Post-Irradiation Examination Guide

Idaho National Laboratory

U.S. DEPARTMENT OF ENERGY

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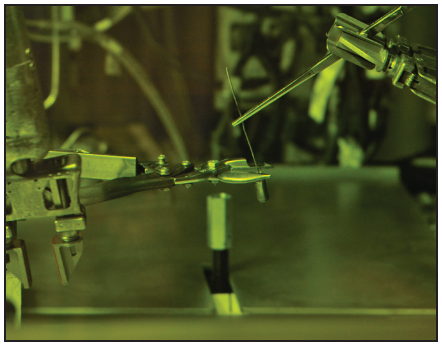
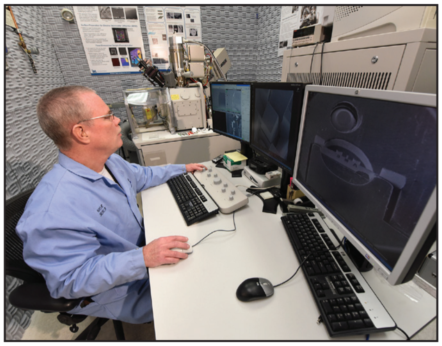
Table of Contents





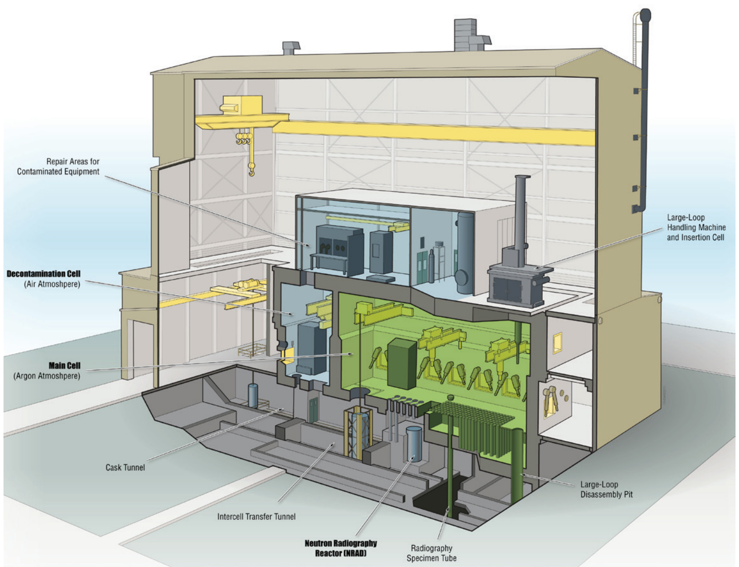
An operator using manipulators at the Hot Fuel Examination Facility.

Hot Fuel Examination Facility

Located at the INL Materials and Fuels Complex, the Hot Fuel Examination Facility (HFEF) is a large, heavily shielded, alpha-gamma hot cell facility designed for remote examination of highly irradiated fuel and structural materials. Its capabilities include non-destructive (dimensional measurements and neutron radiography) and destructive examination (such as mechanical testing or metallographic/ceramographic characterization). It can accept full-size light water reactor fuel assemblies (386 cm or 152 inches).

HFEF comprises two adjacent large, shielded hot cells in a three-story building, as well as a shielded metallographic loading box, an unshielded hot repair area and a waste characterization area. The main cell (argon atmosphere) has 15 workstations, each with a viewing window and a pair of remote manipulators. A decontamination cell (air atmosphere) has six similarly equipped workstations. The cells are equipped with overhead cranes and overhead electromechanical manipulators. Cell exhaust passes through two stages of HEPA filtration. The facility is linked to analytical laboratories and other facilities by pneumatic sample transfer lines.

Each main cell work station has removable electrical and lighting feed-throughs that can be changed to accommodate the mission of the station. The main cell is equipped with two rapid insertion ports for quick transfer of small tools and items into the cell. The decontamination cell contains a spray chamber for decontaminating equipment and non-fissile material using a manipulator-held wand. Material handling takes place via a 750-pound electromechanical manipulator, a 5-ton crane and six sets of master-slave manipulators. The hot repair area is available for contact maintenance on cell equipment; it can also be used for transfer of equipment and materials to or from the decontamination cell. HFEF also has a 250 kW Training Research Isotope General Atomics (TRIGA) reactor, for neutron radiography irradiation to examine internal features of fuel elements and assemblies.



Schematic of the Hot Fuels Examination Facility.

Transfer of radioactive materials from shipping casks to the hot cells generally takes place using the cask tunnel and cart. Shipping casks larger than 17 feet long by 56 inches in diameter or 30 tons use the Loop Insertion Cell and main cell roof penetration. Small casks weighing less than 5 tons can be transferred through the cart room and hot repair area into the decontamination cell, where they can be unloaded remotely.

The destructive and nondestructive examination capabilities listed below are available to NSUF users.

Visual Exam Machine — Eddy Current Tester

This machine provides a dedicated workstation for performing visual examination on fuel elements, capsules and other irradiated items. It comprises a standard in-cell examination stage and a modified Kollmorgan through-wall shielded periscope, designed for full-surface inspection and photo documentation of irradiated fuel elements or capsules. Its commercial photographic strobe lights are used exclusively for photography, while built-in halogen modeling lamps are used for both viewing and photography. The Kollmorgan periscope provides controls for aiming the objective (i.e., pointing the line-of-view), selecting among three magnifications, and focusing the image. The standard (spherical) optics of the periscope have been replaced with special planar optics that maintain the entire surface of a flat object (oriented normal to the optical axis of the system) in focus at the film plane.

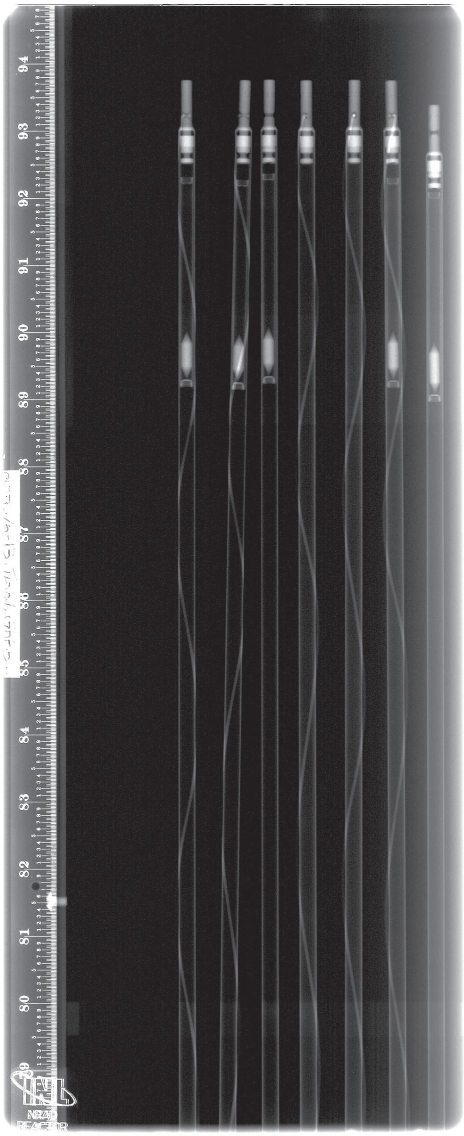
Neutron Radiography

The HFEF TRIGA reactor, known as NRAD, enables neutron-radiography irradiations to verify materials behaviors such as:

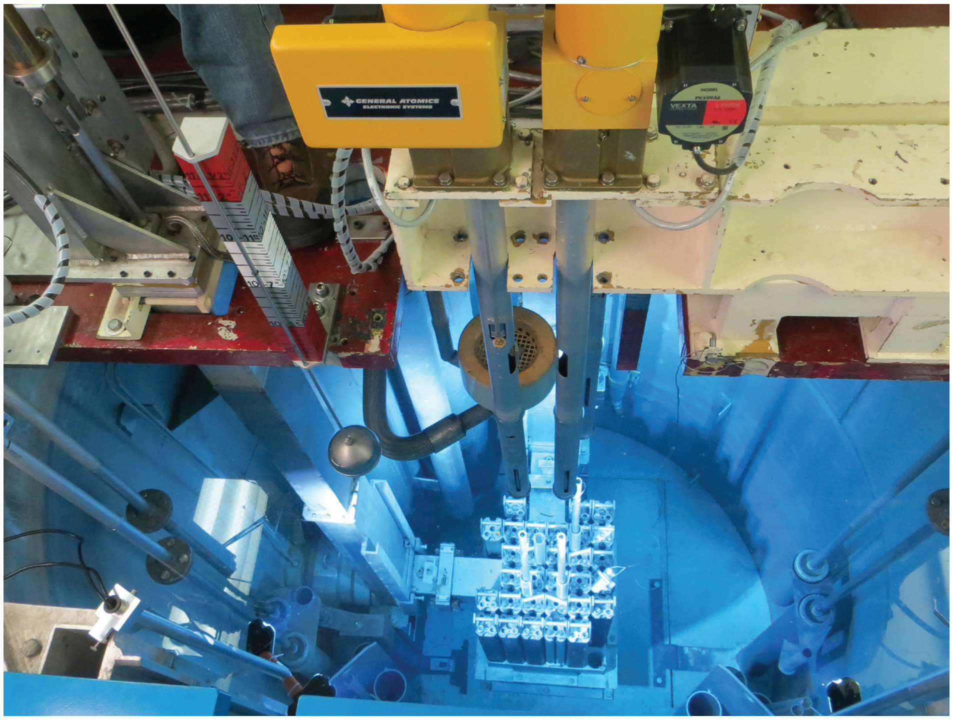
— Fuel pellet separations
— Fuel central-void formation
— Pellet cracking
— Evidence of fuel melting
— Material integrity under normal and extreme conditions

The NRAD beam tubes were designed for examination of fuel elements to identify areas of interest, such as location of fuel for disassembly, identification of cracking, density variations, and hydrides in cladding. NRAD is equipped with two beam tubes and two separate radiography stations. Neutron radiography of elements, capsules and loops is performed in the main cell at workstation 4M. Specimens are placed into a radiograph holder that is lowered into the NRAD neutron beam located below the floor. The holders optimally position the specimen for radiography without excessive neutron scattering.

Both dysporosium and cadmium-covered indium foils are used as neutron detector foils; these are irradiated in the neutron beam, then transferred to a film cassette and allowed to decay for three to four half-lives against ordinary X-ray film to form the image. The dysprosium foils, used for thermal neutron radiographs of low-enriched fuels and thin structural materials, produce excellent detail, but specimen thickness and fuel enrichment is limited. The indium foils are used for



A neutron radiograph of irradiated FFTF fuel.



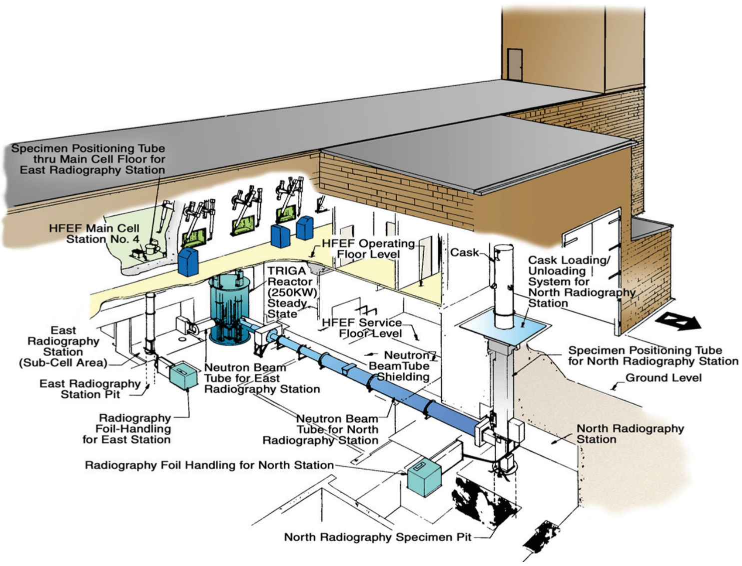
The Neutron Radiography (NRAD) Reactor.

epithermal neutron radiographs of highly enriched fuels and thicker structural materials. These do not show as much specimen detail, but they can be used for thicker specimens and more highly enriched fuel. In many cases, both foils are used to gain an outline of the fuel as well as its internal structure.

Item	Limit
Maximum specimen length	152 in.
Maximum specimen diameter	6.5 in. (round) or 4.5 X 8.5 in. (rectangular)
Specimen weight	600 lbs.

Table 1. System limits for neutron radiography.

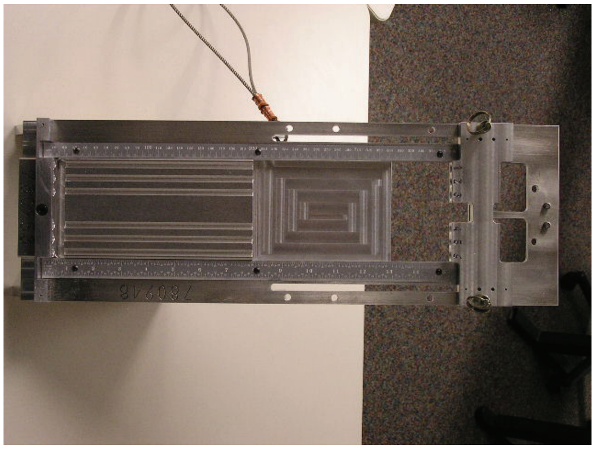
Hot Fuel Examination Facility



Schematic of neutron radiography system at HFEF.

Parameter	Value
Total vertical travel	139 in.
Fast vertical speed	Variable 18 to 30 in. minimum/ maximum
Slow vertical speed	Variable 0 to 6 in. minimum/ maximum
Vertical step travel	0.001 in./step
Lift force	3000 lb maximum
Positioning repeatability	+ 0.005 in.

