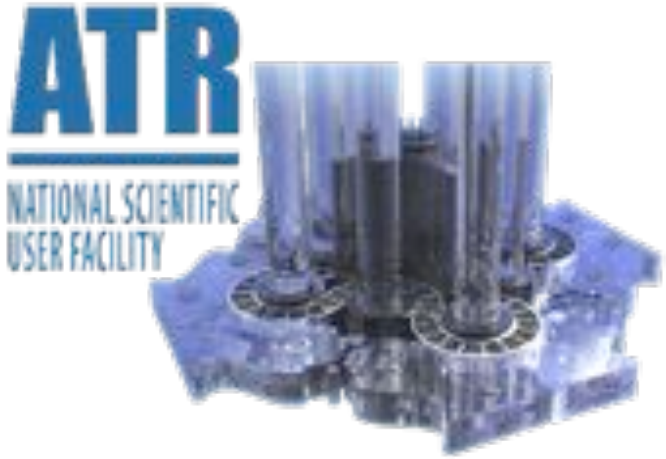
Postirradiation Examination Capabilities

Mitchell K. Meyer, Ph.D

June 7, 2010



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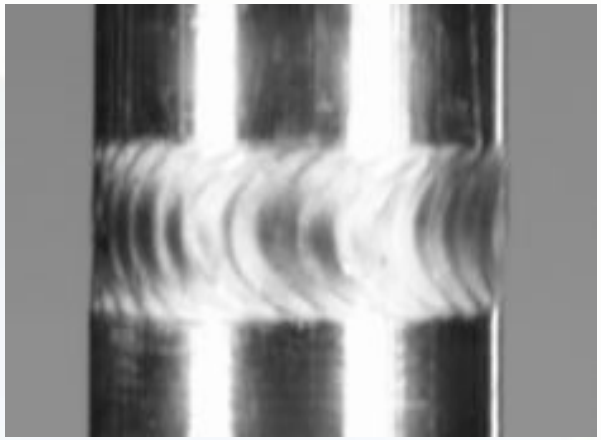
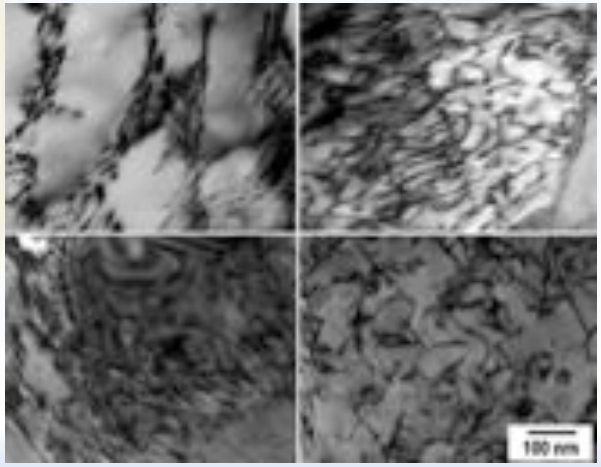
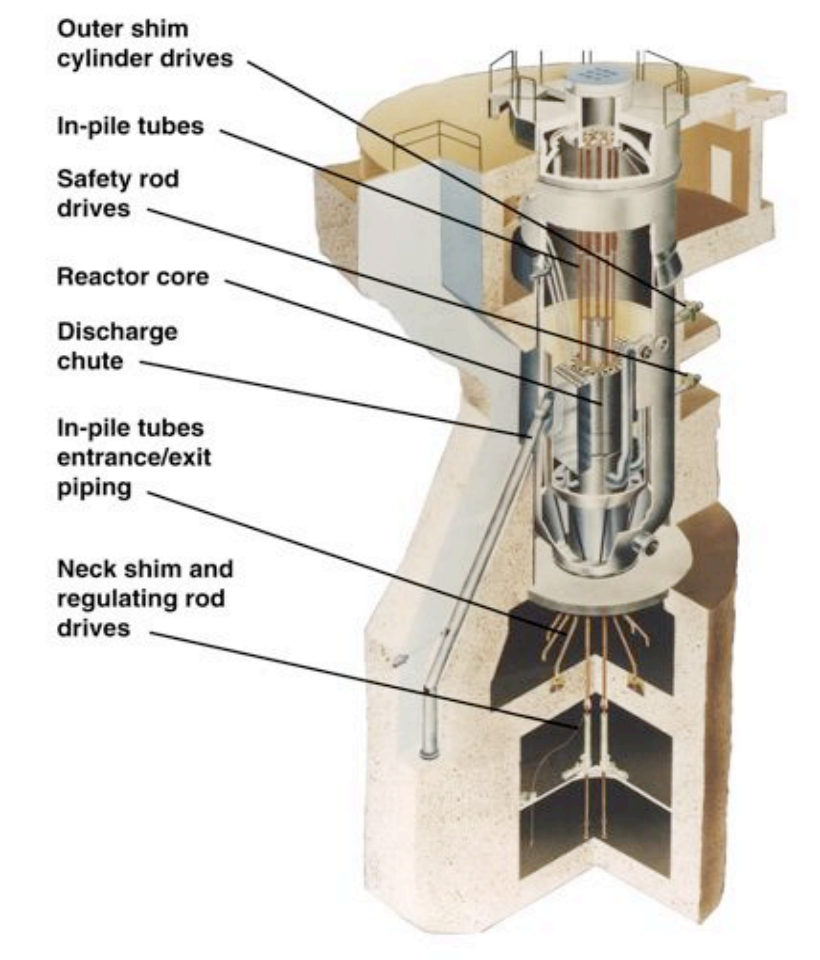
PIE Overview

