Post Irradiation Examination Capabilities Guide

Post-irradiation Examination Capabilities at the Idaho National Laboratory

Post-irradiation examination capabilities available to ATR NSUF users are included in three primary facilities:

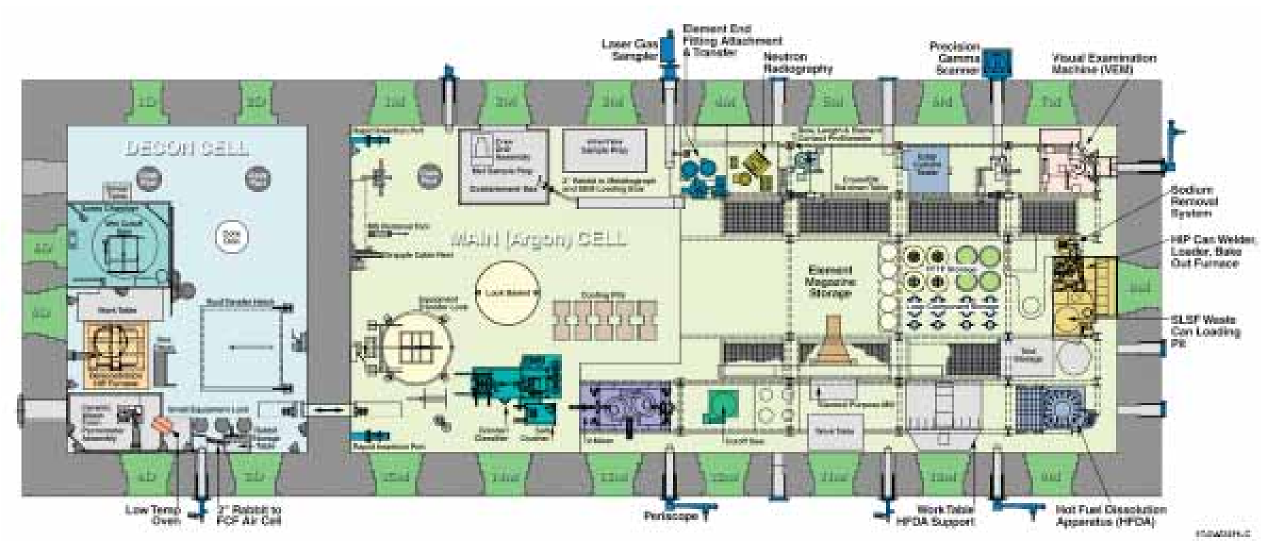
- The Hot Fuel Examination Facility (HFEF), a large hot cell facility
- The Analytical Laboratory (AL), focused on analysis of irradiated and radioactive materials
- The Electron Microscopy Laboratory (EML), a radiological facility containing optical, scanning, and analytical microscopes

HFEF is the primary post-irradiation examination facility at INL, and is described in detail below.

General Description of HFEF

Located at the INL Materials and Fuels Complex, 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 nondestructive (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.

The INL, the HFEF is comprised of 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.



HFEF Process Areas and Equipment Locations

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-lb electro-mechanical 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.

Material Transfers

Transfer of radioactive materials from shipping casks to the hot cells generally takes place using the cask tunnel and cart. Large shipping casks greater than 17 ft. long, 56 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 it can be unloaded remotely. The maximum weight and dimensions for transferring items through the cask tunnel are as follows.

Hot fuel examination facility

Examination Equipment

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

HFEF NDE Capabilities

Non-Destructive Examinations	Equipment Used
Neutron radiography	250 kW TRIGA reactor
Element/capsule diameter measurements	Element Contact Profilometer
Element/capsule gas sampling	Gas Assay Sample and Recharge
Element/capsule weight	Element/Capsule Balance (Mettler)
Element/capsule fission and activation product distribution	Precision Gamma Scanning
Element/capsule bowing and length	Bow and Length Machine
Element/capsule visual exam	Visual Exam Machine
Macro photography	High resolution digital photography
High precision specific gravity measurements	Pycnometer

Neutron Radiography

The TRIGA reactor enables neutron-radiography irradiation 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