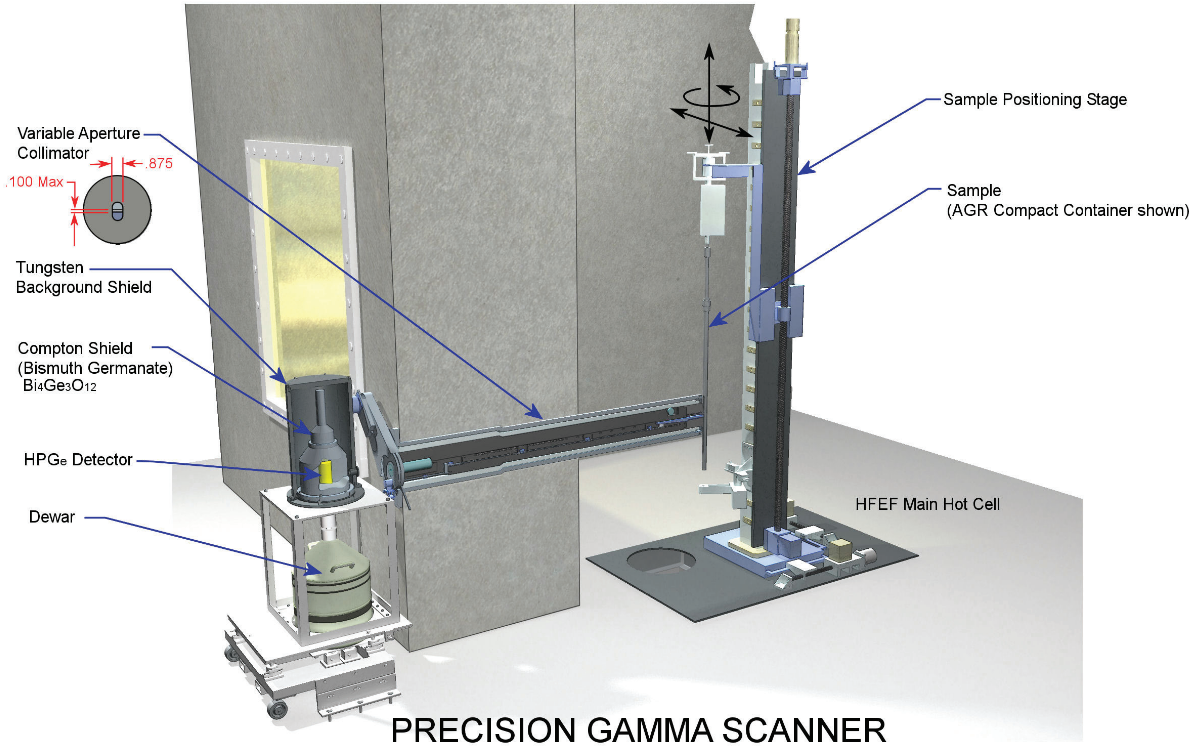
Table 2. Element handling equipment parameters and limits.



Example NRAD rodlet fixture.

Precision Gamma Scanner

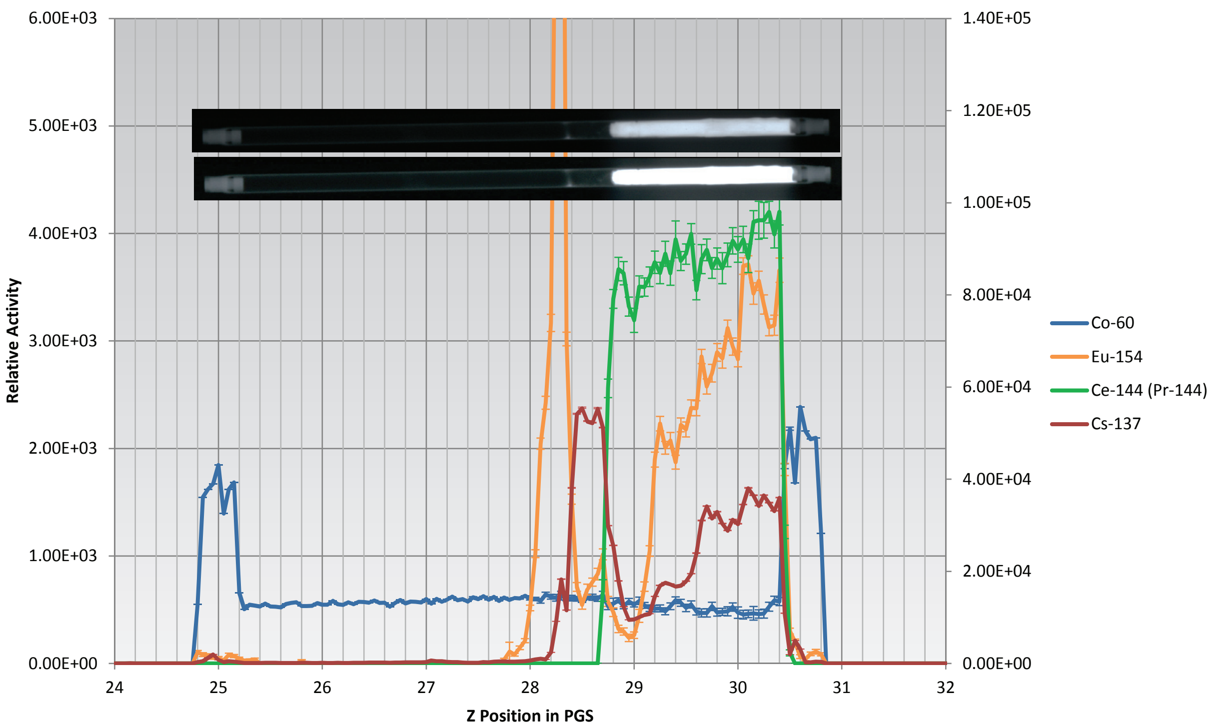
The PGS system consists of a collimator that penetrates the HFEF hot cell wall, a high purity germanium (HPGe) gamma spectrometry system with Compton suppression, and an in-cell stage used to position samples for examination in 4 degrees of freedom (x, y, z, rotation). The hot cell side of the collimator consists of tungsten alloy blocks that can be adjusted to create a slit height varying from 0.254 to 0.00254 cm (0.1 inches to 0.001 inches). The width of the collimator opening is fixed at 2.22 cm (0.875 inches) creating a rectangular viewing area for the PGS system. Gamma-rays entering the collimator travel through a thin aluminum window, pass through the initial tungsten collimator blocks, through a second tungsten alloy beam scraper, and then to the HPGe detector. The PGS collimator can be rotated from a horizontal slit orientation 90 degrees to a vertical slit orientation when needed for specific scanning procedures.



PRECISION GAMMA SCANNER

Parameter	Limit
Maximum weight	500 lb.
Slit dimensions	Rectangular 2.2225 cm x 0.0 to 0.254 cm
Maximum vertical travel	386 cm. (nominal)

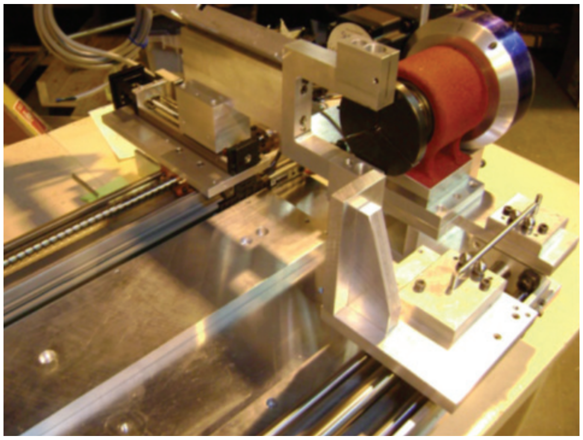
Table 3. Precision gamma scanner parameters and limits.

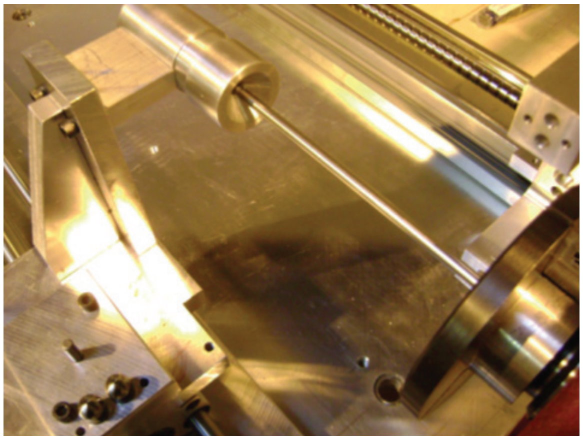


Precision gamma scan of the sample compared to neutron radiograph.

Dimensional Inspection

A modeular dimensional inspection bench is used to measure diameter/plate thickness, irradiation induced swelling, fuel rod growth, and cladding creep down in-reactor service and creep out during dry storage. Length measurement of each rodlet can also be performed with the Remote Fuel Metrology System. Length measurement uncertainty of the system is ±0.0025 cm (±0.001 in.). A calibration check with a certified reference length standard is performed at the beginning of a series of measurements. The length standard is traceable to a National Institute for Standards and Technology (NIST) standard.





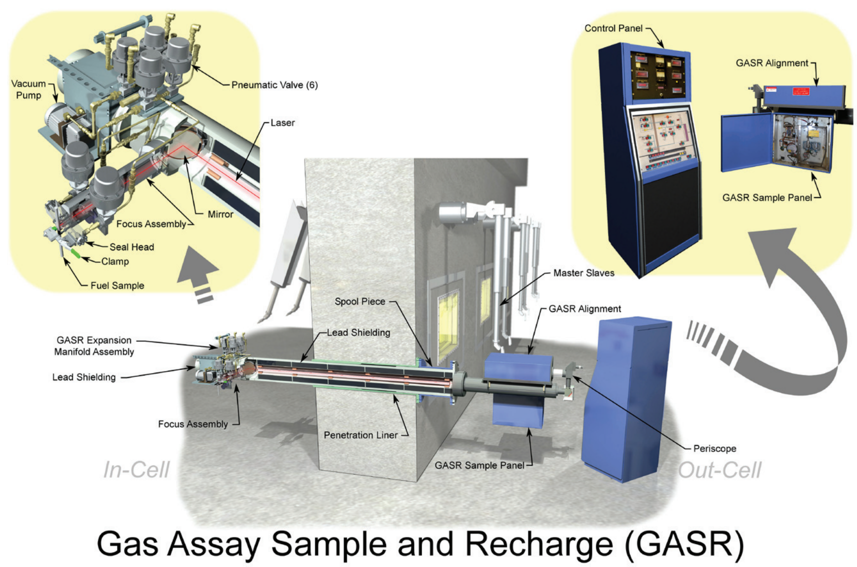
Remote fuel metrology unit for measure length and diameter.

The diameter profile measurement is also performed with the Remote Fuel Metrology System. The Remote Fuel Metrology System and Element Contact Profilometer (ECP) instruments measure the diameter profile by contact profilometry. The diameter measurement is made by two horizontally-opposed magnetic transducers connected to sapphire-tipped contact probes. Linear positions and displacements are derived from a digital-position readout system and are recorded on a data acquisition system. The diameter measurement uncertainty is ±0.0005 cm (±0.0002 in.). The diameter measurement accuracy is validated at the beginning of a series of measurements and at the beginning and ending of each shift of operation during an extended period of use. This calibration check is performed using two certified plug gauge reference standards that bound the lower and upper values of the measurements. The diameter standards are verified, traceable to a NIST standard.

Fission Gas Measurement and Analysis

Fission gas puncture and analysis is performed using the HFEF Gas Assay, Sample and Recharge (GASR) system and gas mass spectrometry. This equipment provides the ability to puncture cylindrical capsules or fuel elements in their plenum regions to measure free volume and pressure and gather a sample for gas composition and isotopic analyses.

The system comprises a 150-watt Nd-YAG pulsed laser, shielded optical and gas cell-wall feed-through, a mechanical pump, calibrated volumes, gauges and controls. Fuel elements or capsules are positioned on the laser by a clamp onto a neoprene gasket. The gasket provides a seal between the element and laser seal head. Void volume is determined by a series of backfills into the punctured fuel rod and expansions into the GASR system. The fuel rod internal gas pressure is derived from the void volume measurement and fission gas puncture pressure measurement. Fission gas composition is analyzed by a gas mass spectrometer to provide krypton (Kr) and xenon (Xe) isotopic and total elemental composition. The rodlet void volume and internal gas pressure uncertainty is ≤ ±5% in the range of 0.03 to 60 liter-atmospheres. The isotopic and elemental analysis uncertainty is ±1-5%, depending on the relative abundance of the analyte.



Schematic of GASR system.

Parameter	Limit
Element diameter range	0.174 to 0.832 in.
Element length range	1 to 152 in.
Cladding thickness	0.010 to .125 in.
Observed accuracy	> ± 5% for pressure and volume
Laser type	Nd-YAG
Element diameter range	0.174 to 0.832 in.
Maximum energy per pulse	20 Joules
Rated average power	150 W
Pulse repetition rate	1 to 150 pulses per second (pps)
Beam width	0.25 in.
Final lens focal length	4 in.
Minimum spot diameter	~0.005 in.
Pressure and Vacuum Instrumentation	
Sealing head pressure	0 to 200 ± 0.1 psia
Manifold pressure	0 to 50 ± 0.01 psia
Manifold vacuum	1 atm to 10 millitorr ± 5 millitorr
Sample line vacuum	1 atm to 10 millitorr ± 5 millitorr
Sample line pressure	0 to 50 ± 0.01 psia

Table 4. Gas assay sample and recharge system specifications.

Sample preparation

Irradiated fuel, cladding and structural materials are sectioned, mounted into metallographic bases, ground and polished in the containment box located in the HFEF main cell. The containment box has its own argon atmosphere and atmosphere control system to prevent cross contamination with the main cell. Irradiated samples prepared in the containment box are pneumatically transferred to the box, where they are examined by either the Leitz metallograph or a digital microhardness tester. The pneumatic transfer system also connects to the Analytical Laboratory.

Metallography

Optical microscopy is used for characterization of fuel grain size and morphology, porosity, phase, fuel-cladding interaction, cladding oxide thickness, and hydride distribution. HFEF houses a shielded metallography cell connected to the main cell via pneumatic transfer tube. The containment box operates under argon atmosphere to prevent rapid oxidation of sample surfaces. Metallographic images of irradiated specimens can be acquired using either a Leitz MM5 RT metallograph or LECO AMH43 microindentation hardness testing system. Digital cameras integrated the metallograph and the microhardness tester are used to capture optical microscopy data for further analysis.

Item	Limits
Magnification range of microscope	20X to 800X
Hardness tester magnification	400X
Minimum hardness tester weight	5 gm
Maximum hardness tester weight	400 gm

Table 5. Leitz MM5 RT metallograph limits.

Item	Limit
Element/Capsule Length	18 to 139 in.
Element/Capsule Diameter	1 in. max
Accuracy of Bow Measurements	0.020 in.
Accuracy of Length Measurements	0.010 in.

Table 6. Bow and length machine limits.

Microhardness testing

Microhardness testing is performed with a LECO AMH43 microindentation hardness testing system installed in a shielded inert atmosphere alpha containment hot cell. The microhardness tester is capable of applying loads from 10 grams to 1 kilogram. The sample stage can be position controlled to within ±1 µm. Image acquisition is through a high resolution CCD camera.

Bow and length machine

The element bow and length machine measures the distortion (bow) and actual length of irradiated cylindrical fuel elements and capsules. It can be used to determine fuel element or core component length and bow, as well as the direction of the plane of the bow.