- There is currently no capability for in-reactor microstructural characterization
- Post-irradiation examination (PIE) is essential to understanding material response to radiation
- Data from ‘traditional’ PIE is limited
- New microstructural characterization tools will allow for much better understanding of nuclear fuel and material irradiation behavior in the future
- A thorough PIE normally takes about one year to complete - I will condense to 30 minutes





First Step: Move from Reactor to Hot Cell



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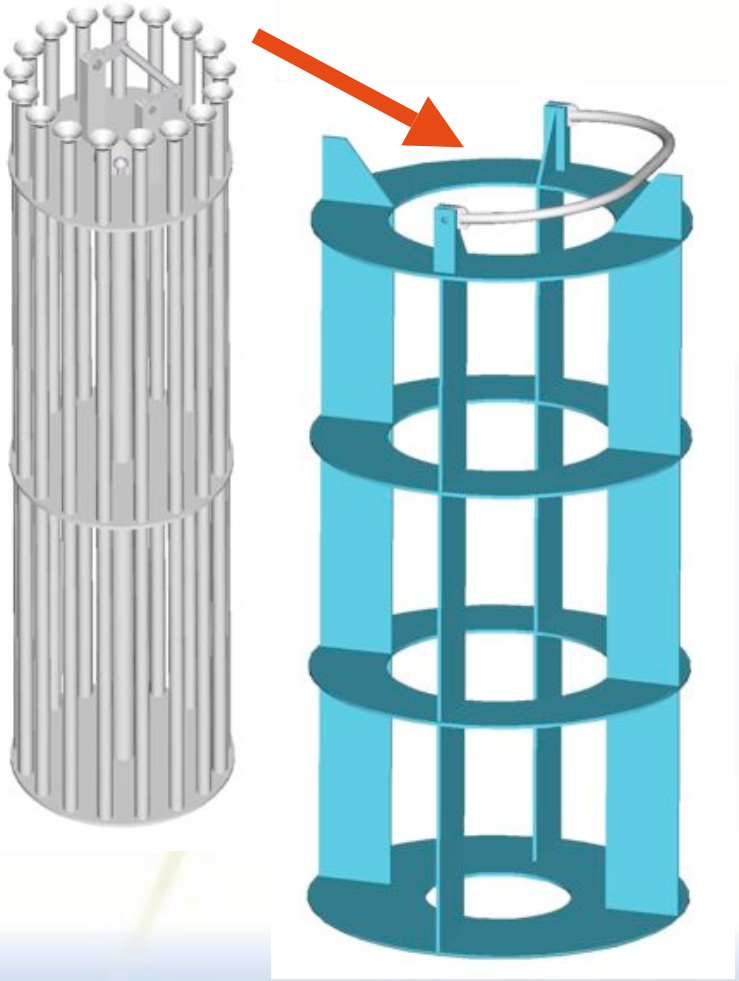
Typical Shipping Basket

- **Shipping Basket**
  - 17 tubes, 0.870-in. ID
  - AFC-1B,D, Æ, F; LWR-1; GFR-F1; up to 11 Cd baskets
  - 15.0-in. diameter
  - 50.4-in. height
  - 96 lbs.
- **Basket Guide**
  - Centers shipping basket in cask cavity
  - No safety function
  - 15.5-in. ID; 26.0-in. OD
  - 53.2-in. height
  - 184 lbs.



Cask Handling at HFEF

Off-loading Cask in HFEF Truck Lock

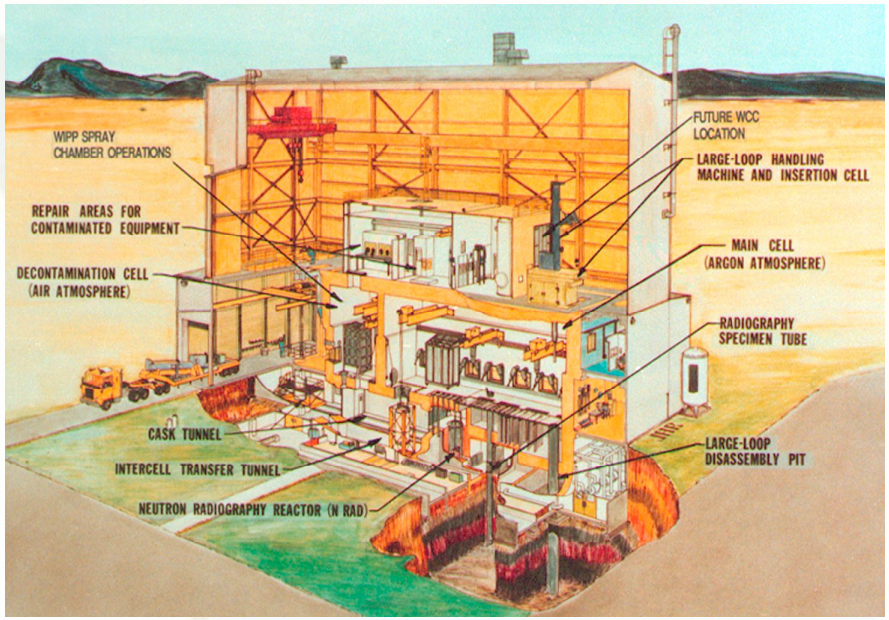
Cask Handling at HFEF

- Cask removed from truck using overhead crane
- Cask positioned above truck lock hatch
- Cask lowered through hatch into cask cart

