis combines line scans with variable <math>2\theta</math> range for phase identification with area scans at fixed <math>2\theta</math> angles for spatial phase mapping. Parallel beam optics also allow for grazing incident measurements to collect data from only the top 10mm - 20mm from the sample surface.</p>	

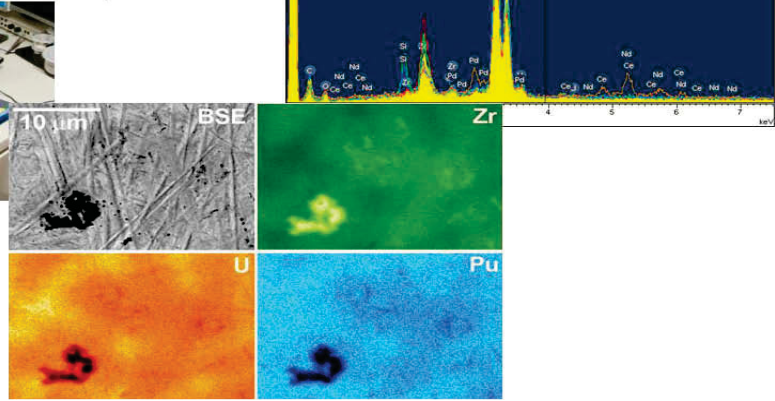




Scanning Transmission Electron Microscope (STEM)

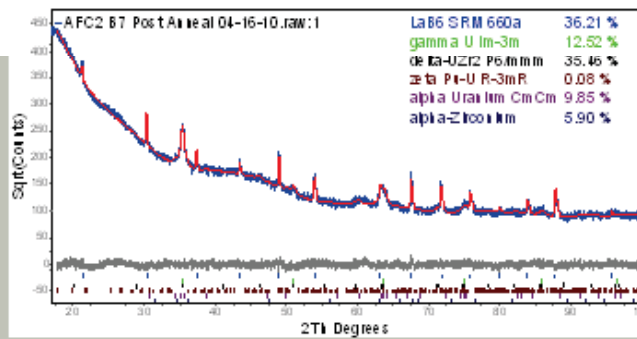


Back scattered electron and Energy dispersive X-ray spectra showing compositions at the indicated points



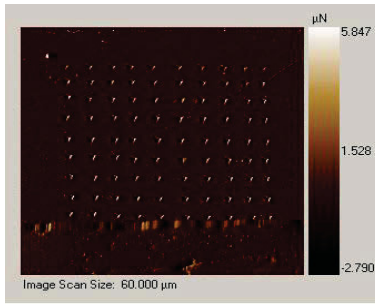
X-ray Diffractometer (XRD)

Alloy	61U-24Pu-15Zr (wt%) after annealing					
Phase	LaB <sub>6</sub> Std	$\delta$ UZr <sub>2</sub>	$\zeta$ - (Pu-U)	$\alpha$ -U	(bcc) $\gamma$ -U	$\alpha$ -Zr
w/ standard	36.2	35.5	0.1	9.8	12.5	5.9
w/o standard		55.6	0.1	15.4	19.6	9.2