Fuel pellet density measurements

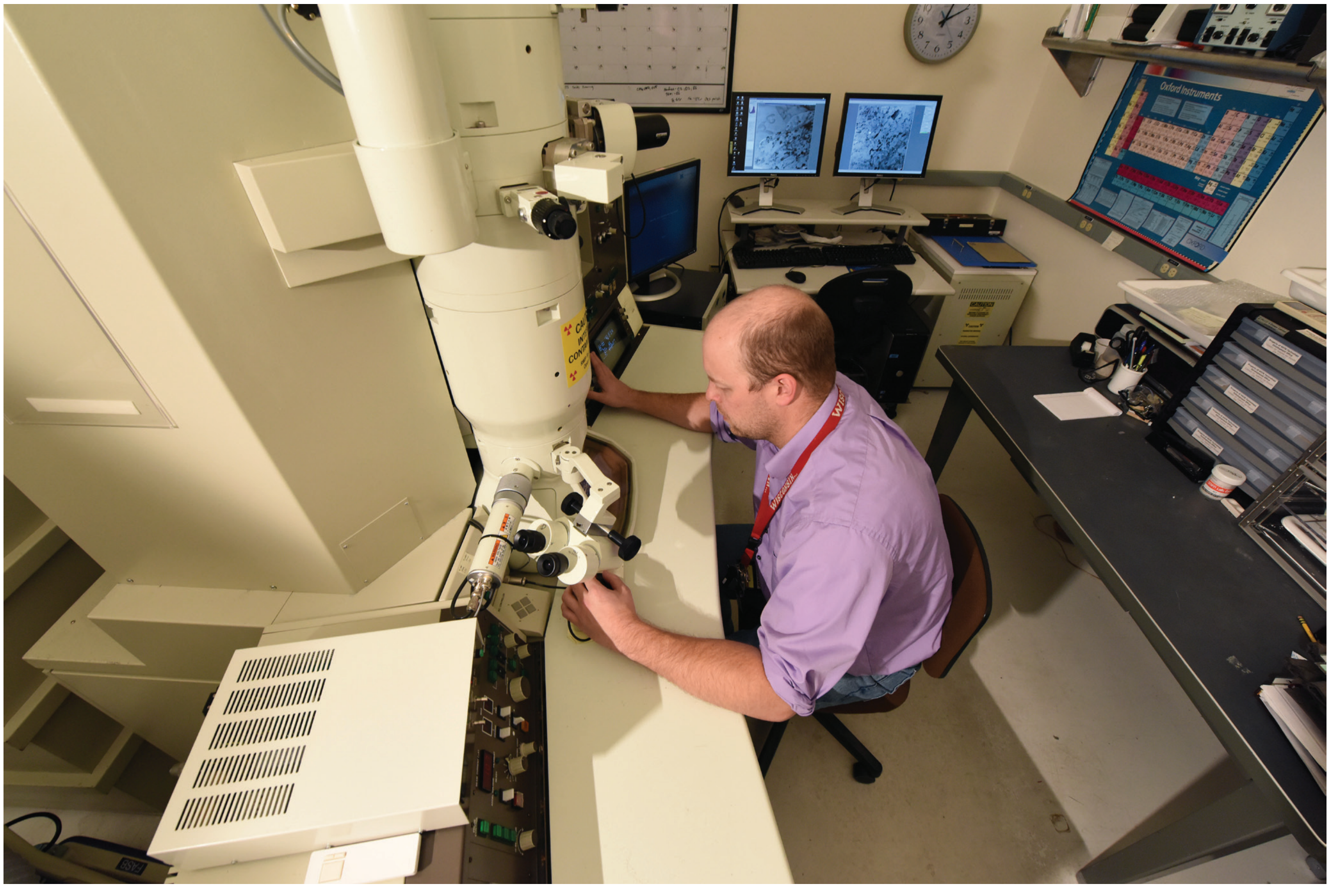
Measurements will be performed by immersion density or in the Analytical Lab. The immersion density apparatus includes an analytical balance equipped with a device to weigh a sample in air and submerged in a liquid, generally water. The pycnometer uses a known volume of gas (helium) to measure the displacement that occurs when the gas is expanded into a closed sample chamber containing the sample. Helium gas penetrates the finest pores, thereby assuring maximum accuracy. The volume of the sample is derived from the pressure changes in the system and the ideal gas law. The sample density is calculated by dividing its weight by its volume. The equipment is designed for remote operation using manipulators. The immersion density measurement uncertainty is less than ±2%. The pycnometry measurement accuracy is ±2% for sample volumes in the range of 0.2-1 cm3. The instruments are calibrated at the beginning of each day prior to use. The pycnometer and immersion density instrument are validated against a traceable density standard.

Tensile tests

Axial tensile tests are conducted on the Remote Operated Instron 5869 screw-driven load frame at the HFEF Main Cell Window 13M. This load frame is equipped with an 11,250 pound (50 KN) capacity primary load cell with option of attaching smaller capacity load cells to allow precision measurement while testing small samples. Crosshead displacement speed can be varied from 0.00004 in/min (0.001 mm/min) to 20 in/min (500 mm/min). With an available test space of approximately 47.4 inches (1205 mm) vertical by 37 inches (940 mm) horizontal a variety of testing setups can be accommodated. The high force capacity load cell combined with adaptable fixturing allows tensile testing of a variety of American Society for Testing and Materials (ASTM) standard (and nonstandard) specimens as well as other tests requiring controlled application of tensile or compressive force such as bend tests or flexure tests. Currently this load frame is not capable of direct strain measurements and crosshead displacement corrected by measured machine compliance is used to infer strain. An attached furnace capable of 1200 °C operation allows high-temperature testing.

Current plans include addition of a multizone furnace to allow more precise control of temperature for larger specimens and installation of an extensometer to allow direct strain measurement.

Hot Fuel Examination Facility



INL researcher operating transmission electron microscope.

Electron Microscopy Laboratory

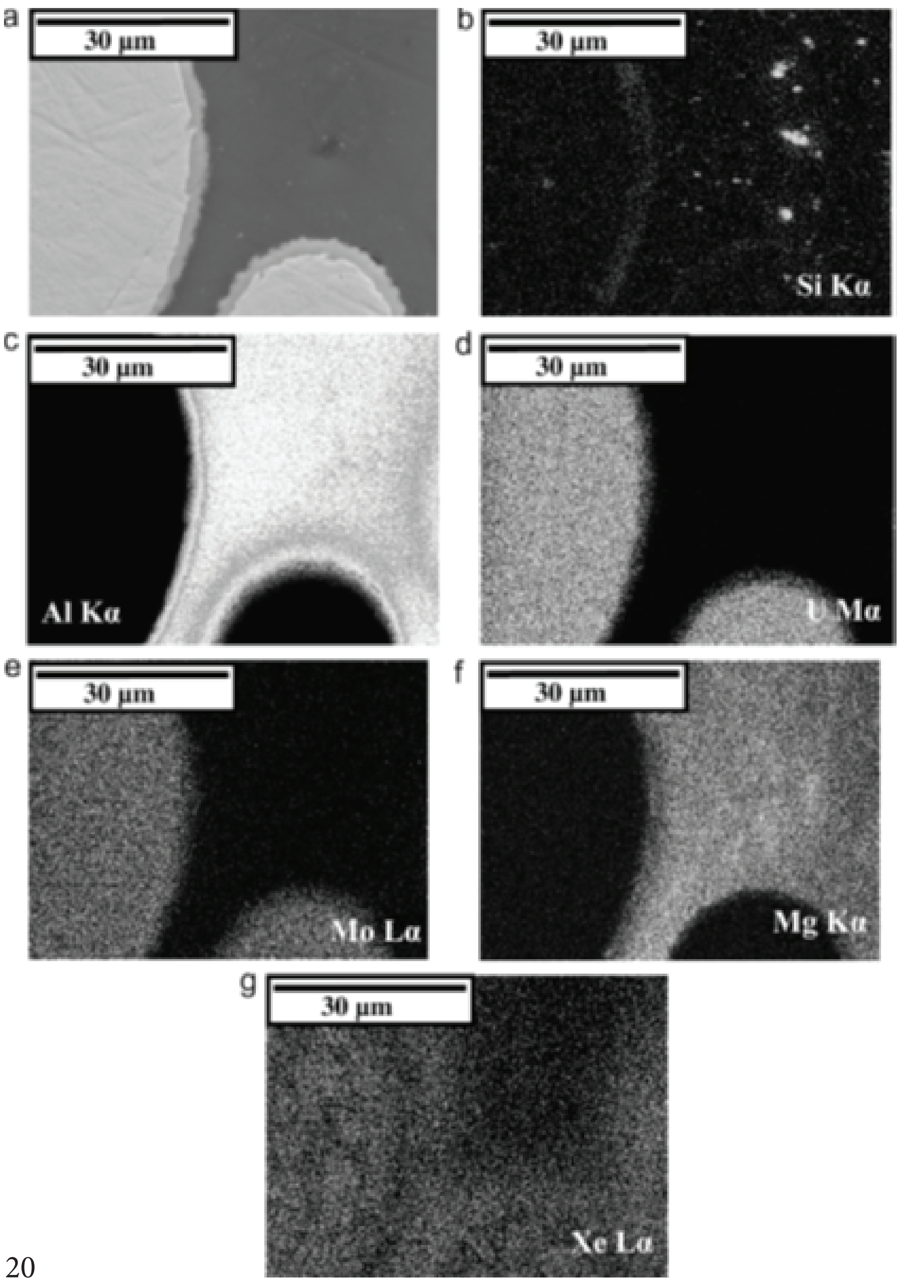
The Electron Microscopy Laboratory (EML) is a user facility dedicated to materials characterization using as its primary tools electron and optical microscopy. EML is a radiological materials area (RMA), permitting work to be performed with both radioactive and non-radioactive materials. A portion of the laboratory is dedicated to sample preparation, providing the researcher with facilities support, equipment, safety systems, and procedures to prepare samples of diverse materials for analysis. The examination capabilities listed below are available to NSUF users.

JEOL JSM-7000f scanning electron microscope

Schottky field emission gun; Secondary electron imaging; Backscattered electron imaging in compositional and topography imaging modes; Accelerating voltages from 50 volts to 30 kilovolts; Probe currents on the order of 10-12 to 2×10-7 A; Magnification ranges from 10X to 500,000X; Resolutions of SE images 1.2 nm at 15 kV and 3 nm at 1 kV; Resolution in analysis 3 nm at 15 kV, probe current of 5 nA and WD of 10 mm; Equipped with Oxford EDS and WDS; Specimen tilt ranges from -5 to 70 degrees; Available specimen holders include 12.5 (dia) × 10 (H) mm and 32 (dia) × 20 (H) mm.

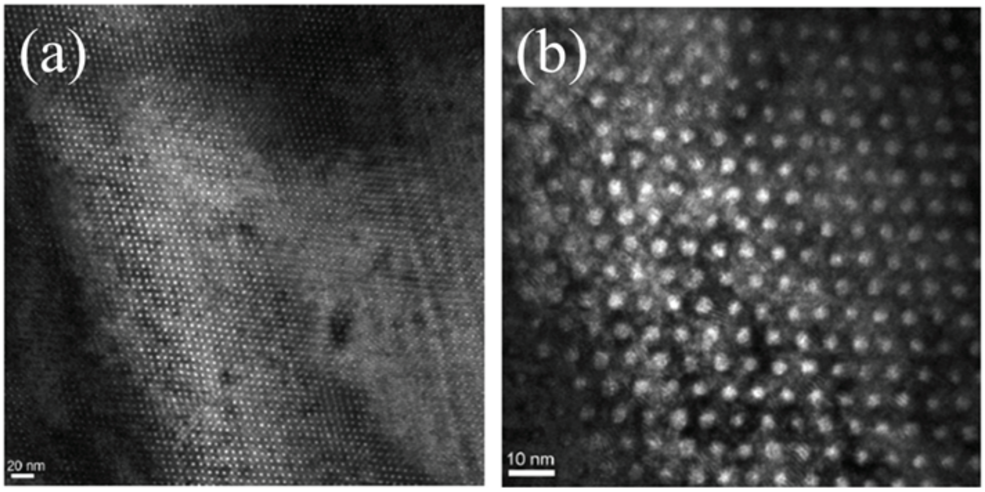
JEOL JEM 2010 scanning transmission electron microscope

Electron gun is a LaB6 filament; Accelerating voltages include 80 kV, 100 kV, 120 kV, 160 kV, and 200 kV; Magnifications range from 1,000X to 800,000X; Selective area diffraction camera length ranges from 15 to 300 cm; Specimen tilt angle ranges from -30 to 30 degrees; Loading capacity is one specimen at a time; Equipped with Bruker Quantax 200 Esprit 1.9 software and Bruker 133 silicon drift EDS detector; Capable of EDS mapping with simultaneous STEM imaging.



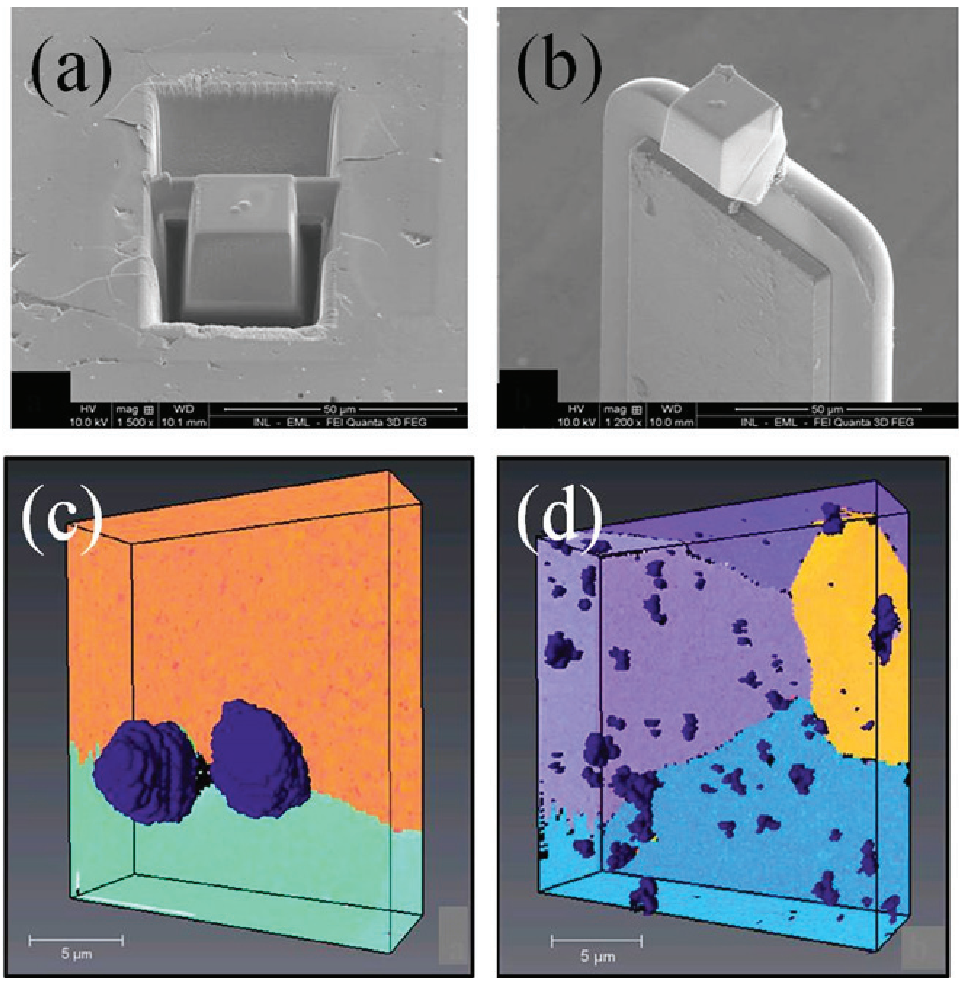
Secondary electron micrograph (a) and WDS X-ray maps for (b) Si, (c) Al, (d) U, (e) Mo, (f), Mg, and (g) Xe. Acquired from U-7Mo/6061 Al alloy matrix dispersion fuel irradiated to average burn-up of 40.8% (RERTR-6 experiment irradiated in ATR) [D. D. Keiser, A. B. Robinson, J.-F. Jue, P. Medvedev, D. M. Wachs, M. R. Finlay, J. Nucl. Mater. 393 (2009) 311-320}.