Cask Handling at HFEF

- Cask with shield ring in cask cart moved into cask tunnel
- Cask cart positioned under main cell penetration

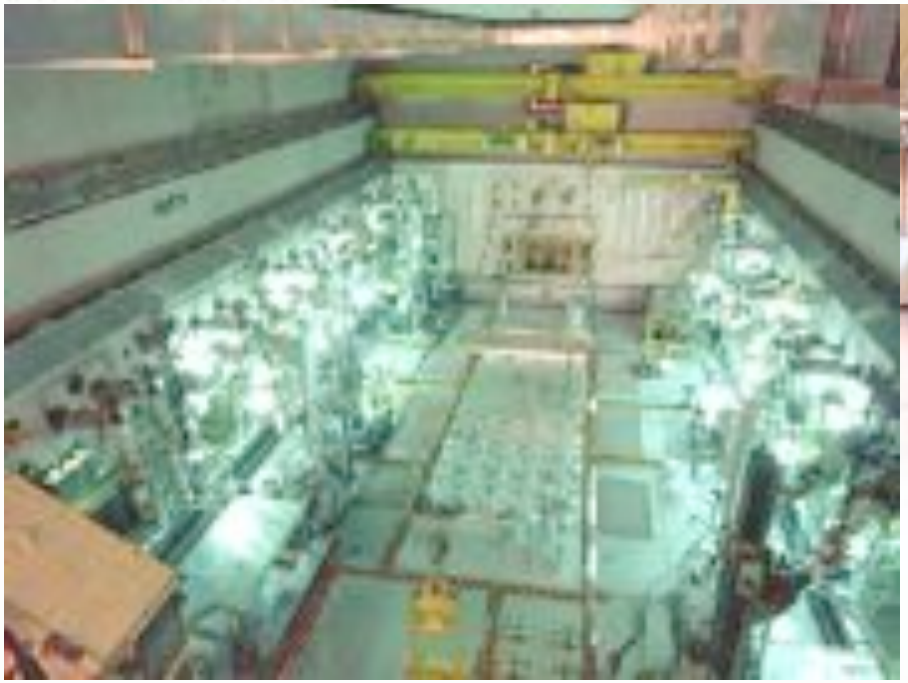
Hot Cell Facilities - HFEF



- **Seismically qualified building housing high-density concrete shielded cell (α, β, γ)**
- **Support infrastructure (pumps, fans, filters, cask handling, radiological control, etc.)**
- **Truck lock, hot repair area**

Hot Cell Facilities - HFEF

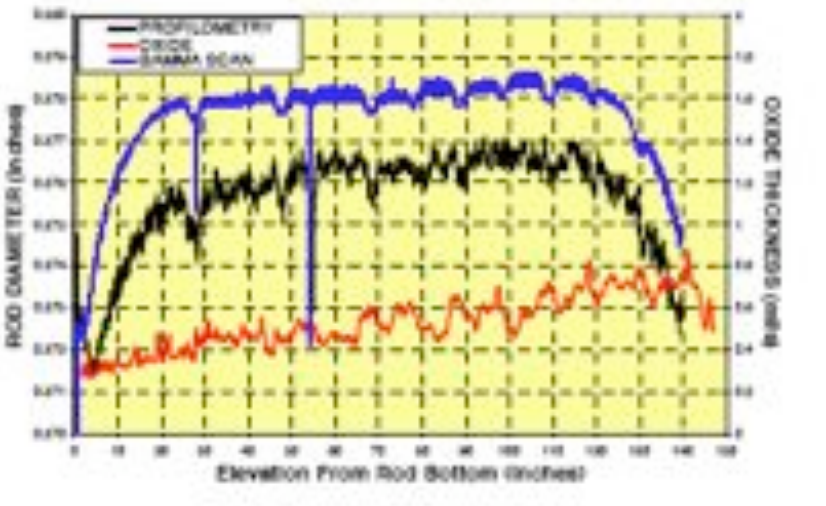
ATR National Scientific User Facility

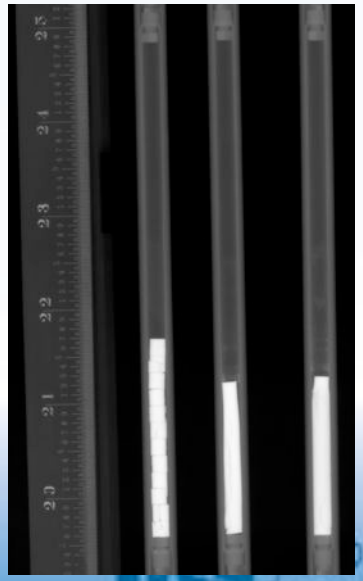


- Walls are thick reinforced concrete (~4 ft)
- Leaded glass windows
- Argon atmosphere
- No entry into main cell > 30 years, all work and repairs are done remotely

Non-Destructive Examinations

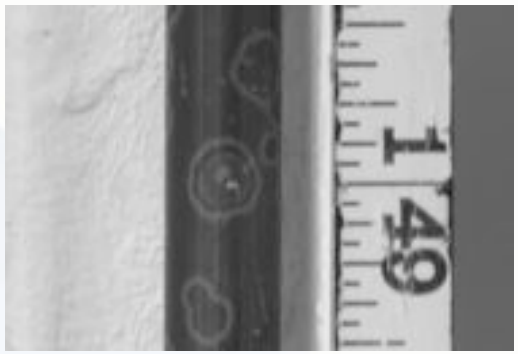
- Visual Inspection
- Neutron Radiography
- Dimensional Inspection
- Gamma Scan
- Eddy current Oxide Layer Tester
- Eddy current Cladding Integrity Tester
- Gas release measurements (fuel)