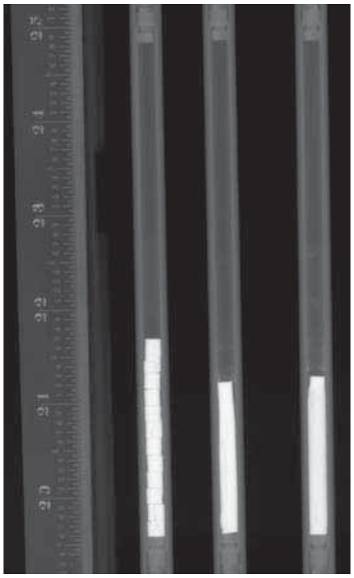
Equipped with two beam tubes and two separate radiography stations, the neutron-radiography capability is one of the finest in the world for irradiating small components, a process not possible using conventional x-ray methods. Neutron radiography of elements, capsules and loops is performed in the main cell at workstation 4M. Specimens are placed into a radiograph holder that is lowered into the TRIGA reactor neutron beam located below the floor. The holders optimally position the specimen for radiography without excessive neutron scattering.

Both indium and dysprosium 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 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. Other methods, including Polaroid and track-etch radiography, are available for special applications.

A neutron generator (14.3 MeV neutrons at 1.0×10^{11} neutrons/sec) on the north beam tube is available for development of non-destructive assay techniques for fissionable material waste forms.

System Limits for Neutron Radiography

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



A neutron radiograph or irradiated fuel test specimens.

Element Handling and Positioning Equipment

Special equipment is available for handling cylindrical fuel elements and capsules; these minimize the amount of damage that might occur from using manipulators during examination. The system allows handling and examination of magazine loads of up to 20 elements and increases the throughput during the examination process. Use of the element handling equipment also enables safe handling of elements up to 152 in. long.

Element Handling Equipment Parameters and Limits

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.

Precision Gamma Scanner

This equipment measures fission and activation-product activity distribution in fuel elements or capsules, providing valuable information about how reactor operation and storage affect components. These measurements can provide data about

- Relative fuel burnup and power profiles of reactor fuels
- Structural activation profile of core components
- Position and dimension of internal structures within fuel assemblies
- Relative distribution of various isotopes of interest in fuel
- Breached elements or capsules

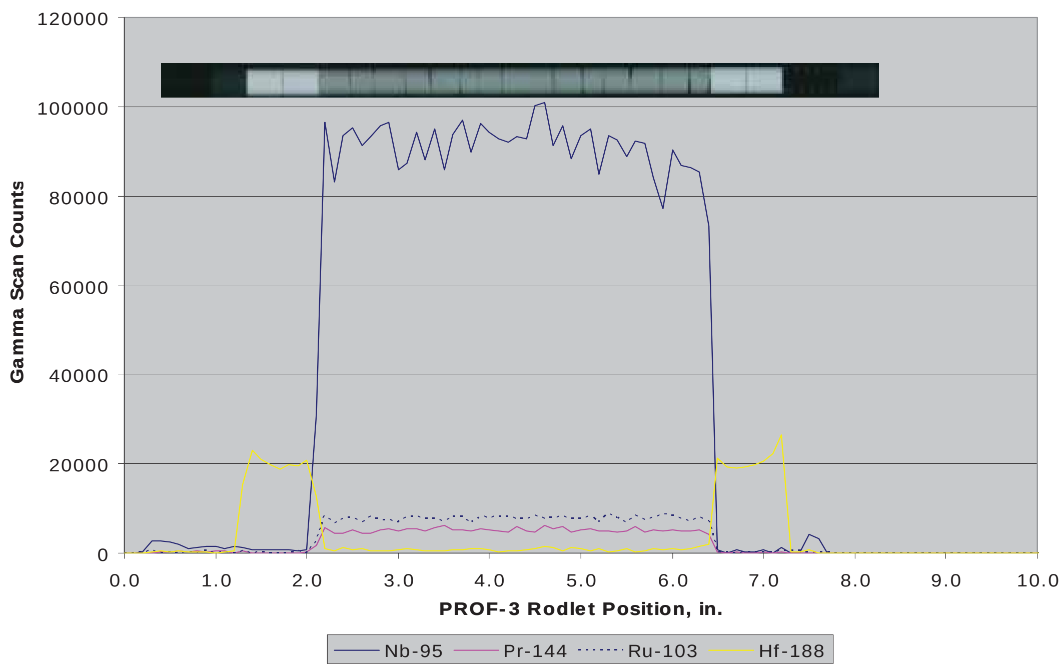
The gamma scanner can be used for scanning large components such as test loops, as well as reactor components and fuel elements. Two types of gamma scans are generally performed:

- Gross gamma scans to determine the distribution of activity over the component's length or width
- Isotopic gamma scans to determine the isotopic distribution of activity over a component's length or width.