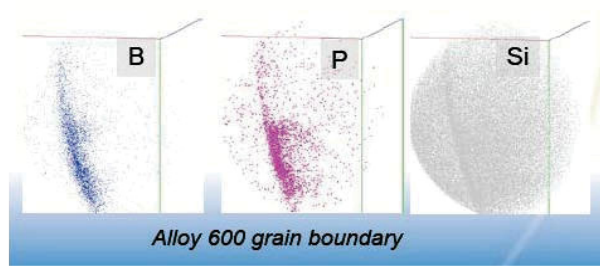
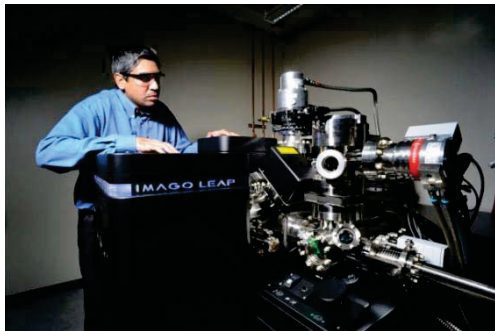
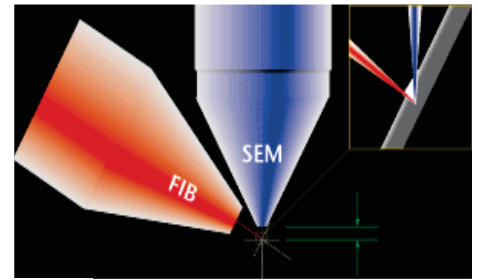
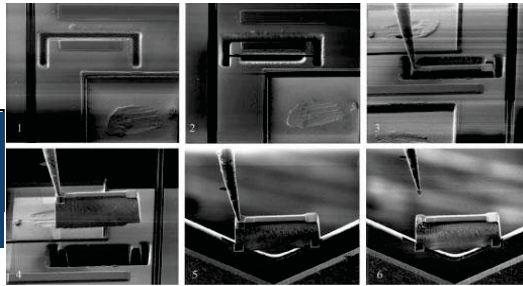
## Microstructure, Phase and Property Analysis Capabilities

Capability / Equipment	Application	Description
Nano Indentation - <b>CAES</b>	Yields relation of mechanical properties on a very small length scale	Hysitron Tribo Indenter (TI-950); 400 °C Stage, AFM Head, MFM capability, In-situ SPM imaging, Dual heads for high and low loads (10 mN – 5 N)
Dual Beam Focused Ion Beam (FIB)  EML for higher hazard materials, will be moved into a shielded cell in the IMCL for use on high activity / hazard materials. Also in <b>CAES</b> .	Field Emission Gun (electron and ion imaging) enables site specific microsectioning for use in TEM membrane preparation and material imaging	-FEI Quanta 3D -Imaging resolution: <3 nm 3D image and chemical reconstruction of submicron features Advanced analytical capabilities -CAES FIB also has Electron Backscatter Detector (EBSD) and Energy Dispersive Spectroscopy (EDS) -EML FIB also has EBSD, EDS, and Wave Dispersive Spectroscopy (WDS)
Field Emission Gun-Scanning Transmission Electron Microscope (FEG-STEM);  <b>CAES</b> , an additional FEG-STEM is planned for IMCL for use on high activity / hazard materials.	Provides better penetration of heavy element nuclear fuels and better resolution with ferritic/martensitic materials	Tecnai TF30 Field Effect Gun Super Twin (STwin) with Scanning Transmission Electron Microscope, 300kV
Local Electrode Atom Probe (LEAP);  <b>CAES</b>	Provides atom by atom chemical and isotopic data in 3D. Determining precipitates, grain boundary segregation, and dopant distributions.	Uses an electric field, and needle-shaped sample tip then using time-of-flight spectroscopy to determine the specific atom type. 3D reconstruction of up to hundreds of millions of atoms Resolution is atom by atom.  Imago LEAP 4000X HR,
Electron Probe Micro Analyzer (EPMA); AL – Elemental redistribution, coupled with FIB gives atomic scale elemental redistribution analysis  (Will be moved into a shielded cell in the IMCL for use on high activity/hazard materials)	Characterize compositional homogeneity of as-fabricated actinide-bearing transmutation fuels Analyze fuel constituent migration in irradiation fuels. Quantify radial distribution of fission products such as rare earths Characterize fracture surfaces of irradiated materials using electron imaging capability	Measure the localized micro-scale chemical composition of whole transverse cross-sections of irradiated fuels and materials and perform electron imaging of these samples.
Pushrod Dilatometer  FASB – for low-field beta/gamma materials  New dilatometers planned for AL Fresh Fuels Glovebox and for IMCL for high radiation/hazard materials	Measures volume changes caused by a physical or chemical process. A familiar application of a dilatometer is the mercury-in-glass thermometer, in which the change in volume of the liquid column is read from a graduated scale.	