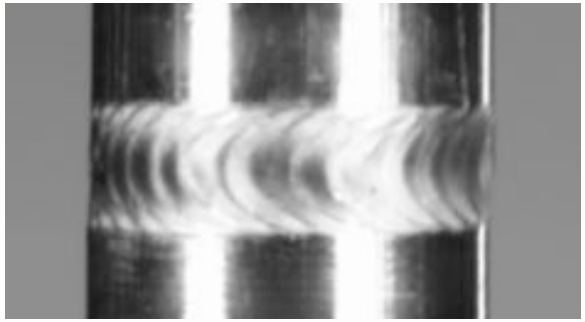


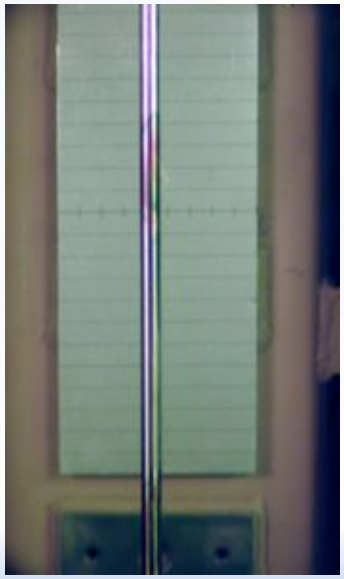


Visual Inspection

- Detailed Visual Inspection
  - Thru-cell wall periscope 2X, 10X, 25X
  - Digital Still Camera Photography
- Macro photography inspection
  - Thru Hot Cell window
  - Digital Video Camera Photography
  - Digital Still Camera Photography

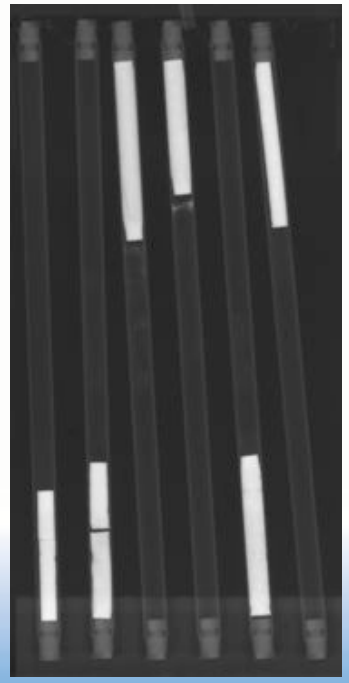


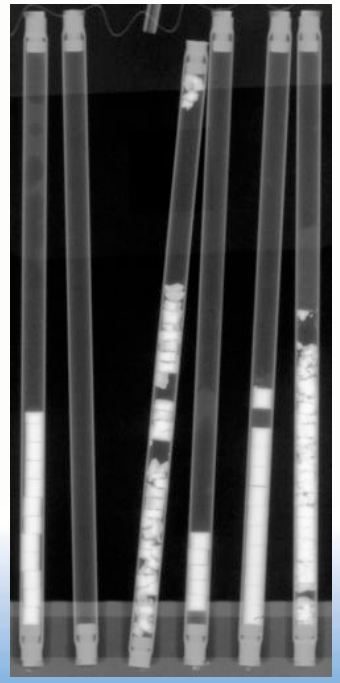


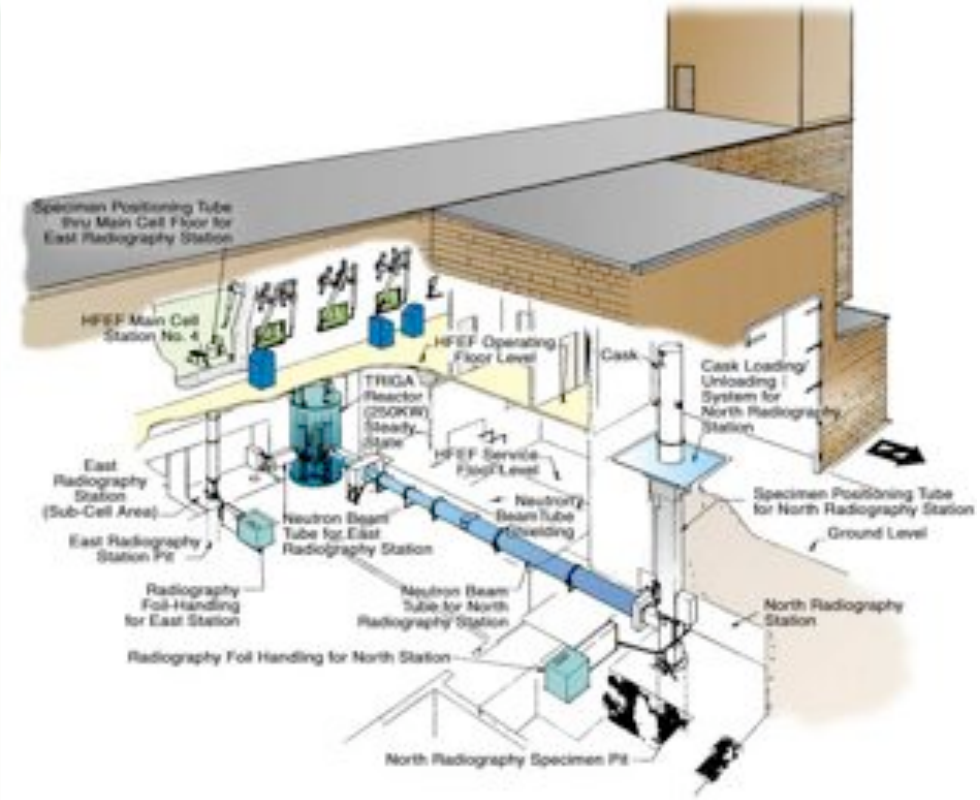


Neutron Radiography

- Purpose: **Non-destructively interrogate internals**
- Application:
  - **Evaluate fuel integrity and movement**
  - **Hydriding in LWR cladding**