Bright-field transmission electron micrographs acquired from monolithic U-10wt%Mo fuel irradiated in ATR to the average fission density of 5.25×1021 fiss/cm3 during RERTR-7 irradiation campaign: (a) micrograph depicting long range ordering of the bubbles superlattice in U-10Mo fuel and (b) high resolution micrograph of bubble superlattice [B. Miller, J. Gan, J. Madden, J. F. Jue, A. Robinson, D. D. Keiser, J. Nucl. Mater. 424 (2012) 38-42].



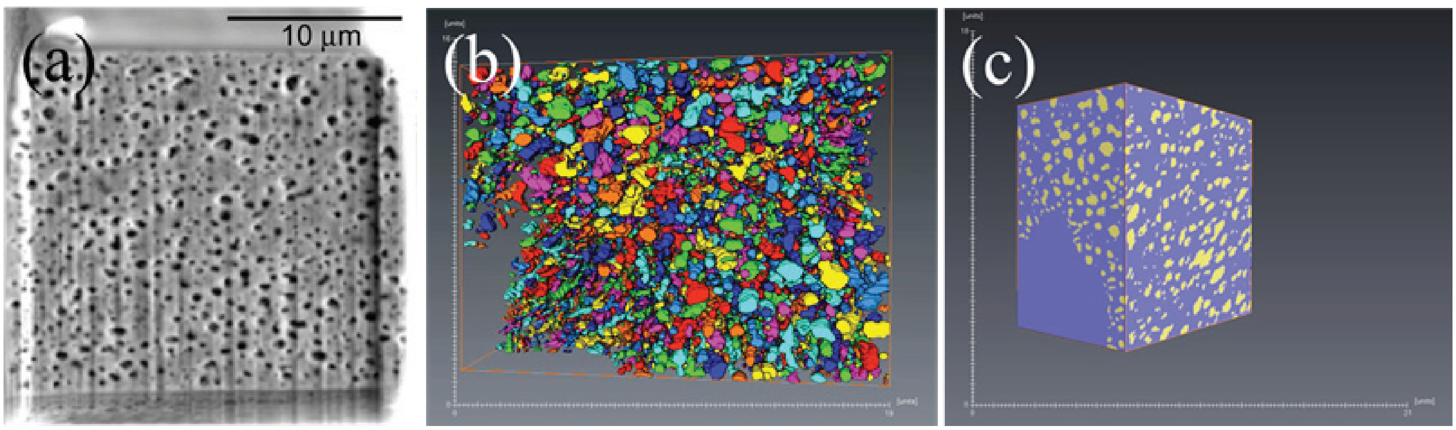
FEI QUANTA 3D FEG dual beam focused ion beam

High-resolution field emission SEM column optimized for high brightness and high current; High-current ion column with Ga liquid metal ion source; Electron beam accelerating voltages range from 200 V to 30 kV and has continuous probe current up to 200 nA; Ion beam accelerating voltages range from 2 kV to 30 kV; Ion probe current ranges from 1.5 pA to 65 nA in 15 steps; Ion beam resolution is 7 nm at 30 kV at beam coincident point; Equipped with Everhart-Thornley SED, Secondary electron and secondary ion detector (CDEM); Specimen tilt angles range from -10 to 60 degrees; Available gas injection systems are Pt and C; Equipped with EDAX EDS, WDS, EBSD, and Omniprobe micromanipulators; Analytical working distance is 10 mm (eucentric height); Available specimen holders include single stub mount, multi-stub holder, pre-tilted mounts, universal lift-out holders to hold TEM grids and a single stub facilitating in-situ lift-outs, custom met-mount holder, custom met-mount holders which hold TEM grids and APT/electron tomography holders; Set up to work with a number of different samples, ranging from irradiated metals and ceramics, fresh nuclear fuels, transuranic-bearing (Np, Pu, Am) samples to irradiated fuels.

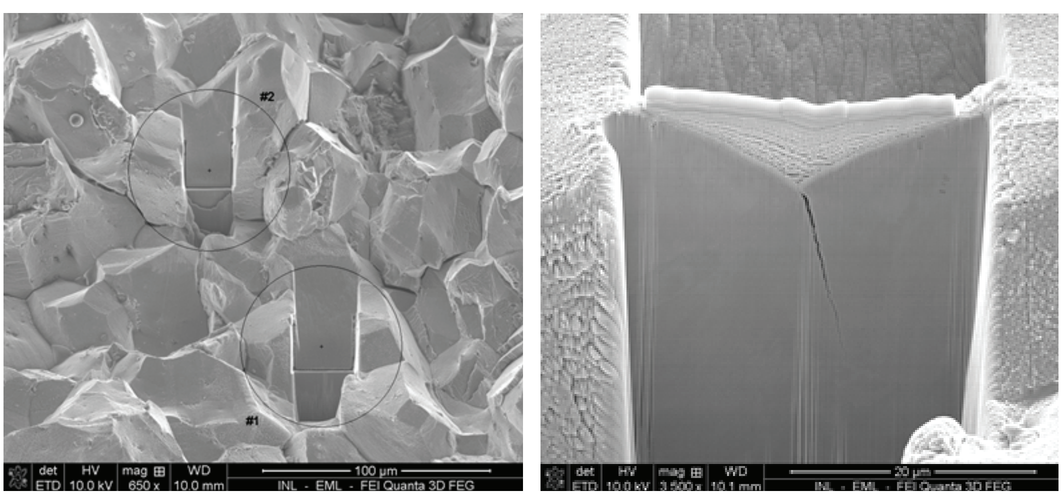


Secondary electron micrographs of a mixed oxide fuel (MOX) fuel pellet with HT-9 cladding following burn-up of 6.7% fissions per initial metal atom (FIMA) (a) prior to lift-out and (b) after lift-out and mounting on FIB grid. EBSD reconstruction of the distribution of metallic precipitates in two different cubes is shown in (c) and (d), respectively. Metallic (Mo-Pd-Ru-Rh-Tc) precipitates are shown in indigo and different grains of (U,Pu)O2 matrix are depicted by orange, royal blue, aquamarine, and violet colors to illustrate the difference in grain orientations. [M. Teague, B. Gorman, B. Miller, J. King, J. Nucl. Mater. 444 (2014) 475-480].

Electron Microscopy Laboratory



U-7Mo fuel irradiated in ATR as part of the RERTR-7A experiment to the fission density of 5.2×1021/cm3. (a) Secondary electron micrograph of the frontal view of the sample acquired near the end of the cross-sectioning process, (b) 3D reconstruction highlighting with different colors the regions where different pores may have begun to interconnect, and (c) 3D reconstruction of the cube highlighting the pores (shown in yellow) on the outer surfaces [D. Keiser, B. Miller, J.-F. Jue, J. Gan, A. Robinson, P. Medvedev, J. Madden, M. Teague, D. Wachs, Proc. Research Reactor Fuel Management Conference, March 30-April 3, 2014 in Ljubljana, Slovenia].



Scanning electron micrographs of the fracture surface observed in X-750 springs and cross-sectional view of the lamella before lift-out.

Multiple-coated, parfocal high-performance optical system with two parallel beam paths; available zoom 12.5:1; magnifications ranging from 8X to 100X; various stages, including rotatable polarization stage; field diameter ranges from 0.4 mm to 52.5 mm; resolution is 375 line-pairs/mm with 1.0X plano or planapochromatic objective and 600 line-pairs/mm with 1.6X planopochromatic objective.

Gatan Model 691 precision ion polishing systems (PIPS)

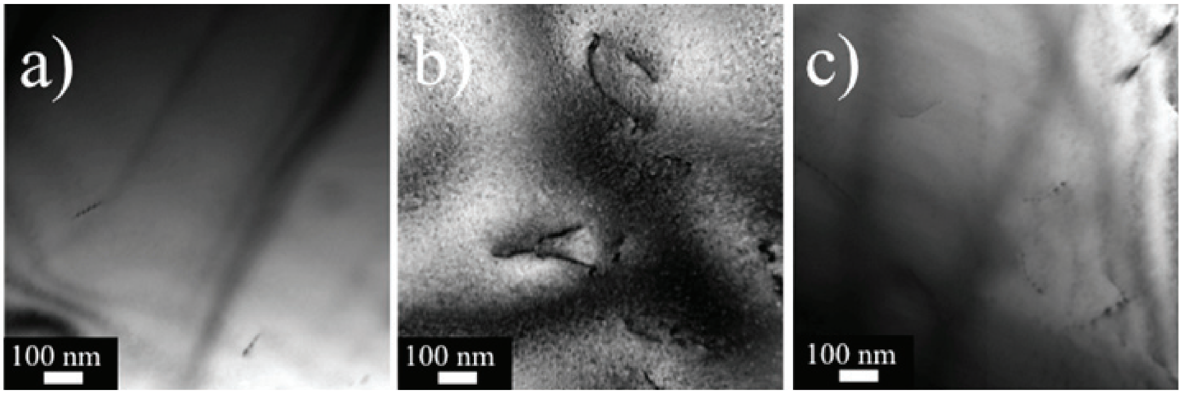
Two penning Ar ion guns with rare earth magnets; milling angles range from -10 to 10 degrees with each gun independently adjustable; ion beam energy ranges from 1 kV to 6 kV; ion current density is 100 mA/cm2 at the peak; accommodates two sample size 2.3 mm and 3 mm; LN2 specimen cooling; set up to produce conventional TEM specimens with large electron transparent areas.

Gatan Model 695 PIPS-2

Two penning Ar ion guns with low energy focusing electrodes; milling angles can be changed from -10 to 10 degrees for each gun independently; ion beam energies range from 100 eV to 8 kV; ion current density is 100 mA/cm2 at the peak; whisperlock with X, Y stage to center the region of interest for repolish; digital zoom microscope that operate in real time during milling; color image stage in digital micrograph; accommodates 2.3 mm and 3 mm conventional TEM disks, and FIB lamella; LN2 specimen cooling; set up as a postprocessing step for FIB lamella to remove FIB Ga ion beam damage.

Gatan Model 682 precision etching and coating system

Three penning ion guns with miniature rare earth magnets; ion beam energy ranges from 1 kV to 10 kV; ion current density for etch gun is up to 3 mA/cm2 and for coating gun is up to 10 mA/cm2 each; Etched area ranges from 7 to 10 mm depending on gun energy; coating area is uniform over a 1 inch diameter; standard coating materials are C, Cr, Pt, Pd, Au; accepts 1.25" metallographic mounts and most SEM stubs.



Bright field TEM micrographs of unirradiated Fe-12Cr: a) after jet-polishing, b) after preparation in FIB, and c) after preparation in FIB and post-processing in PIPS-2 [A. Aitkaliyeva, J. W. Madden, B. D. Miller, J. I. Cole, J. Gan, J. Nucl. Mater. 459,241 (2015)].

Specimen preparation equipment

— SouthBay Model 550D single vertical jet electro-polisher
— Buehler Isomet low speed saws
— Buehler EcoMet 3 variable speed grinder-polisher
— Allied MetPrep 3 Grinder/Polisher
— Struers Accutom-50 automatic precision cut-off and grinding machine
— Gatan Model 656 dimple grinders
— Denton Desk V coating system

The shielded sample preparation area system (SSPA) at IMCL.

Irradiated Materials Characterization Laboratory

The Irradiated Materials Characterization Laboratory (IMCL), located at INL's Materials and Fuels Complex, was designed and built to house state-of-the-art nuclear fuels and materials characterization equipment in a shielded environment. The mission of the IMCL is to examine highly radioactive materials at the nanometer and atomic scale, a scale not readily attainable on these types of materials with existing instrumentation. The examination capabilities listed below are available to NSUF users.

FEI QUANTA 3D FEG dual beam focused ion beam

High-resolution field emission SEM column optimized for high brightness and high current; High-current ion column with Ga liquid metal ion source; Electron beam accelerating voltages range from 200 V to 30 kV and continuous probe current up to 200 nA; Ion beam accelerating voltages range from 2 kV to 30 kV; Ion probe current ranges from 1.5 pA to 65 nA in 15 steps; Ion beam resolution is 7 nm at 30 kV at beam coincident point; Equipped with Everhart-Thornley SED, Secondary electron and secondary ion detector (CDEM), and retractable BSE detector; Specimen tilt angles range from -10 to 60 degrees; Available gas injection system is Pt; Equipped with Omniprobe micromanipulators; Analytical working distance is 10 mm (eucentric height); Available specimen holders include single stub mount, multistub holder, pretilted mounts, universal lift-out holders to hold TEM grids and a single stub facilitating in-situ lift-outs, custom met-mount holder, custom met-mount holders which hold TEM grids and APT/electron tomography holders; Set up to work with a number of different samples, ranging from irradiated metals and ceramics, fresh nuclear fuels to irradiated fuels; Equipped with IEE etching gases and has organic etch capabilities.