HFEF has an extensive isotope library that can be expanded to meet a user's particular needs.

Gamma Scanner Limits

Parameter	Limit
Maximum count rate	86,000 cps
Maximum weight	500 lb
Slit dimensions	0.875-in. long X 0 to 0.099-in wide
Maximum vertical travel	152 in. (nominal)

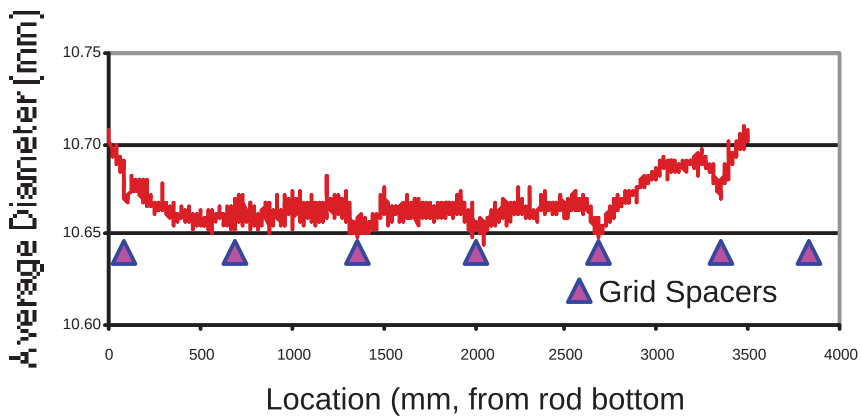


Gamma scan of irregular MOX fuel pellet stack after irradiation compared to neutron radiograph.

Dimensional Inspection

A continuous-contact profilometer measures axial and spiral diameter profiles of elements and capsules. Horizontally opposed linear transducers contact the element as it is pulled vertically through sapphire-tipped probes. Guide rollers positioned above and below the transducers maintain the element vertical with respect to the transducers.

Measurement range is for element diameters between 0.174 in. and 0.840 in., with a maximum diametral swelling of 0.02 in. The swelling range is limited by the linearity of the probes for the size of elements handled. Certified calibration standards for each element size are used for zero, mid-span and full-span calibration. Measurement accuracy through this range is within $3x10^{-4}$ in. ($7.6x10^{-3}$ mm).



Diameter of light water reactor fuel rod as a function of position.

Fission Gas Measurement and Analysis

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. After puncturing and measurement, the element may be refilled with any specified gas and rewelded. Although primarily for contamination control rather than in-reactor service, these welds are well characterized and have been tested to 100 psia. Reactor quality welds could be produced with further characterization.

The system is comprised of a 150-W pulsed laser, shielded optical and gas cell-wall feed-through, a mechanical pump, calibrated volumes, gages 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.

Gas Assay Sample and Recharge System Specifications

System Limits and Capabilities	
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	Better than ± 5% for pressure and volume
Laser	
Type	Neodymium - 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	l 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

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.

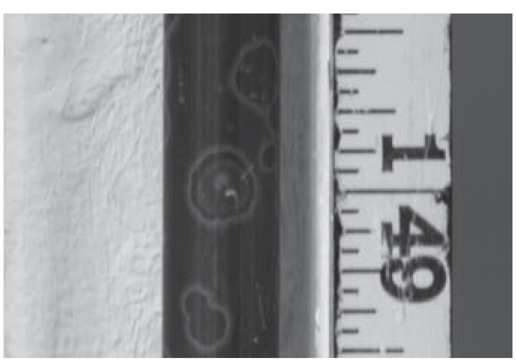
Bow and Length Machine Limits

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.

Visual Exam Machine

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.