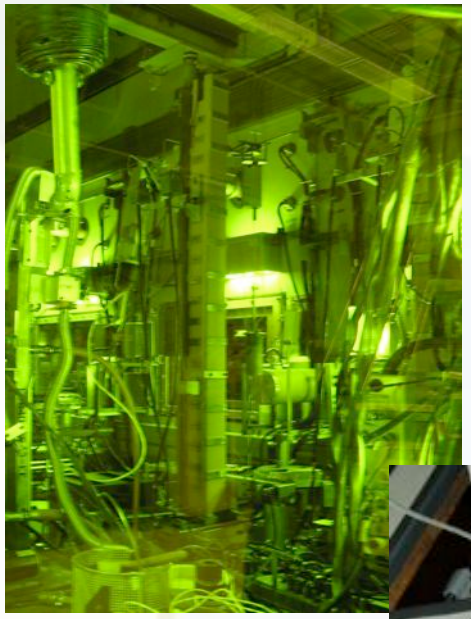


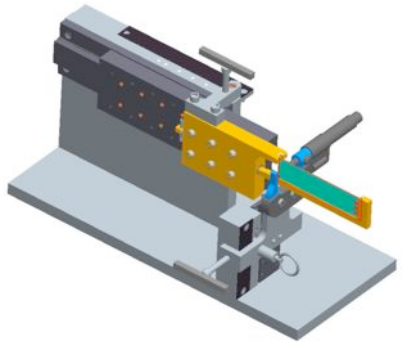


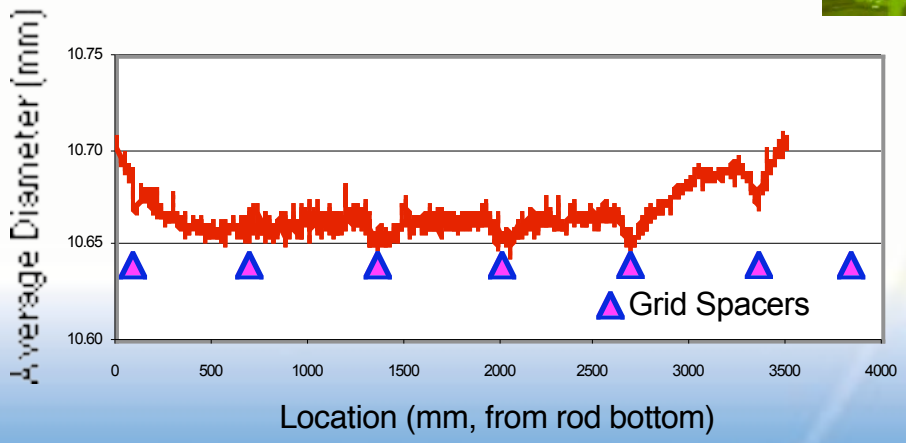


Dimensional Inspection

- Purpose: **Measure diameter / plate thickness and bow and length**
- Application:
  - **Cladding creep down in-reactor service and creep out during dry storage**
  - **Irradiation induced swelling**
  - **Fuel rod growth**
- Description:
  - **Element contact profilometer: diam. ±0.0002 in. (±0.0051 mm)**
  - **Bow and Length: ±0.02 in. (±0.51 mm)**