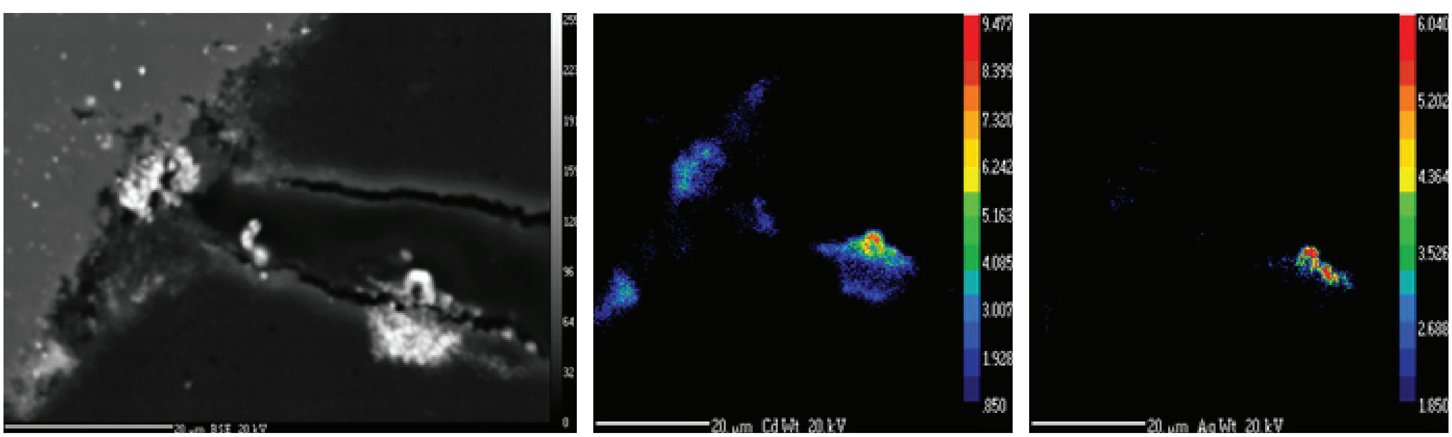
FEI Titan Themis 200 scanning transmission electron microscope

High brightness Schottky field emitter gun (X-FEG); Flexible high tension range, can be adjusted to 80, 120 and 200 kV; Electron gun monochromator for high energy resolution EELS and improved spatial resolution; Specimen tilt ranges from -40 to 40 degrees for analytical double tilt holder and from -75 to 75 degrees for tomography holder; Point resolution of 240 pm in TEM and 160 pm in STEM modes with energy spread of 0.8 eV at 200 kV without image or probe corrections; Equipped with HAADF detector and on-axis triple DF1/DF2/BF detectors; Equipped with super-X a high-sensitivity, windowless EDX detector system based on SSD technology; 0.7 srad solid angle and 120 mm2 combined

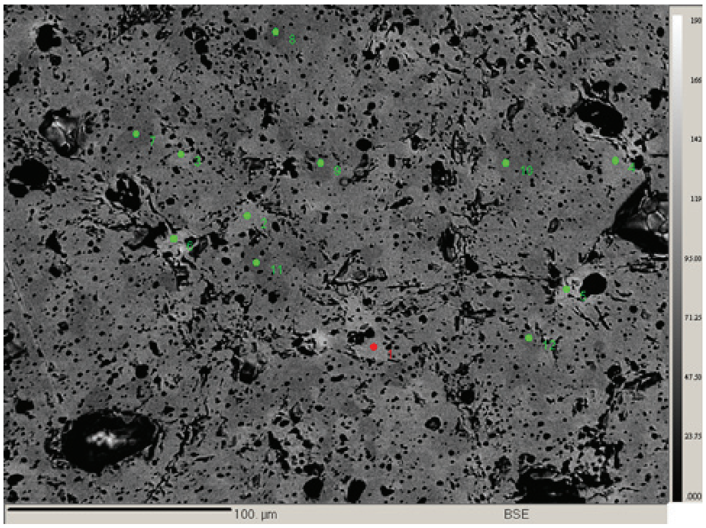
EDS detector area; Fast mapping with pixel dwell times down to 10 µs; Dedicated holders include FEI single tilt holders, double tilt holders and tomography holders.

Cameca SD100-R electron probe microanalyzer (EPMA)

High-intensity tungsten source; Accelerating voltages range from 5 to 50 kV; Regulated beam current up to 250 nA; Equipped with WDS capable of measuring elements from B to Cm; Equipped with SE and BSE detectors; Shielded EPMA capable of protecting electronics and detectors from fields up to 1.1×1011 Bq (3 Ci of 137Cs); Probe for EPMA software allows resolution of complex peak overlaps; Quantitative spot and X-ray map measurements. The shielded EPMA capability to analyze transverse cross-section specimens will characterize the migration and redistribution of fuel constituents and fission products, fission product radial profiles for burnup, plutonium and other actinide agglomeration, and fuel-cladding chemical interaction. Elements to be analyzed may include U, Pu, Np, Am, Cm, Zr, Xe, Cs, Mo, Tc, Ru, Rh, Pd, La, Ce, Pr, Nd, and others as requested by the PI.



Left, backscattered electron image of metal precipitates in an irradiated TRISO particle. Center and right, interference-corrected quantitative X-ray maps of the region shown on the left.



spot	U wt%	Pu wt%	Am wt%	Total
1	65.1	15.7	4.03	84.9
2	66.1	16.8	2.95	85.8
3	65.4	18.0	2.28	85.7
4	65.6	17.3	2.73	85.6
5	43.7	43.9	<0.5	87.7
6	50.4	37.3	<0.5	87.7
7	69.0	18.8	<0.5	87.8
8	70.9	15.4	<0.5	86.3
9	69.7	18.1	<0.5	87.8
10	69.7	19.8	<0.5	89.5
11	70.3	16.9	<0.5	87.2
12	77.9	9.24	<0.5	87.2

Left, backscattered electron image of unirradiated MOX. Right, quantitative analysis of metals at locations shown on the left.

JEOL Neoscope JCM-5000 benchtop scanning electron microscope

Small cartridge electron source; Accelerating voltages can be adjusted to 5, 10, and 15 kV; Magnifications range from 10X to 40,000X; Equipped with secondary and backscattered electron detectors; Maximum loadable specimen size is 70 mm in diameter and 50 mm in height; Has low vacuum and high vacuum modes; Specimen stage allows 35 mm travel in X and Y.

Olympus BX 41 optical microscope

UIS2 optical system; Built-in Kohler illumination for transmitted light; Magnifications ranging from 1.5X to 100X; Ceramic-coated coaxial stage with rotating and torque mechanisms.

Meiji EMZ-5 optical microscope

Multi-coated optical components; Available zoom ratio is 6.5:1; Magnifications range from 0.7X to 45X; Field of view is 32mm-5.1mm; Extended magnification range is 2.1X-270X; Rugged all metal body.

A researcher operating electron probe microanalyzer (EPMA) at IMCL.

Leco LM247AT microhardness tester

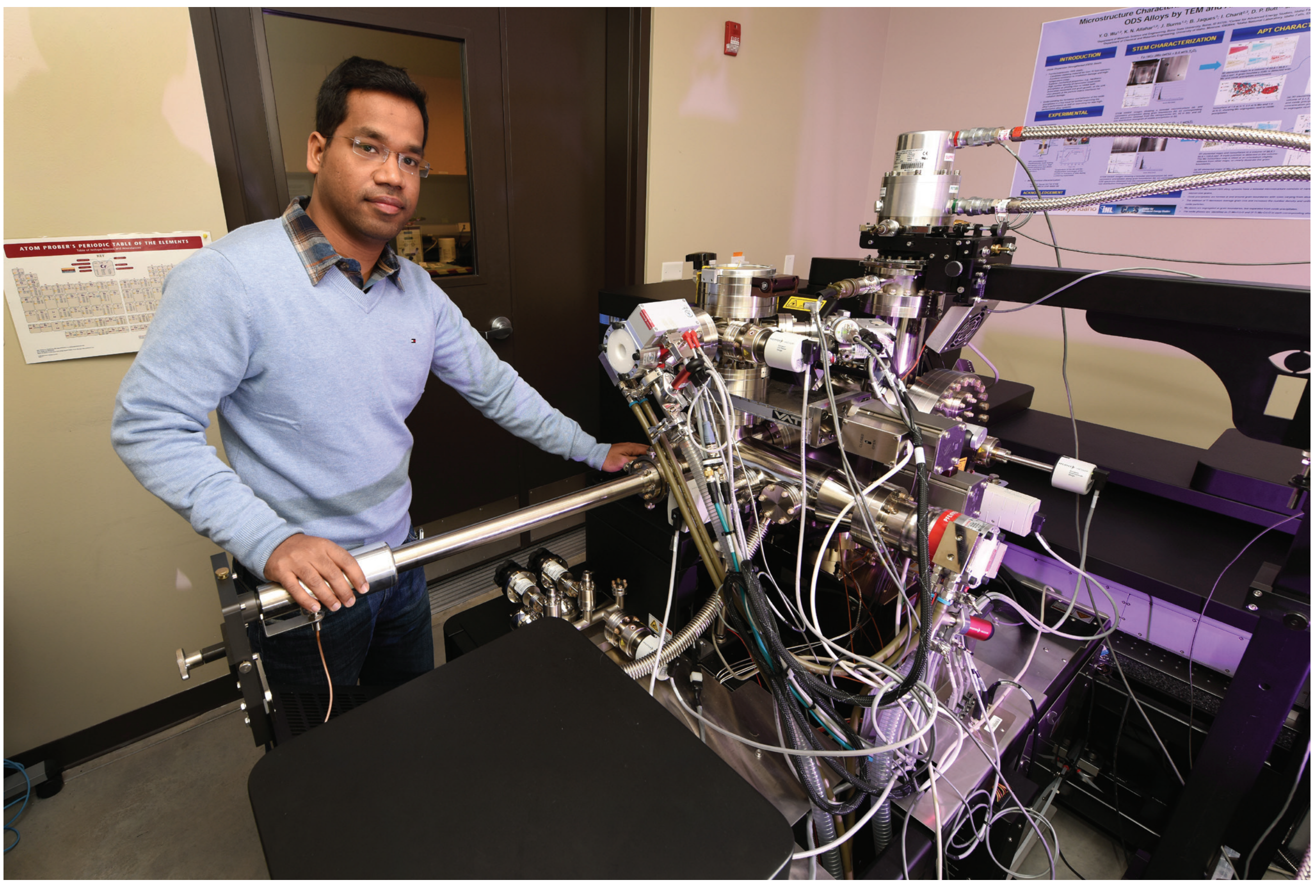
Dual indenter turret that provides Knoop and Vickers on a single tester; Accurate and repeatable load cell Rockwell-type tester; Load ranges from 1 to 2000 gf; Fixed aperture diaphragm, a centerable light source, and available interchangeable filters provide metallograph quality illumination; Automatic turret accommodates up to 4 objectives (2.5X to 100X).

Shielded sample preparation area (SSPA)

The SSPA is a two-bay shielded glove box/hot cell. Its primary purpose is sample preparation of highly radioactive fuels and materials. Sample preparation includes grinding, polishing, sectioning, cleaning, and etching of materials. Digital optical microscope is present allowing for characterization of samples prior to transfer to the electron microscopes throughout IMCL.

Specimen preparation equipment

- Buehler Isomet low speed saws
- Buehler MiniMet 1000 grinder-polishers



An INL researcher loading the Local Electrode Atom Probe (LEAP) instrument.

Microscopy and Characterization Suite

The Microscopy and Characterization Suite (MaCS) is a state-of-the-art materials characterization laboratory that provides crosscutting capabilities. The current rad operating envelope includes depleted uranium, solid activated ceramics, and fixed radiation sources. Depleted uranium may be in solid forms. Fixed radiation sources include activated metals: solid, nondispersible, smearably clean (i.e., considered to be free of removable radioactive contamination) samples of activated metal and solid ceramic materials that are prepared to minimize sample mass are generally acceptable for analysis and testing in CAES. The examination capabilities listed below are available to NSUF users.

JEOL JSM-6610LV scanning electron microscope

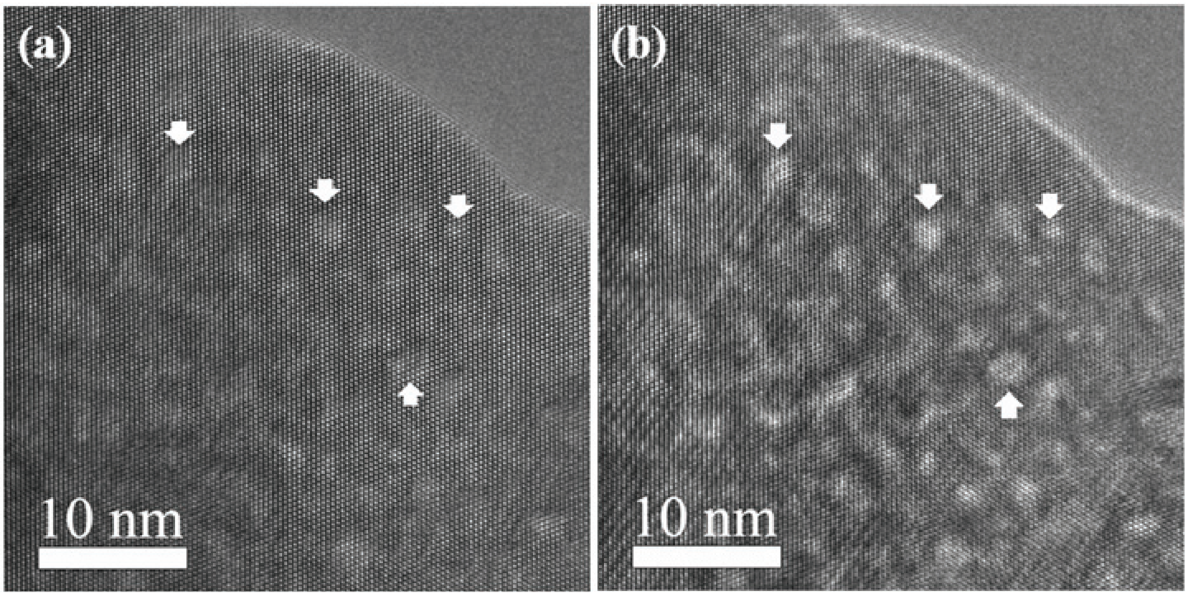
High-resolution imaging with tungsten source; Multielement solid-state BSE detector; SE detector; Accelerating voltages from 300 V to 30 kV; Magnifications from 5x to 300,000X; Resolutions 3 nm at 30 kV and 8 nm at 3 kV; Equipped with EDS, EBSD, and CL; Specimen tilt up to 90 degrees; Maximum loadable specimen size is 300 mm in diameter and 80 mm in height; Embedded color CCD camera for sample navigation.