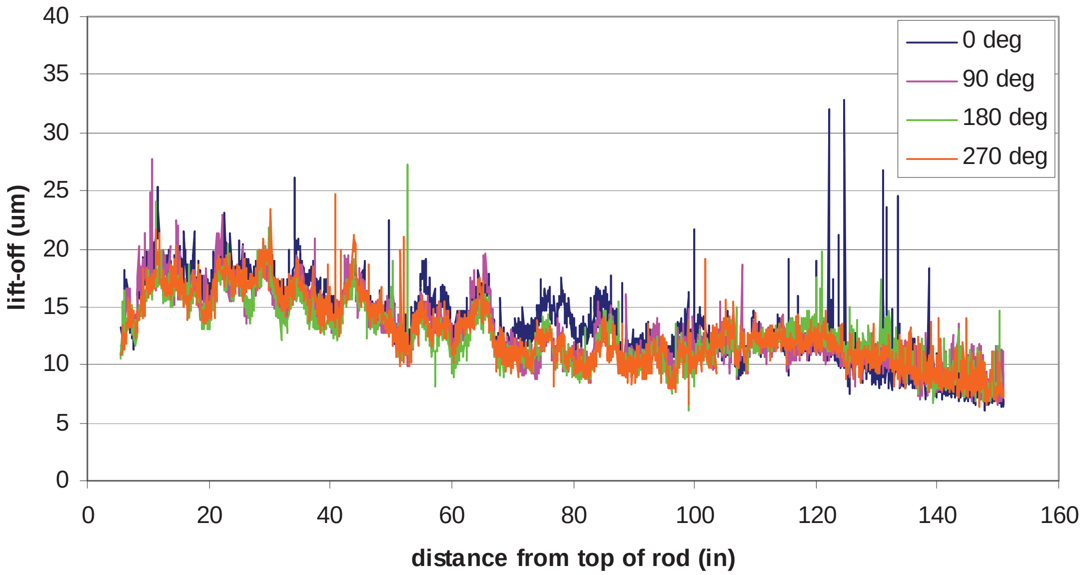
The Kollmorgan periscope provides controls for aiming the objective (i.e., pointing the line-of-view), selecting among three magnifications, and focusing the image. The standard (spherical) optics of the periscope have been replaced with special planar optics that maintain the entire surface of a flat object (oriented normal to the optical axis of the system) in focus at the film plane.



Visual image of features on a light water reactor fuel rod.

Eddy Current Testing

Two eddy current devices are available to measure oxide thickness and material defects on both rod-type and flat plate specimens to an accuracy better than ±0.13 mm (±5 microns). The maximum sample size is 25.4 mm dia. x 3.9 m L (1”x 154”).



Destructive Examination Equipment

Destructive examination capabilities are available for characterizing spent nuclear fuel and other irradiated materials. The following table lists the destructive examinations and the equipment used to perform the examinations. Brief descriptions of the equipment follow.

HFEF Destructive Exam Capability

Destructive Exams	Equipment Used
Sample cutting and preparation	Containment box in main cell Zone 2M
Mounting samples in metallographic mount	Containment box in main cell Zone 2 M
Fuel sample visual exam and photography	Leitz metallograph in metallographic loading box
Detailed photography of sample	High resolution digital photography
Scanning Electron Microscopy	SEM in metallographic loading box
Microhardness	Leitz metallograph in metallographic loading box
Punch samples TEM sample preparation	Subassembly Hex-Can punch/sample thinning Zones 3M and 2M

- Sample Preparation (Containment Box)

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

HFEF houses a shielded metallography cell connected to the main cell via pneumatic transfer tube. The containment box operates under nitrogen or argon atmosphere to prevent rapid oxidation of sample surfaces. Metallographic images of irradiated specimens can be acquired using either a Leitz metallograph or the optical system of the LECO AMH43 microhardness tester.

Leitz Metallograph Limits	
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

Microhardness

A LECO AMH43 automatic microhardness tests is located in the HEFF metallography cell. The microhardness tester is capable of applying loads from 10g to 1 kg. The sample stage can be position controlled to within ±1 μm. Image acquisition is through a high resolution CCD camera.