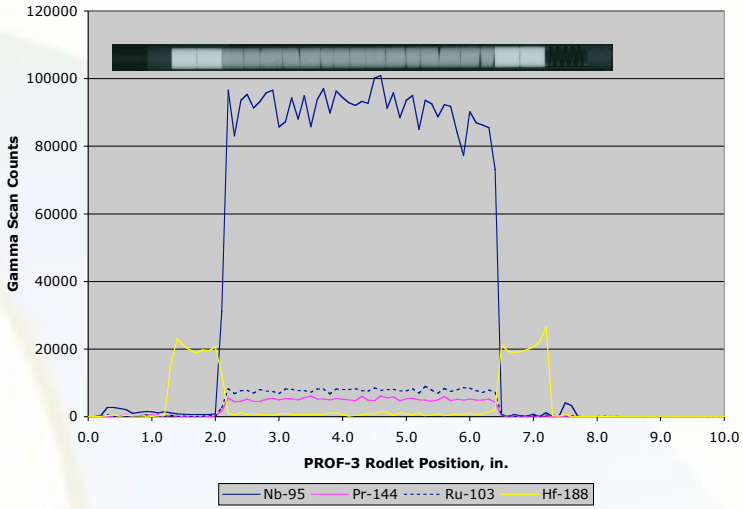


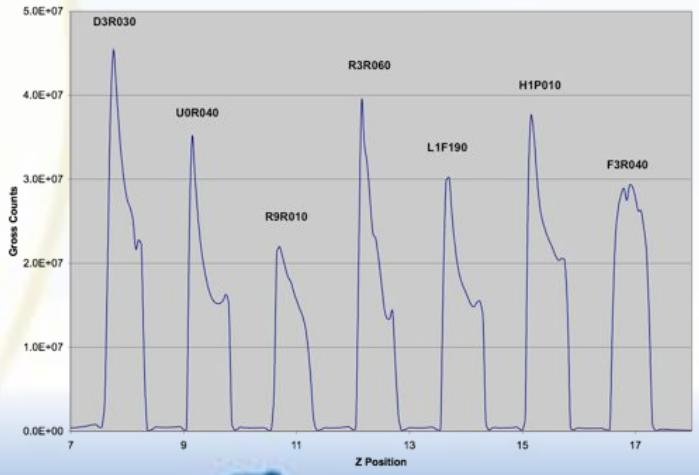




Gamma Scan Analysis

- Purpose: **Measure gross and isotopic profile of irradiated fuels**
- Application:
  - **Relative fuel burnup profile**
  - **Pellet-pellet interfaces**
  - **Relative distribution of various isotopes of interest in fuel**
- Description:
  - **Basically an Energy Dispersive X-ray detector (EDS)**
  - **Multi-channel Analyzer w/ software Library**
  - **Vertical Step Travel Limit 0.01 in./step (0.05 - 0.1 in. typical)**





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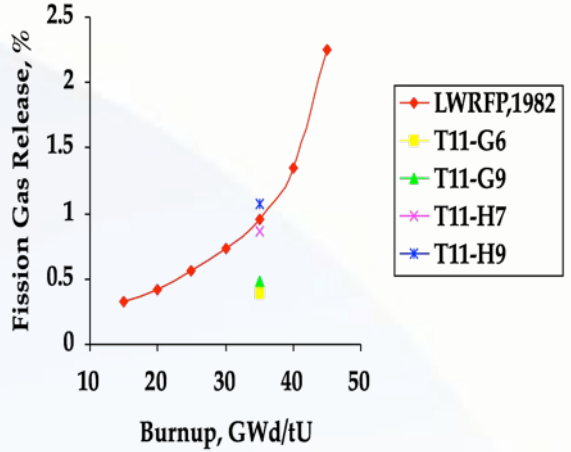
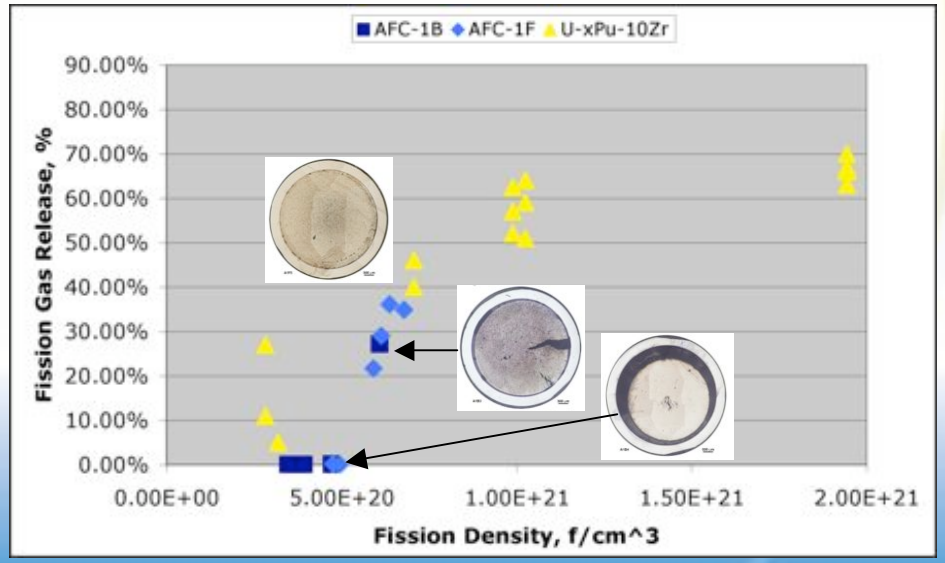
Fuel Fission Gas Puncture & Analysis

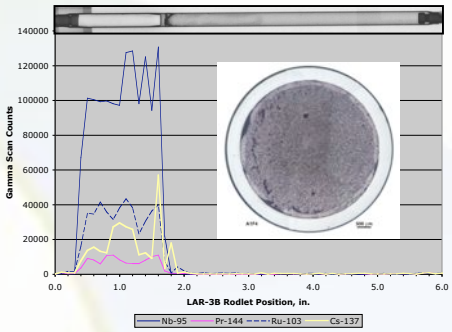
- Purpose: **Puncture fuel rod and analyze fission gas pressure/ internal void volume and chemical/isotopics**
- Application:
  - **Determine fission gas and helium release**
- Description:
  - **Laser puncture system**
  - **Fuel rod internal void volume and gas pressure, ±5%**
  - **Plenum gas batch sample chemical analysis**