FEI QUANTA 3D FEG dual beam focused ion beam

High-resolution field emission SEM column optimized for high brightness and high current; High-current ion column with Ga liquid metal ion source; Electron beam accelerating voltages range from 200 V to 30 kV and continuous probe current up to 200 nA; Ion beam accelerating voltages range from 2 kV to 30 kV; Ion probe current ranges from 1.5 pA to 65 nA in 15 steps; Ion beam resolution is 7 nm at 30 kV at beam coincident point; Equipped with Everhart-Thornley SED, Secondary electron, secondary ion detector (CDEM), and STEM detector; Specimen tilt angles range from -10 to 60 degrees; Available gas injection systems are Pt and C; Equipped with EDAX EDS and EBSD, and Omniprobe micromanipulators; Analytical working distance is 10 mm (eucentric height); Available specimen holders include single stub mount, multi-stub holder, pre-tilted mounts, and universal lift-out holders to hold TEM grids and a single stub facilitating in-situ lift-outs.

FEI Technai TF-30-FEG STwin scanning transmission electron microscope

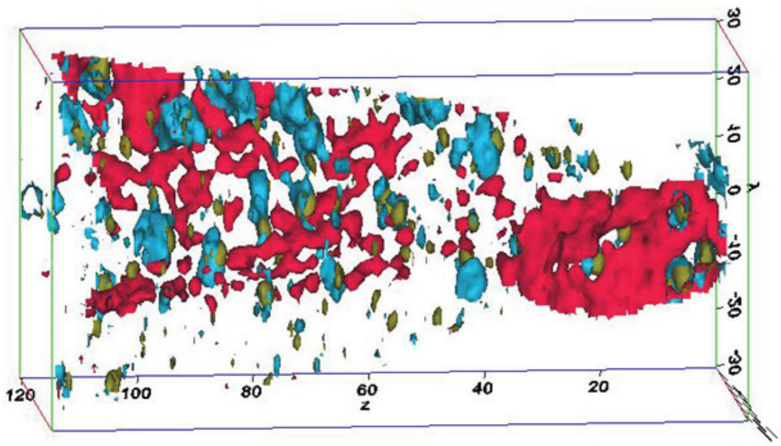
Schottky field emitter with high maximum beam current (>100 nA); Flexible high tension, ranging from 50, 100 to 300 kV and values in between); High probe currents of 0.6 nA in 1 nm spot and 15 nA in a 10 nm spot; Small energy spread of 0.8 eV; TEM point resolution of 0.2 nm; TEM line resolution of 0.102 nm; Magnification ranging from 60X to 1,000,000X; Camera length ranging from 80 to 4,500 mm; STEM HAADF resolution of 0.19 nm; Specimen tilt ranging from -40 to 40 degrees for a double tilt holder and -80 to 80 degrees for a tomography holder; EDS solid angle of 0.13 srad; High resolution STEM with HAADF detector; Embedding of EDX, PEELS and energy filter; spectrum imaging with multiple detectors; Simultaneous data recording by STEM, CCD camera, EDX detectors, EELS spectrometers, and energy filters.



High-resolution transmission electron micrographs of a CeO2 irradiated with 150 keV Kr at 600C to 1×1016/cm2 (a) with the electron beam parallel to the [110] zone axis and (b) corresponding under-focused condition. White arrows indicate location of selected bubbles [L. He, C. Yablinsky, M. Gupta, J. Gan, M. A. Kirk, T. R. Allen, Nucl. Tech. 182 (2013) 164].

Cameca 4000X HR local electrode atom probe (LEAP)

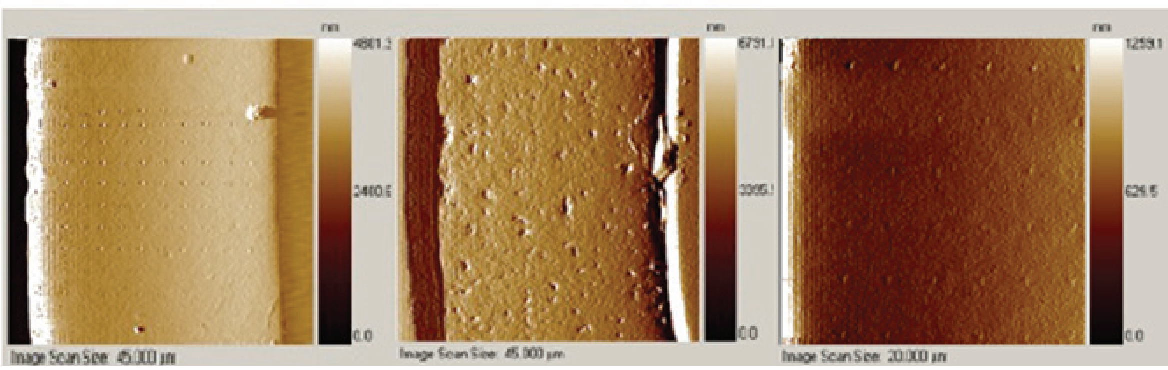
Provides high-voltage pulse mass resolution; Field of view exceeding 150 nm; Local electrode technology; High spatial resolution and sensitivity; Automatically focused voltage and UV laser pulsing with small spot enable improved mass resolution, better yield from poorly conductive samples, and best in class data acquisition rates; Improved signal-to-noise ratio; Large angle reflectron (LAR) design is optimized for very high mass resolution while maintaining a wide field of view.



APT results of alloy Fe-Cr-Mo+yttria, isosurface of 1.0 at.% Y (green), 3.5 at.% Mo (red) and 1.0 at.% O (blue), showing Mo segregates next to oxide precipitates. The scale is in nanometers.

Hysitron TI-950 triboIndenter nanoindenter and AFM

Dual head testing capability; Available force ranges from ≤30 nN to 10 N; In-situ imaging provides nanometer precision test positioning and the convenience of SPM topography; 500 nm resolution staging for sample positioning; Automated testing for high throughput; Top-down, high-resolution color optics for viewing and selection of testing sites; SPM imaging; ScanningWear to observe and quantify wear volumes and rates; Scratch testing for quantification of scratch resistance, critical delamination forces, and friction coefficients; Quasistatic nanoindentation to measure Young's modulus, hardness, fracture toughness, and other properties; AFM/MFM imaging; Heating/cooling stages for investigation of mechanical properties at non-ambient temperatures; Vacuum wafer mounting system that eliminates necessity of gluing or cutting wafers prior to testing; NanoECR a conductive nanoidentation system that provides simultaneous in-situ electrical and mechanical measurements; High load head that goes up to 2 N; Is equipped to operate in Ar and N environments.



Scanning probe microscopy results taken from the three regions in the surrogate TRISO particles which illustrate the nanoindentation regions. Taken from J. Martos, M. A. Okuniewski, R. J. Stromberg, K. Terrani, and P. Hosemann, Material Characterization of TRISO Fuel Particles Using Nanoindentation, The 5th Annual Asia-Pacific Nuclear Energy Forum on Materials for Nuclear Applications and 2nd Annual U.S. Department of Energy Nuclear Energy Programs Materials Science and Engineering Materials Cross-cut Workshop (Berkeley, CA, June 2011).

Fischione model 1040 nanomill

Filament-based ion source combined with electrostatic lens system; Accelerating voltages range from 50 eV to 2 kV and are continuously adjustable; Beam current density is up to 1 mA/cm2; Beam diameter can be reduced to 1 µm at 2 kV; Milling angles range from -12 to 30 degrees; Ar ion beam can be targeted at one spot on the specimen or scanned within a selected area; LN2 conductive cooling; Stage temperature can be reduced to -170 degrees; Everhart-Thornley detector; SED-based imaging technology with 3 mm field of view; Set up to post-process FIB lamella and remove Ga ion beam damage.

Gatan model 695 PIPS-2

Two penning Ar ion guns with low energy focusing electrodes; Milling angles can be changed from -10 to 10 degrees for each gun independently; Ion beam energies range from 100 eV to 8 kV; Ion current density is 100 mA/cm2 at the peak; Whisperlock with X, Y stage to center the region of interest for re-polish; Digital Zoom microscope that operate in real time during milling; Color image stage in Digital Micrograph; Accommodates 2.3 mm, 3 mm conventional TEM disks, and FIB lamella; LN2 specimen cooling; Set up as a post-processing step for FIB lamella to remove FIB Ga ion beam damage.

Microscopy and Characterization Suite

Gatan model 682 precision etching and coating system

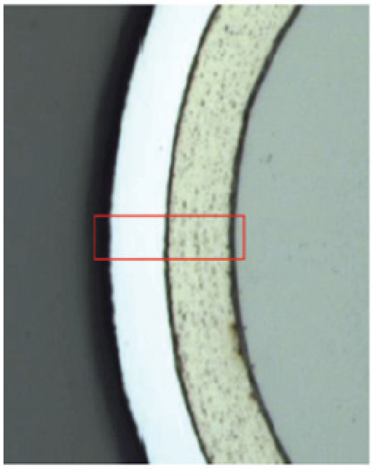
Three penning ion guns with miniature rare earth magnets; Ion beam energy ranges from 1 kV to 10 kV; Ion current density for etch gun is up to 3 mA/cm2 and for coating gun is up to 10 mA/cm2 each; Etched area ranges from 7 to 10 mm depending on gun energy; Coating area is uniform over a 1 inch diameter; Standard coating materials are C, Cr, Pt, Pd, Au; Accepts 1.25” metallographic mounts and most SEM stubs.

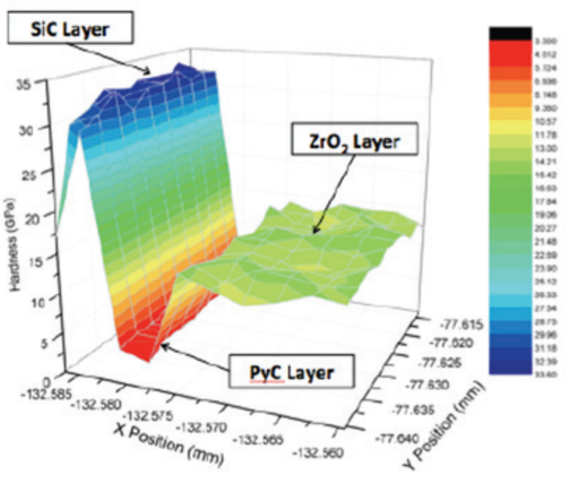
Specimen preparation equipment

Pace Technologies Giga 0900 Vibratory Polishers
Beuhler MiniMet 1000 Polisher-Grinder
Buehler IsoMet Low Speed Saws
Anatech Hummer sputtering system

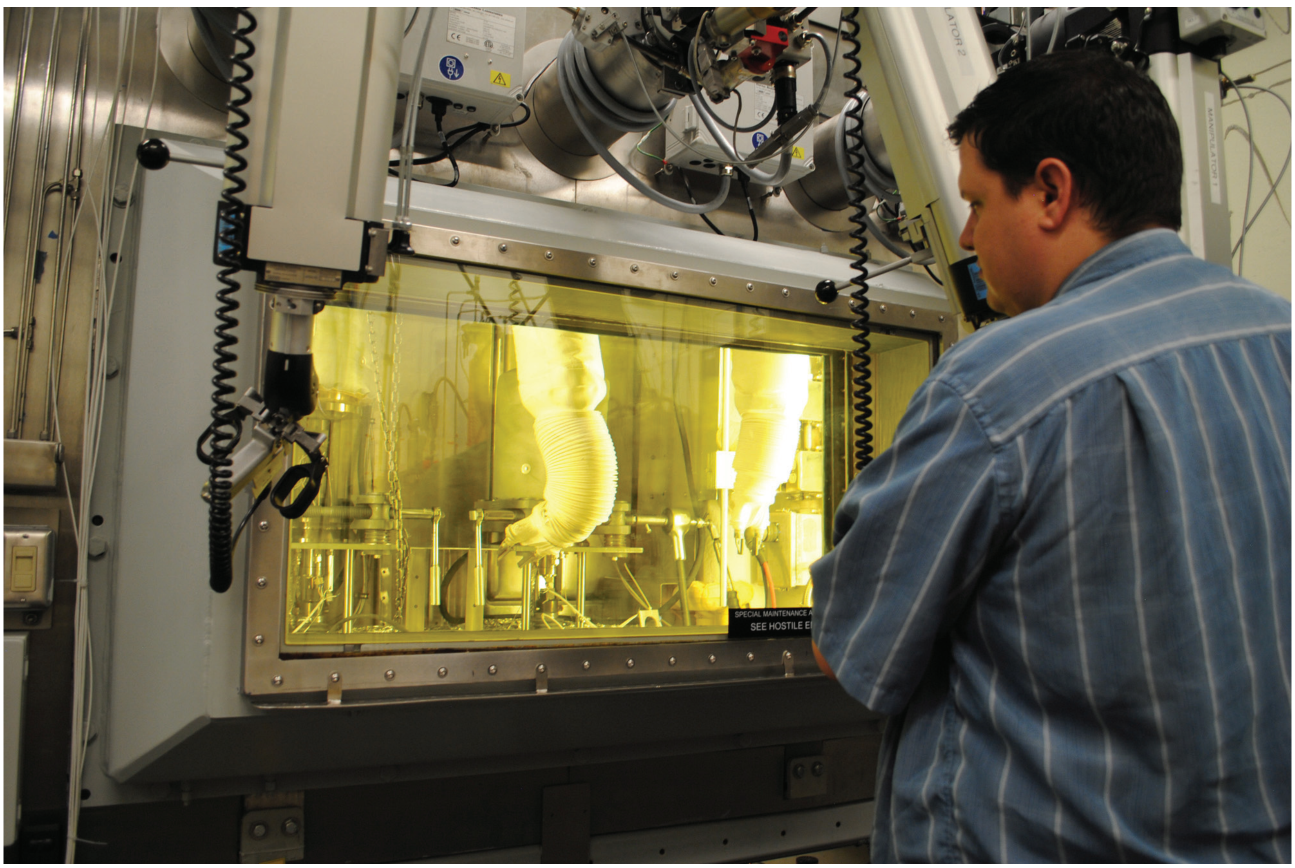
Other available equipment

— Thermal Technology High Temperature Vacuum Furnace
— Thermal Technology High Temperature Vacuum Furnace
— Carver 3853.0H4 Press
— Microlux Variable Speed Miniature Drill Press
— Motic Digital Optical Microscope





Example of nanohardness data acquired on surrogate TRISO fuel (right figure). The locations of the indentations are indicated in the TRISO particle micrograph (left figure). Taken from P. Hosemann, J. N. Martos, D. Frazer, G. Vasudevamurthy, T.S. Byun, J.D. Hunn, B.C. Jolly, K. Terrani, and M. Okuniewski, Journal of Nuclear Materials, 442 (2013) 133-142.



A researcher performing work in the Irradiation Assisted Stress Corrosion Cracking system at FASB.