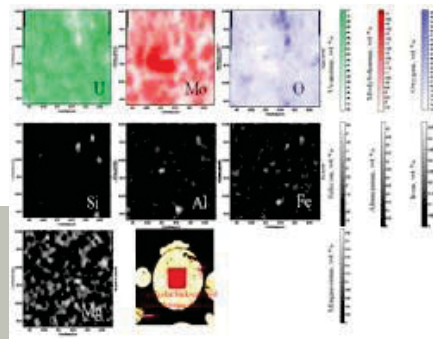
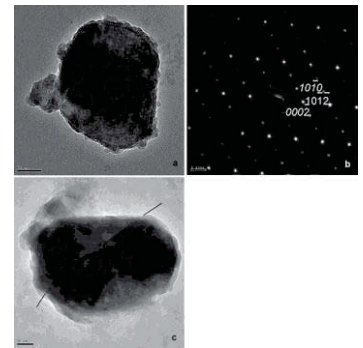
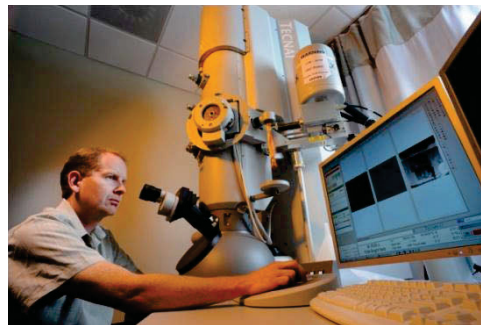
*Nano Indentation*

*Focused Ion Beam: two columns for simultaneous imaging and microscale milling and sectioning*



*Local Electrode Atom Probe*

*Field Emission Gun-Scanning Transmission Electron Microscope*



*EPMA analysis of fuel and material constituent redistribution produces critical data needed to understand and describe fuel and material behavior and performance.*



### 3. Future Capabilities [3, 5, 6, 7, 9, 10]

Idaho National Laboratory hosts a large variety of PIE capabilities and is upgrading and adding more. For example:

1. INL currently has plans to stand up an Irradiation Assisted Stress Corrosion Cracking Rig (IASCC) in FASB in FY2012 to provide valid crack growth data on unirradiated benchmark material specimens.
2. INL also plans to stand up a Hot Cell Electron Discharge Machine (EDM) for use in sample preparation. (FY2012)
3. INL is adding a sample preparation/analysis shielded hot cell to the EML facility in FY2012.
4. A mechanistic fuel failure examination system is planned for the HFEF hot cell
5. An upgrade to the HFEF main cell chiller
6. New TGA/TMA in the AL

Feedback from potential users was recently solicited via the U.S. National PIE Workshop to ensure any new capability is optimized for customer needs. The workshop helped identify many needs that are currently unfilled as well as capabilities that otherwise don't meet current needs due to capacity, configuration, etc. The top four needs identified in the workshop are as follows:

- (a) Instrumentation that supports analysis of materials at the nano-scale to support increasing knowledge of mechanisms that causes material degradation at the nano-scale. As such, an advanced PIE facility designed and constructed to support analysis on highly activated fuels and materials that incorporates strict environmental controls (vibration, electro-magnetic interference, temperature, etc.) and can also support environmental testing of materials (e.g., high temperature testing) with sample storage and preparation that is adjacent to other irradiation and PIE capabilities to support the advanced characterization tools is critical.
- (b) Coupling the advanced characterization capabilities with advanced modeling so that better and more predictive models can be developed, thus reducing the need for extensive empirical integral effects testing.
- (c) Accessibility and infrastructure that support sharing of data and creating administrative systems that facilitate collaborations with

entities that are subject to different constraints than national laboratories. This need includes the physical infrastructure to perform PIE and the non-physical needs of creating systems that are flexible to deal with entities (i.e., national laboratory, industry, university) and projects (e.g., experiment, project and reactor timeline vs. career scale) that have different time cycles and support the development, hiring, and retention of nuclear material R&D talent.

- (d) Development of in-situ techniques, analysis, and instrumentation that supports real-time data acquisition for deformation mechanics, damage development, and other time resolved measurements. In-situ characterization shows the development of material characteristics with time compared to traditional static PIE work.