Chemical and Isotopic Analysis

The Materials and Fuels Complex Analytical Laboratory (AL) is coupled to HFEF via a pneumatic sample transfer system. The AL offers NIST traceable chemical and isotopic analysis of irradiated fuel and material via wide range of techniques, such as ICP-MS (Inductively Coupled Plasma- Mass Spectrometry), ICP-OES (Inductively Coupled Plasma- Optical Emission Spectrometry), and ICPMS-DRC (Inductively Coupled Plasma-Mass Spectrometry – Dynamic Reaction Cell), and TIMS (Thermal Ionization Mass Spectrometry).

Electron Microscopy

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 three primary instruments in EML are a JEOL 2010 scanning transmission electron microscope (TEM), a JEOL JSM-7000f scanning electron microscope (SEM), and a Zeiss DSM 960a SEM. The TEM is capable of operating at 200 kV, and is capable of magnifications from 2,000 X to 1,500,000 X. It is equipped with an Oxford Instruments energy dispersive X-ray spectrometer that can be used to gather information about the elemental make-up of a sample. Crystallographic information can be obtained by recording the diffraction patterns formed by electrons as they pass through the sample.

The JEOL SEM is a field emission instrument capable of operating at 30 kV, and is capable of magnifications from 15 X to 100,000 X. It is equipped with Oxford Instruments energy dispersive (EDS) and wavelength dispersive X-ray spectrometers (WDS) that can be used to obtain quantitative information about the elemental composition of a sample. It is also equipped with an electron back scatter diffraction detector (EBSD) that can be used to obtain crystallographic information about a sample by recording the diffraction patterns formed by electrons when they tunnel through a sample at glancing angles.

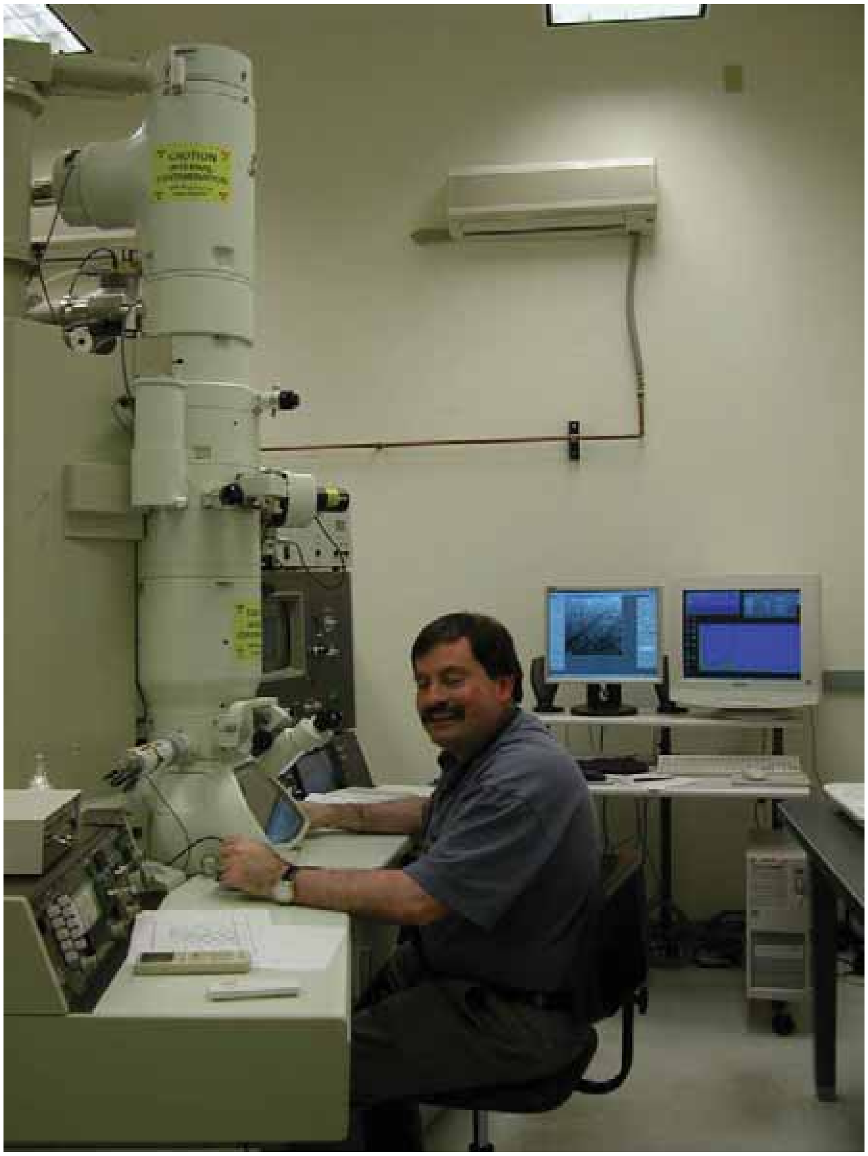
The Zeiss SEM is capable of operating at 30 kV, and is capable of magnifications from 6 X to 50,000 X. It is equipped with Oxford Instruments energy dispersive and wavelength dispersive X-ray spectrometers and an electron back scattered diffraction camera.