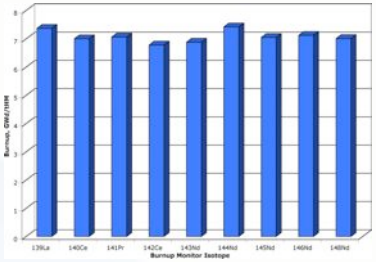


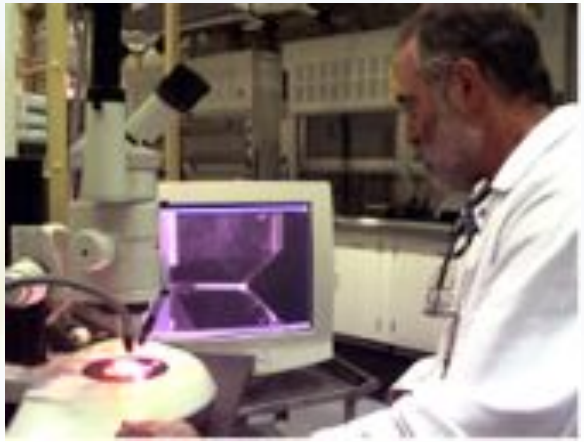


Destructive Examinations

- Isotopic & Burnup Analysis
- Metallography
- Microgamma scan Analysis
- Scanning Electron Microscopy
- Transmission Electron Microscopy
- Fuel annealing Furnace
- Mechanical Properties
- Physical properties
  - Density
  - Thermal conductivity
  - Cladding Hydrogen Analysis

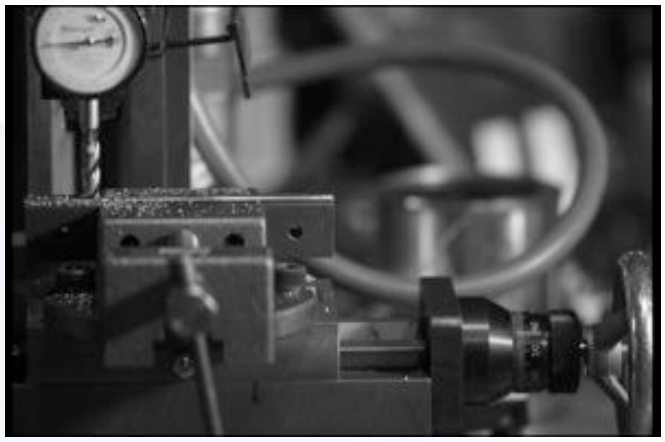






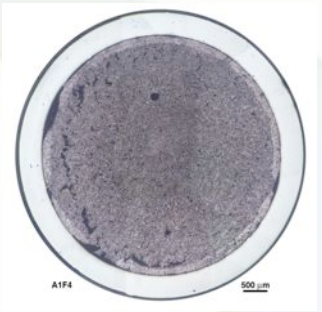
In-cell Machining

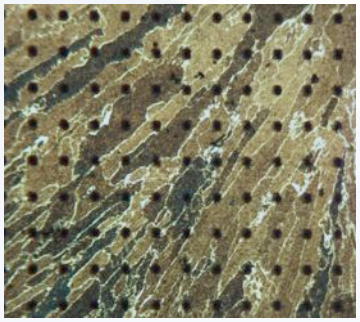
- Small, 3 axis mill
  - **Currently used for disassembly of experiments**
  - **Up to 6 inches of travel in each direction**
  - **Tolerances to 0.001”**
- CNC mill
  - **40 inches in x-direction and 8 inches in y-z directions**
  - **Tolerances to 0.003”**
- Electro Discharge Machine
  - **6 inches in x-y directions, 5 inches in z-direction**
  - **Tolerances to 0.001”**
  - **Resolving some performance issues**
  - **To be installed at INL in 2010**

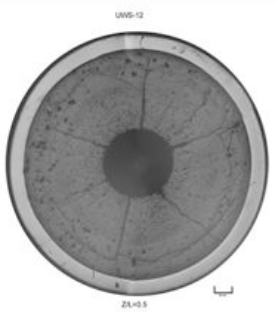


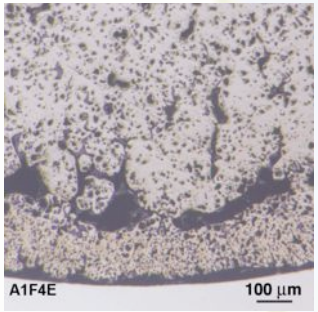
Metallography / Ceramography

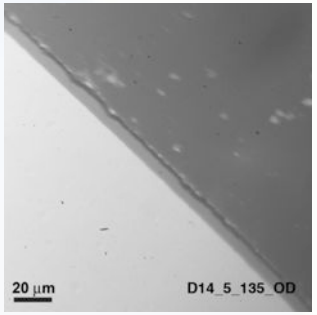
- Purpose: **Characterize microstructure and micromechanical properties of irradiated fuels and materials**
- Application:
  - **Characterize irradiated fuel grain size and morphology, porosity, phase, fuel-cladding interaction**
  - **Measure cladding oxide thickness, hydride distribution**
- Description
  - **Leitz MM5 RT Metallograph (80X to 800X)**
  - **Microindenter Hardness Tester**
  - **Automatic stage control**
  - **Integrated data collection and analysis**
  - **Digital Still Photographic Image**