INL is pursuing these four areas and is establishing the capability to perform advanced PIE by standing up unique, reconfigurable, accessible, modularized support facilities. To support the INL's customers, the CAES will house many of these new instruments that focus on nano-scale and atomic-level characterization, where examinations can be completed using micrograms or nanograms of irradiated specimens prepared at the MFC. As new capability is created by the scientific community, the CAES will be the entry point for bringing new analysis technologies to the INL.

The rapid evolution of analytical electron microscopes and the advent of high-performance computer interfaces with instruments poses an exciting future for performing PIE. A new laboratory operational model will promote and support continual implementation of state-of-the-art tools and technologies.

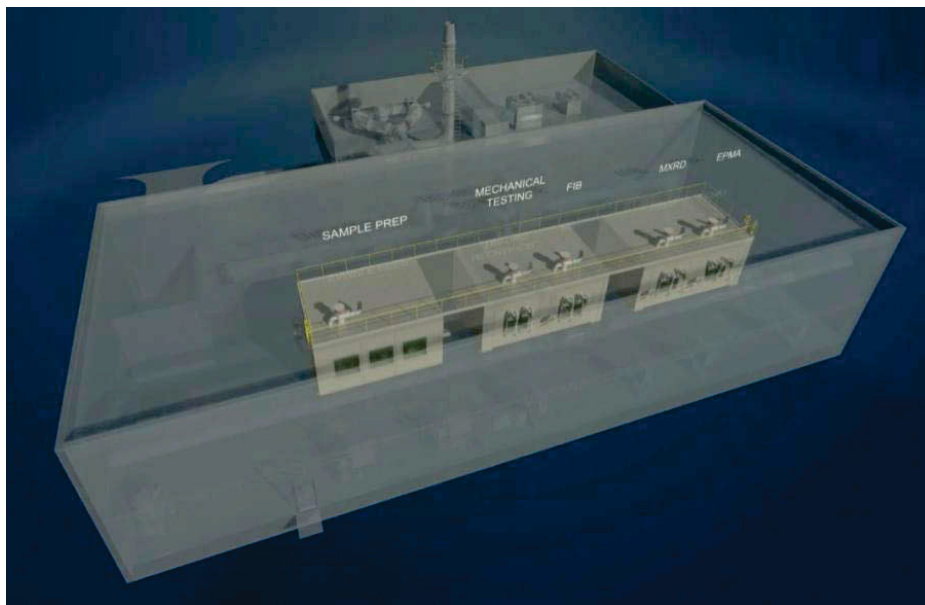
To meet the needs of the future, INL is standing up new capabilities that have the sophistication and refinement to house next generation PIE characterization equipment. The near term capability is the Irradiated Materials Characterization Laboratory (IMCL); the subsequent longer term facility will be an Advanced Post-Irradiation Examination capability.

### 3.1 Irradiated Materials Characterization Laboratory

The IMCL (see Fig. 3) will be the first facility of its type in the United States designed specifically for advanced instrumentation and equipment. The IMCL will contain space for installation of instruments and equipment within shielding structures that can be redesigned and refitted whenever necessary. The IMCL will also have mechanical systems that tightly control temperature, electrical and magnetic noise, and vibration to the standards required for advanced analytical equipment. Although some of the advanced characterization equipment is already in use in other industries, IMCL will be unique in the United States because the equipment will be housed in a nuclear facility and dedicated to the examination of irradiated fuels and materials.