In addition to the TEM and SEM, EML also has several optical microscopes. Some of these are used to support sample preparation, and others are used for optical characterization of samples. Capabilities for sample preparation include cutting, grinding, and polishing, as well as specialized methods such as ultramicrotomy (cutting ultrathin slices of material with a special machine using a diamond knife), chemical and ion milling to produce thin, electron-transparent samples, etching, and coating. Fume hoods (radiological and non-radiological) and a radiological glovebox are available to protect workers and the environment from hazardous materials.

Air glovebox for sample preparation

Zeiss Scanning Electron Microscope

JEOL Field Emission Scanning Electron Microscope with EDS, WDS, and EBSD



JEOL Transmission Electron Microscope

PIE Capability Upgrades

Looking to the future, the state-of-the-art post-irradiation examination capabilities at HFEF will continue to play a vital role in nuclear energy development. The INL is will install the following equipment from in the near term:

- Shielded Electron Microprobe

Designed to assess fission product distribution in irradiated fuels, this new instrument performs micro-structural and micro-chemical analysis of fresh and irradiated fuels and waste forms. As a specialized scanning electron microscope, it can also analyze localized micron-scale chemical composition data of irradiated fuels and materials.

- Thermal Ionization Mass Spectrometer

Replacing an existing instrument that has reached the end of its operational life, this instrument will perform elemental assay and isotopic composition on plutonium, uranium and minor actinides prepared from fresh and irradiated fuels.

- Focused Ion Beam Instrument

This new instrument has the ability to analyze the three-dimensional structure and chemistry of materials on a submicron scale. The goal is to characterize irradiated nuclear fuels to detect submicron-level damage, which would make the INL instrument unique in the world. A better understanding of this process has significant potential to improve in-reactor fuels and materials performance.

- Micro X-Ray Diffractometer

The purpose of this device, which performs micro-scale phase identification, small-sample powder diffraction and texture determination, is to track the evolution of fuel structure during irradiation.

- Mechanical Test Equipment and Sample Preparation Equipment

Funded by Battelle Energy Alliance, these upgrades include new mechanical test and sample preparation equipment in the HFEF hot cells – specifically a mechanical test load frame, power supply and an out-of-cell control console as well as sample cutting and preparation tools.

- TN-FSV Cask NRC License Modification

This work comprises modifying the Certificate of Compliance for the TN-FSV transportation cask to include payloads important to the mission of INL fuels research and reactor development. Also funded by Battelle Energy Alliance, the scope includes fabrication of a new inner-shielded cask insert.

Irradiation Assisted Stress Corrosion Cracking (IASCC) facilities

In late 2012, the INL completed installation of the first of two hot cells dedicated to stress corrosion cracking and fracture toughness testing of irradiated materials. These test systems are ready for use beginning October, 2013. 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 Pressurised Water Reactor (PWR) environments using a closed loop, recirculating chemistry control system, and an autoclave to enclose test specimens within the environment. Currently only BWR environments are used in order to preserve the cleanliness of the chemistry loop and autoclaves but PWR environments could be considered.

Figure 1: IASCC test systems at INL.

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 (1T-CT) compact tension specimens in addition to the irradiation assisted stress corrosion cracking (IASCC) test capability. Crack growth is monitored using a reversing current, DC Potential Drop (DCPD) system similar to those employed by GE Global Research Company and Studsvik AB. 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.