Fuels and Applied Science Building

The Fuels and Applied Science Building (FASB) is primarily a fuel fabrication facility, but also has the characterization capability for unirradiated and irradiated materials. Fabrication equipment in the facility is used for research and development of nuclear fuels on the laboratory-scale. The fuels are formed into various geometries for use in research experiments at various reactors throughout the world. In addition to fuel fabrication, FASB is used for characterization of fabricated fuels and lightly irradiated structural materials. Characterization includes microscopy (optical and electron), thermal properties and mechanical testing. FASB houses two hot cells used for irradiation assisted stress corrosion cracking studies of highly irradiated structural materials irradiated at the Advanced Test Reactor. The examination capabilities listed below are available to NSUF users.

LEO 1455 scanning electron microscope

Schottky field emission gun, in-lens secondary electron detector, below-the-lens secondary electron detector. Magnifications range from 20X to 60,000X, probe current ranges from 1 pA to 1 mA, accelerating voltages range from 200 V to 30 kV. Accommodates samples as large as 15×15×15cm. Can accommodate long working distances for imaging at low magnifications (20X). Equipped with Oxford EDS and WDS.

JEOL JSM-7600F scanning electron microscope

Schottky field emission gun, in-lens secondary electron detector, below-the-lens secondary electron detector. Low angle backscatter electron detector. Magnifications range from 25X to 1,000,000X, accelerating voltages range from 0.1 to 30 kV, probe currents range from 1 pA to 200 nA. Accommodates samples as large as 100 mm in diameter and 50 mm (H) or 200 mm in diameter and 20 mm (H). Equipped with Oxford EDS, WDS and EBSD.

Zeiss D1M metallographic microscope

Apochromatic correction for the beam path, 6-place reflector turret for convenient change of reflector modules. Automatic component recognition, bright-field, dark-field, polarization, DIC, C-DIC and fluorescence in reflected light modes, LED illumination.

Instron 3366 tensile tester

Used to determine tensile properties and ductility for axial and circumferential directions at room temperature and elevated temperature prototypic of reactor operation or storage. Load capacity of 10 kN, maximum speed of 500 mm/min, return speed of 600mm/min, cross head travel of 1122mm. Vertical test space is 1193 mm, 100:1 force range, load accuracy of 0.5% of indicated load; 500 Hz data acquisition rate. Automatic transducer recognition. Can do both tension and compression tests.

Leco LM247AT microhardness tester

Dual indenter turret can provide Knoop and Vickers on a single tester. Accurate and repeatable load cell Rockwell-type tester. Load ranges from 1 to 2000 gf, fixed aperture diaphragm, a centerable light source and available interchangeable filters provide metallograph quality illumination. Automatic turret accommodates up to four objectives (2.5X to 100X).

Netzsch DSC 404C system

High-temperature differential scanning calorimeter designed for determination of specific heat. Temperatures range from RT to 1500C±2.5%. Heating rates range from 0.01 to 50 K/min. Sensor types include DTA, SDC, DSC-cp. Can perform quantitative enthalpy and Cp determinations under vacuum and in pure gas atmosphere.

Netzsch LFA 427 laser flash system

Temperatures range from RT to 1,300 C. Heating rates range from 0.01 to 50 K/min. Laser power is adjustable and is at 25 J/pulse. Temperature rise is measured with an IR detector. Measuring range for thermal conductivity is between 0.1 and 2000 W/mK and for thermal diffusivity from 0.01 to 1000 mm2/s. Has several atmospheres (inert, oxidizing, static and dynamic). Can accommodate the following specimen sizes: 6 mm to 12.7 mm in diameter, 10×10 mm square, and 0.1 mm to 6 mm in thickness.

Netzsch Dilatometer 402

Temperature ranges from RT to 2000 C. Heating rate ranges from 0.01 to 50 K/min. Measuring ranges 500/5000 m. Maximum sample length of 50 mm and diameter of 12 mm, resolution 0.125 nm/1.25 nm. Equipped with several atmospheres (inert, oxidizing, reducing, static and dynamic).

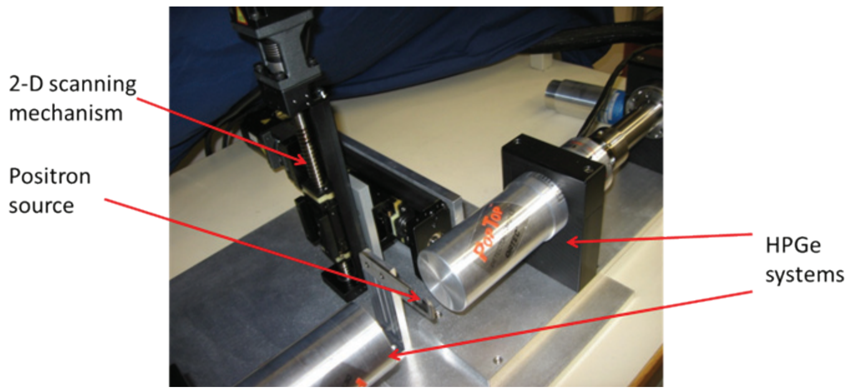
Fuels and Applied Science Building

INEL Equinox 1000 X-Ray diffractometer

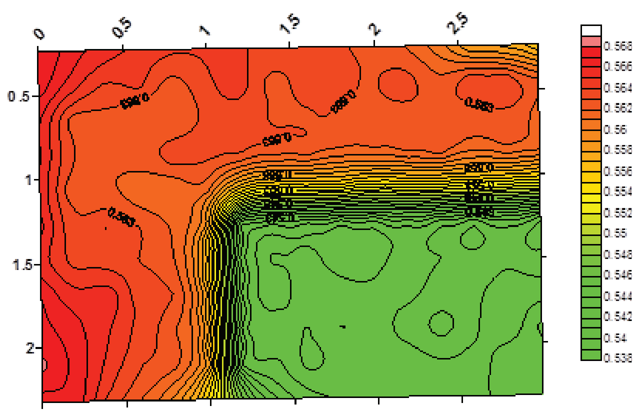
Wavelengths: Cu, Co in a standard sealed X-ray tube; monochromators: Kα1,α2; X-ray generator: 3500 watts (60 kV/60 mA); curved position sensitive X-ray detector, CPS180; curvature radius of 180 mm; real-time acquisition across 110o 20, 30 sample changer positions; software allows for real-time diffractogram display, peak search, deconvolution with several shapes, phase identification and quantification, determination of degree of crystallinity, cell parameters, crystallite size, lattice strain, crystal structure analysis and Rietveld analysis.

Positron annihilation spectroscopy

Positron annihilation spectroscopy (PAS) has the capability of measuring defects within materials within an atomistic to nanometer level. Positrons are sensitive to open volume defects within materials such as vacancies, voids, and bubbles. Therefore, PAS measurements are ideal for irradiated, deformed, or porous materials. A Doppler broadening PAS system that can be used in singular or coincidence mode provides information related to both the concentration and type of defects



Positron annihilation spectroscopy system shown in coincidence mode.



PAS Doppler broadening S parameter results in a surrogate fuel plate. Taken from M. A. Okuniewski, D. W. Akers, M. W. Drigert, C. L. Shull, and L. G. Roybal, Spatially Resolved Positron Annihilation Spectroscopy in Nuclear Fuels, The 16th International Conference on Positron Annihilation (ICPA-16), (Bristol, United Kingdom, August 2012).

present within materials. This system possesses two positron sources, a Na-22 and Ge-68, which allows for the detection of defects at the near surface of materials or further in the bulk. The positrons from the source enter the material and annihilate with electrons. This annihilation results in two gamma rays (~511 keV) that are generated and subsequently measured with high purity gamma ray spectrometers (as shown in the figure below). This system has been designed to conduct two dimensional scanning or point measurements of specimens. The specimen size that can be accommodated ranges from millimeters in length and width up to 75 mm by 150 mm.

Irradiation assisted stress corrosion cracking (IASCC) system

The IASCC test system is installed in two hot cells. The first test cell houses two autoclaves outfitted with Instron, servohydraulic actuators and capable of simulating boiling water reactor (BWR) normal and hydrogen water chemistries as well as pressurized water reactor (PWR) environments using a closed loop, recirculating chemistry control system, and an autoclave to enclose test specimens within the environment. The servohydraulic actuators and associated specimen testing hardware located inside the autoclaves are designed to handle up to a 100 KN applied force, allowing fracture toughness testing of full-sized (1TCT) compact tension specimens in addition to the irradiation assisted stress corrosion cracking test capability. Crack growth is monitored using a reversing current, DC potential drop (DCPD) system. The cell is shielded to allow up to a 45,000 R/hr gamma (contact) source to be handled for extended periods. The IASCC test cell has a companion utility cell with equivalent shielding. The utility cell is outfitted with a tool port for introduction of necessary hardware and serves as the transit point for irradiated specimens as it is also outfitted with a cask ring underneath the cell designed to mate with a modified GE-100 cask. It is also outfitted with a small, JEOL JCM-5000 benchtop SEM to allow fractography of tested specimens.

Irradiation assisted stress corrosion cracking system.

Specimen preparation equipment

— Buehler Isomet low-speed saws
— Buehler IsoMet 1000 precision saw
— Buehler MiniMet 1000 grinder-polisher
— Buehler EcoMet 250 grinder-polisher
— Buehler Vibromet 2 vibratory polisher
— Struers Tegra Doser-5 system
— Struers Accutom 50 high-speed saw
— Anatech Hummer sputtering system
— Anatech CEA 2.2 carbon evaporation system

Furnaces

— Kerrlab Auto Electro-Melt maxi furnace
— Thermolyne 79300 tube furnace
— RD Webb Company EuroTherm 2704 furnace
— High-temperature furnace model 100400
— Electra model 121340 furnace
— Lindberg/Blue furnaces
— Barnstead Thermolyne 48000 furnace

An INL researcher works in the casting lab glove box in MFC's Analytical Laboratory.

Analytical Laboratory

The Materials and Fuels Complex Analytical Laboratory (AL) provides high-quality processing, analysis and characterization of radiological materials. The analytical lab has six shielded hot cells for receiving and analyzing highly radioactive materials. The hot cells are made of 2-feet thick, high-density concrete, with leaded glass shielding windows. A pneumatic transfer system brings samples to the hot cells from other facilities or moves finished samples to a waste cask for disposal. The NMCD hot cells include:

Cell 1: Sample Receiving – Samples are received using a pneumatic system.
Cell 2: Sample Dissolution – Samples or small portions of samples are dissolved.
Cell 3: Sample Storage – A rotating sample rack is used for sample storage.
Cell 4: Gamma Spectroscopy – Some samples are too radioactive to be taken to the counting lab. Therefore, a gamma spectrometer is used to count hot samples inside the hot cell.
Cell 5: Used for multiprogrammatic R&D instrumentation.
Cell 6: Transfer tube to lead-shielded ICP-AES glove box – Once the samples have been prepared in the hot cell, they can be transferred to the ICP-AES glove box without removal from the hot cell.

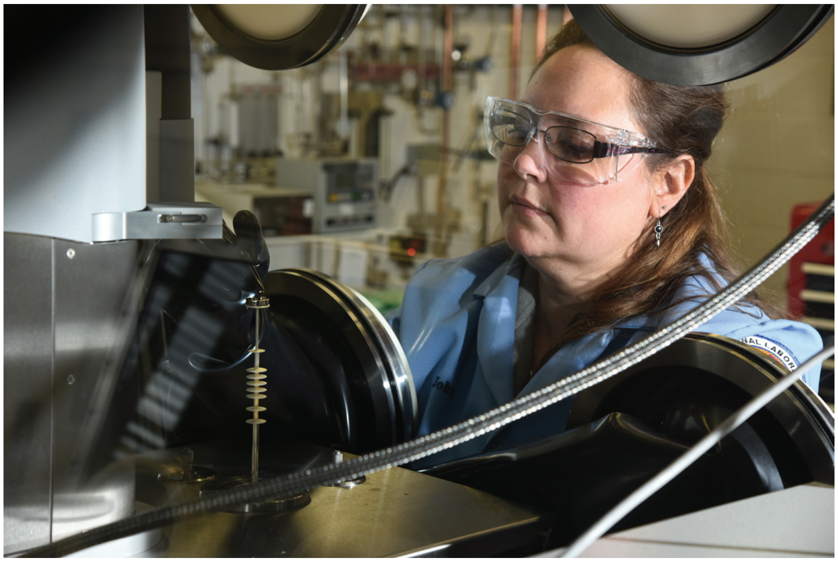
A chain-driven cart moves samples from one cell to the next and the manipulators allow staff to work with the samples without danger of exposure.

PANalytical Empyrean XRD

The PANalytical Empyrean XRD is used for bulk analysis. It uses a copper tube and has a rotating stage. It can be adjusted to the requester's specifications for step size, increment and run time. This instrument can accommodate samples in 1-inch MET mounts as long as they are smearably clean of any radioactive counts. It can accommodate approximately 0.5 inch^2 material that is not smearbly clean of radioactive counts that can be placed in a specialized containment apparatus.

Bruker D8 Discover XRD

The Bruker D8 Discover XRD is a parallel beam instrument. It also uses a copper tube and can be adjusted to the requester's specifications for step size, increment and run time. It can be used for bulk analysis of small samples and has some high focus capability with reliable spatial resolution down to approximately 3mm. This instrument is equipped with cradle and can be used to perform texture studies. Bruker Multex software is used to build pole figures and analyze texture data. Bulk samples up to approximately 0.5 inch^2 that are not smearbly clean can be examined on this instrument, but all samples submitted for texture analysis must be smearably clean.



A researcher performs thermophysical properties testing the Analytical Laboratory's fresh fuels glove box.

Netzsch DSC 404C system

High-temperature differential scanning calorimeter designed for determination of specific heat. Temperatures range from RT to 1,500C±2.5%. Heating rates range from 0.01 to 50 K/min., sensor types include DTA, SDC, DSC-cp. Can perform quantitative enthalpy and Cp determinations under vacuum and in pure gas atmosphere.

Netzsch LFA 427 laser flash system

Temperatures range from RT to 2,000 C. Heating rates range from 0.01 to 50 K/min., laser power is adjustable and is at 25 J/pulse. Temperature rise is measured with an IR detector, measuring range for thermal conductivity is between 0.1 and 2000 W/mK and for thermal diffusivity from 0.01 to 1000 mm2/s. Has several atmospheres (inert, oxidizing, static and dynamic). Can accommodate the following specimen sizes: 6 mm to 12.7 mm in diameter, 10×10 mm square, and 0.1 mm to 6 mm in thickness.

Netzsch dilatometer (DIL) 402

Temperature ranges from RT to 1,600 C. Heating rate ranges from 0.01 to 50 K/min., measuring ranges 500/5000 µm. Maximum sample length of 50 mm and diameter of 12 mm, ΔI resolution 0.125 nm/1.25 nm. Equipped with several atmospheres (inert, oxidizing, reducing, static and dynamic).