Designed as a multipurpose facility suitable for many different missions over its projected 40-year life, the IMCL will have as its first mission the task of housing modern, state-of-the-art PIE instrumentation. The IMCL will be used to routinely perform micro- and nano-scale characterization of material specimens and irradiated fuel samples in the mass range of tens of grams down to micro-grams. The facility will also be designed to allow easy routine maintenance of the instruments. The initial suite of equipment planned for installation into IMCL includes:

- (a) Small Cask Receiving
- (b) Electron Probe Micro Analyzer (EPMA)
- (c) Focused Ion Beam (FIB)
- (d) Micro-X-ray Diffractometer (MXRD)
- (e) Field Effect Gun-Scanning Transmission Electron Microscope (FEG-STEM)
- (f) Mechanical Testing
- (g) Sample Preparation



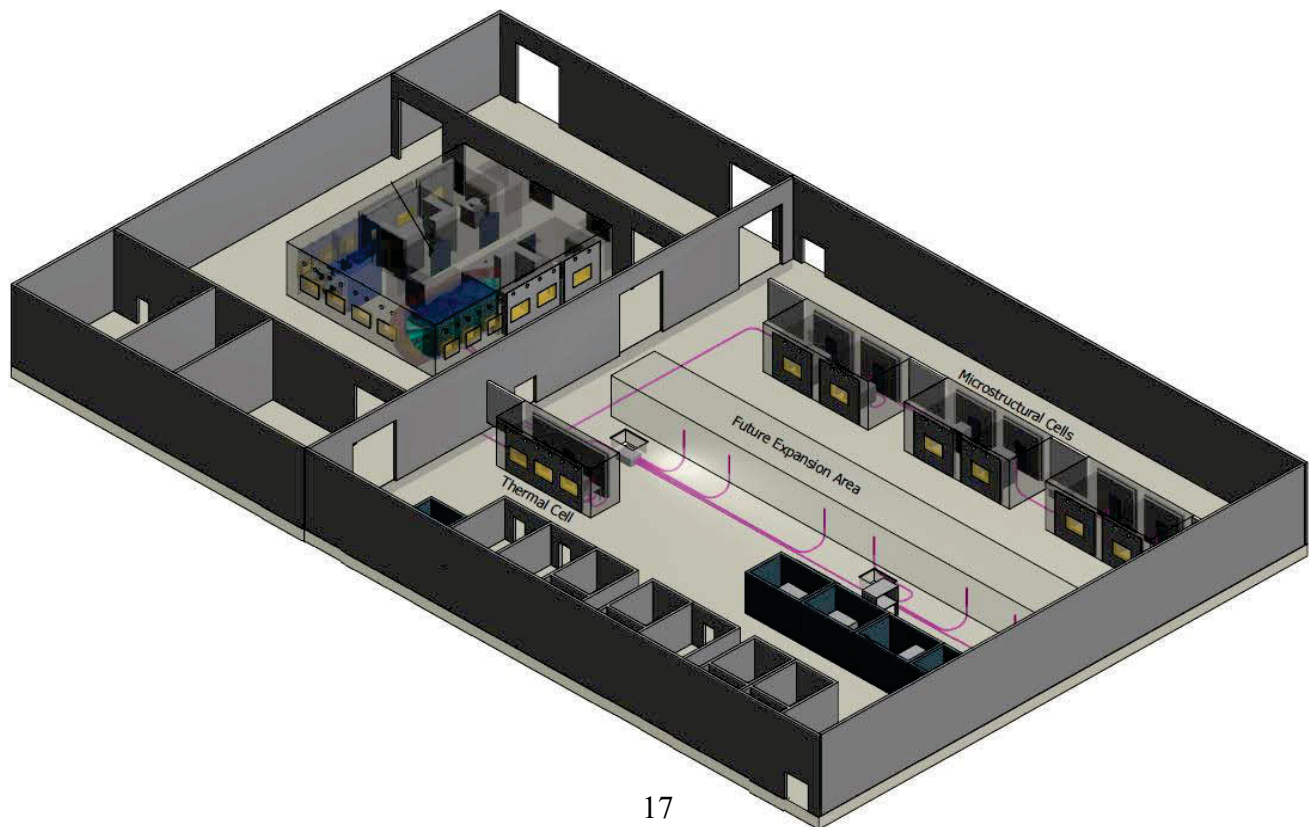


### 3.2 Advanced Post-Irradiation Examination Facility

Although the IMCL represents a significant advancement over current U.S. nuclear energy R&D capabilities, DOE has approved the mission to significantly expand the PIE and full-spectrum nuclear research capabilities of the United States through various alternatives, primary among them is a new multi-program facility called the Advanced Post-Irradiation Examination capability (see Fig. 2). This capability will handle considerably more sample and examination equipment than current facilities, both in types and quantities. Importantly, the shielded cell design will allow highly activated fuels and materials. As the project matures and the anticipated facility is built, some of the capability demonstrated in IMCL will transition to the new facility, freeing up IMCL to take on a key role of developing and deploying the next generation of “state-of-the-art” examination equipment. This would be consistent with the useful lifetime of such research equipment and would provide the newer facility with demonstrated state-of-the-art instrumentation.

The Advanced Post-Irradiation Examination facility alternative is anticipated to be a modular design to facilitate equipment specific shielding and flexibility for future equipment development, configuration alteration, and ease of replacement. The initial suite of equipment anticipated for installation includes:

- (a) Small Cask Receiving
- (b) Comprehensive sample preparation
- (c) Expanded Mechanical Testing
- (d) Dual Beam Focused Ion Beam (FIB)
- (e) High Temperature X-ray Diffractometer (HT-XRD)
- (f) Local Electrode Atom Probe (LEAP)
- (g) Secondary Ion Mass Spectrometer (SIMS), also Nano-SIMS
- (h) Full suite of thermal property characterization: Laser Flash Thermal Diffusimeter (LFD), STD, Differential Scanning Calorimeter (DSC), Thermogravimetry (TGA)
- (i) Knudsen Cell
- (j) Thermal Arrest Melting Point Determination
- (k) Auger Electron Microprobe (AEM)
- (l) Positron Annihilation Spectrometer (PAS)
- (m) Spherical Aberration (Cs) Field Emission Gun-Scanning Transmission Electron Microscope (Cs FEG-STEM)
- (n) Atomic Force Microscope (AFM)
- (o) Laser Resonant Ultrasound Spectrometer (LRUS)
- (p) Nuclear magnetic resonance using Raman and Fourier Transform Infrared Spectroscopy (FTIR)
- (q) Ankylography



#### 4. Partnerships/ATR-NSUF [8]

In addition to increasing physical capabilities through equipment and facilities, INL is expanding its partnerships and collaborations. Recently the ART-NSUF added ORNL and the University of California Berkeley as facility partners. Such partnerships increase access to national irradiation and testing capabilities and provide greater flexibility to respond to user needs.

The new partnerships will make ORNL's High Flux Isotope Reactor (HFIR) and its associated capabilities available to the ATR-NSUF users. The HFIR is a versatile, 85-megawatt isotope and test reactor that provides one of the highest steady-state neutron fluxes of any reactor in the world. Irradiation experiment facilities include a wide variety of test positions, a hydraulic shuttle, and the capability for multiple instrumented target positions. Target fabrication, hot

cell facilities for the examination of nuclear fuels and irradiated materials, the Radiochemical Engineering Development Center, and a set of special radiological laboratories at ORNL will also join the partnership. HFIR is operated by DOE, Basic Energy Sciences.

University of California Berkeley will bring several capabilities for examining irradiated material samples. Its facilities include a nano-indentation system for nano- and microscale hardness testing at ambient and elevated temperature and inert environments, positron annihilation spectroscopy, and warm sample preparation.

Access to the ATR-NSUF is accomplished through university-led proposals. Additional information on the proposal process can be found at: [atrnsuf.inl.gov](http://atrnsuf.inl.gov)

#### 5. Conclusions [3]

The INL incorporates all the necessary elements for successfully performing PIE and meeting the research needs to enable the advancement of nuclear energy. It allows parties interested in the advancement of scientific knowledge access to the powerful and versatile irradiation capabilities of the ATR and

provides them with the diverse equipment and methods needed to analyze their experiment after irradiation. The quality of the program will only improve with time as the INL adds equipment and facilities to what is already available.

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<[https://inlportal.inl.gov/portal/server.pt?open=514&objID=1555&mode=2&featurestory=DA\\_574357](https://inlportal.inl.gov/portal/server.pt?open=514&objID=1555&mode=2&featurestory=DA_574357)>.
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