Netzsch STA409 thermogravimetric analysis (TGA)

Temperature ranges from RT to 1,600 C. Heating rate ranges from 0.001 to 50 K/min., weighing range 35,000 mg, TG resolution over entire weighing range is 0.1 µg; DSC resolution < 1 µW. Equipped with several atmospheres (inert, oxidizing, reducing, static and dynamic). Automatic sample changer for up to 20 samples. All Netzch thermal property analysis instruments are located within the Fresh Fuels glove box to contain transuranic materials and irradiated non-fuel materials.

Inductively coupled plasma-mass spectroscopy (ICP-MS)

The Analytical Laboratory has two inductively coupled plasma mass spectrometers (ICP-MS). The VG PQ3 instrument is enclosed in a one fourth glovebox, while the ELAN DRC II instrument makes use of a radiological hood. Both containment devices (the glovebox and hood) are used to provide containment while performing analyses on solutions containing radioactive material. In both cases, the plasma and sample introduction components are enclosed in the containment, while pumps and electronics are outside of containment.

The ICP-MS instruments are used to provide elemental and isotopic information on a fast time scale. The unit mass resolution instruments (resolution is about 400), allows the determination of a wide variety of analytes relatively quickly. The instruments are most often used in the "peak jumping" mode, which requires about 1-3 seconds per analyte chosen. A typical analysis will take approximately five minutes of instrument time for a wide variety of analytes. The instrument is capable of determining major components in fuels, to fission products. Typically, most fission products from Rb-85 through Sm-154 are measureable, depending on the materials of construction and the irradiation history.

The analytical figures of merit of the ICP-MS are quite good, considering the quadrupole mass filter. Most analyses have a two sigma error of plus/minus 3-5%. The error determination (determined by the Guide to Uncertainty Measurement (GUM)) is dominated by

A researcher performing work in a glove box.

the error of the standards and the stability of the signal. Detection limits for most species (3 sigma) are less than one pg/mL in solution.

The ELAN DRC II ICP-MS is equipped with a Dynamic Reaction Cell (DRC). In essence, the DRC allows for chemical reactions to take place before the quadrupole mass filter. This is an excellent research tool that can be applied to the removal of isobaric interferences such as U238H-1 on Pu-239 and Ar-40O-16 on Fe-56. There are a variety of gases used for the reaction cell, but the most common ammonia gas.

Multi-Collector ICP-MS (MC-ICP-MS)

The multi-collector ICP-MS is one of the newest and provides one of the largest leaps in technology in recent plasma spectrochemistry. The MC-ICP-MS is a hybrid of the quadrupole ICP-MS and the TIMS. The resulting instrument is one that has a much more robust ion source (plasma) and the magnetic sector capabilities of the TIMS. However, the resulting instrument makes high precision isotope ratios available to almost any element in the periodic table, regardless of the ionization potential of the element.

The Nu instrument installed in the AL makes use of a radiological hood as the other ELAN ICP-MS. The instrument is equipped with 15 separate detectors, 12 Faraday Cups and 3 full size electron multipliers. Two of the three electron multipliers are equipped with high abundance filters that increase the abundance sensitivity from approximately 5 ppm to approximately 200 ppb. This is a useful tool when analyzing small isotopes next to very large ones.

The analytical figures of merit are essentially the same for the MC-ICP-MS and the TIMS. Totals and isotopic compositions have been compared and have been statistically the same. The control charts for U and Pu measurements are in the process of being developed.

Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP-AES)

The ICP-AES instrument attached to Hot Cell 6 utilizes a lead lined glovebox. The plasma and sample introduction equipment is located inside the glovebox. The atomic emission is transported to the spectrometer via a quartz window that is installed in the glovebox. The lead lining allows for the analyses of samples that have considerable gamma signature, with minimal whole body exposure to the scientific staff.

The ICP-AES is used to measure elemental concentrations of metallic elements from mid nanogram per milliliter (ng/mL or ppb) up to species that are major components. Most species have detection limits in the low ng/mL.

Thermal ionization mass spectrometry (TIMS)

The longest established and proven method for doing uranium and plutonium in PIE samples is the TIMS instrument. The TIMS instrument utilizes a magnetic sector mass filter and seven faraday cups to simultaneously measure the ions generated from the thermal ionization of the sample. By utilizing isotope dilution (spiking with a known amount of U-233 or Pu-244), a very accurate and precise measurement is achieved. The end result is a total assay of less than 1 percent RSD at 2 sigma for U and Pu. The precision of isotopic determinations for major components are better than 0.1 percent, two sigma.

Uranium and plutonium measurements on the TIMS have one of the highest QC regimens in the AL. Uranium and plutonium measurements are made with longstanding QC samples which have control charts for both isotopic composition and totals.

Waste form testing glove box

The WFTG is used for contamination control during metallographic sample preparation of actinide bearing materials and associated samples, and other analytical sample preparation applications which include but are not limited to sectioning of materials. It is a single glove box system with a main operating floor and a floor level well on the west end of the box. The east end of the glove box is equipped with a transfer port with sealed doors, three glove ports. The glove box has a "one pass" air atmosphere with pre-filters that are exhausted through double high-efficiency particulate air (HEPA) filters. The glove box is equipped with multiple furnaces.

Special form glove box

The SPG is used for contamination control during sample preparation and testing of fuel materials using thermal characterization instrumentation and other analytical sample applications. It is a combination of two Cenham glove boxes connected by a pass-through port. The glove box is equipped with transfer assemblies, a transfer port with sealed doors, and a bag-out port. The glove box has a "one pass" air atmosphere with pre-filters that are exhausted through double high-efficiency particulate air (HEPA) filters.

Radiochemistry glove box

The RG is used for contamination control during chemical dissolutions, separations, sample preparation, electrolytic deposition, electro-polishing, and other chemistry on high-contamination alpha- and beta-gamma emitting radionuclides. It is a single glove box system with four gloveports, a transfer port with sealed doors, a rapid transfer port, and a small smear port. It has a "one pass" air atmosphere with intake and exhaust HEPA filters.

Wet prep glove box

The WPG is an air atmosphere, flow-through style glove box. The glove box train consists of three components: two interconnected glove box bays and a fume hood. The WPG is used as contamination control for general manipulations and sample preparation of materials having alpha and beta-gamma emitting radionuclides of higher activity than is allowed for use in fume hoods. A small polycarbonate hood located in the north WPG contains a hot plate which is used for the hot dissolution of samples.

Casting laboratory glove box

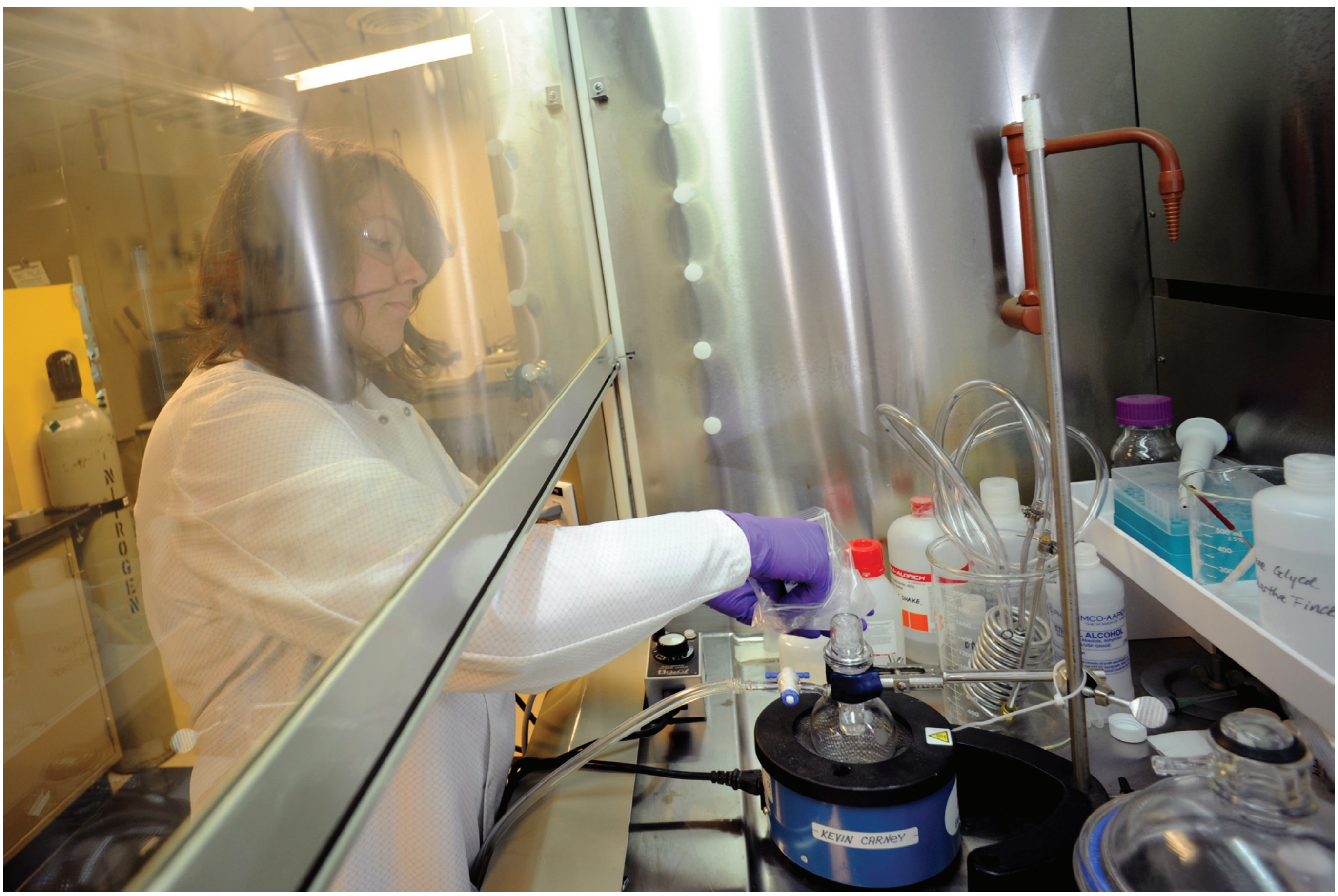
The CL glove box is an inert (Ar) atmosphere glove box that is utilized to develop equipment, processes, and procedures to cast molten nuclear materials into various shapes, which will be suitable for fuel applications. The glove box contains a specially designed furnace enclosed in a stainless steel enclosure that houses structures to accommodate a mold(s), crucible, induction coils, thermocouples, secondary containment to capture melt upon failure of other internal components, and can reach a temperature of 1650C. The glove box contains two additional furnaces with a maximum temperature of 900C.

Analytical Laboratory

The fresh fuels glove box

The FF glove box contains a suite of thermophysical properties characterization instruments and an optical metallograph. The glove box has an inert atmosphere of argon that is regenerated to high purity below 10 ppm Oxygen and moisture monitoring. The instruments are connected to separate purge gas, vacuum and exhaust lines to protect the purity of the glove box atmosphere.

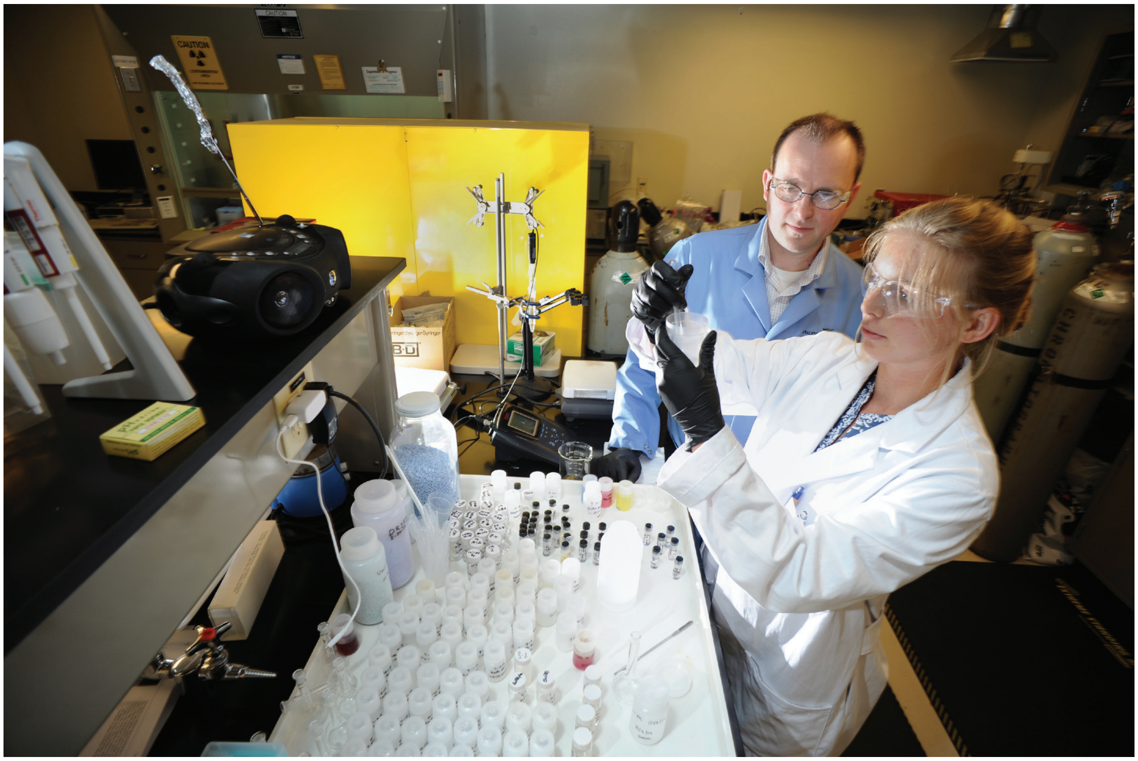
It also has a fume hood located on the northwest corner and the transfer chamber is located inside of this fume hood. This will allow for contamination control during transfer of materials both into and out of the glove box.



INL researcher performing work in a fume hood.

Radiochemistry Laboratory

The laboratory is equipped with standard analytical equipment, including motorized pipettes, pH meters, conductivity meters, computer-controlled automated potentiometric and Karl Fischer titrators, fraction collectors, ovens, centrifuges, analytical balances and vacuum systems. A spectrophotometer (Cary 6000i) is located next to the radiological hood and connected to the cuvette in the hood via fiber optic cables. Radiometric counting equipment includes a Perkin Elmer Tri-Carb 7170TR/SL liquid scintillation counter with alpha/beta discrimination capability, two HPGe gamma spectrometers, a Changer Lab Model 6000N gamma spectroscopy sample chamber equipped with an ORTEC GEM50P4 coaxial HPGe detector and DSPEC gamma spectrometer.



A researcher analyzing a solution sample in RCL.