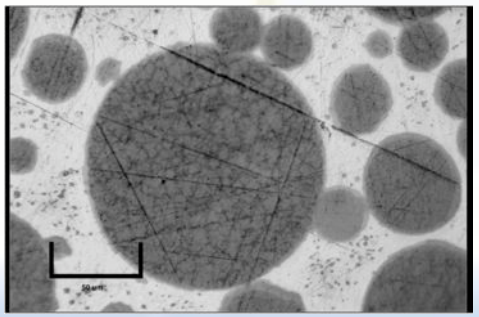


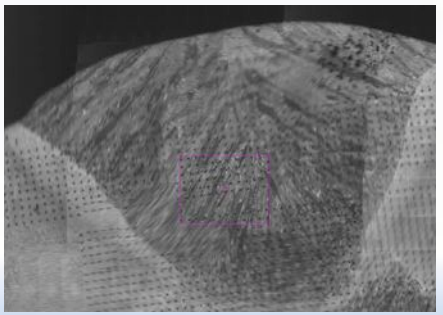












Isotopic and Burnup Analysis

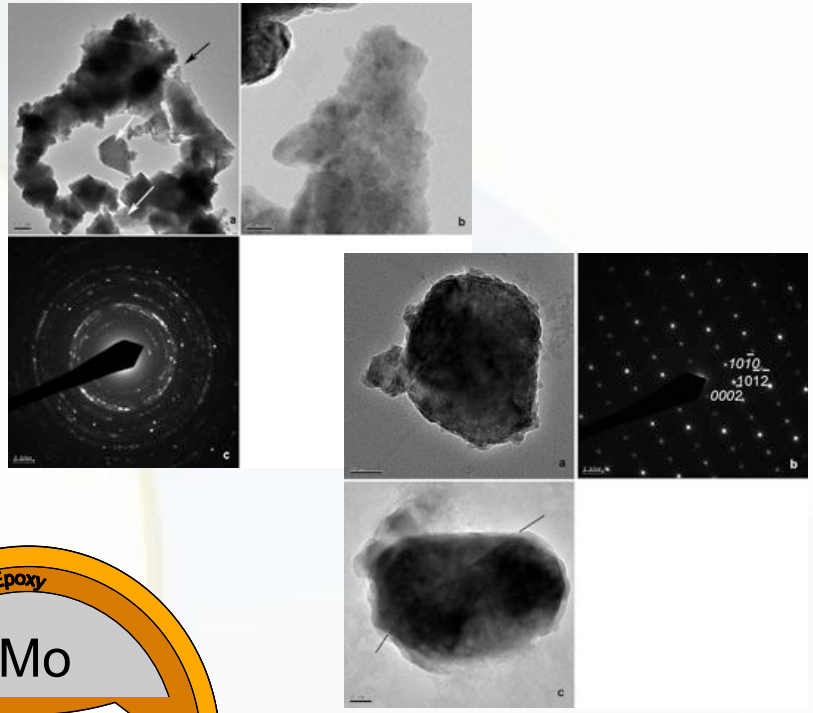
- Purpose: **Measure bulk isotopic and chemical composition of actinide fuel samples as-fabricated and postirradiation.**
- Application:
  - **Perform nuclear material accountability measurements by Thermal Ionization Mass Spectrometry (TIMS) isotope dilution**
  - **Inductively Coupled Plasma Mass Spectrometry-Dynamic Reaction Cell (ICPMS-DRC) to mitigate isobaric interferences and obviate chemical separation**
  - **ICP-Optical Emission Spectrometry**
  - **Derive burnup of metallic, oxide, nitride, carbide and dispersion fuel forms.**
- Description:
  - **U, Pu isotopics: <±1.0%**
  - **Fission product isotopes: ±2%**
  - **Elemental analysis: ±2-5%**
  - **NIST traceable standards**

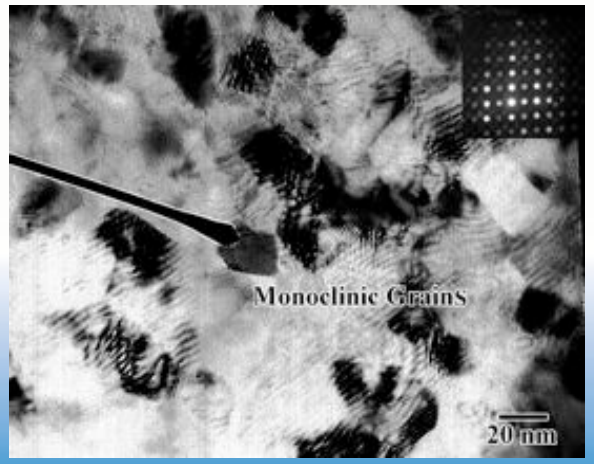
Scanning Electron Microscopy

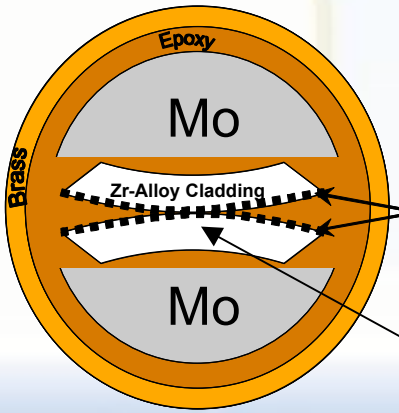
- Purpose: **Characterize as-fabricated microstructure, phases and homogeneity of actinide fuel forms**
- Application:
  - **Determine composition by energy dispersive spectroscopy and wavelength dispersive spectroscoy**
  - **Identify phases using crystal structure information by electron backscatter diffraction**
  - **Study fuel cladding chemical interaction**
- Description:
  - **JEOL 7000F SEM with EDS, WDS, EBSD detectors.**
  - **Mag: 200,000x (4 nm resolution)**
  - **Probe current: 200 nA**
  - **Airlock for rapid sample exchange**
  - **Infrared chamber scope for observing detector and sample positions in the microscope**
  - **Software: User input standards**

Transmission Electron Microscopy

- Purpose: **Characterize nanometer-scale microstructure, phases and homogeneity of actinide fuel forms**
- Application:
  - **Characterize small scale structural features: precipitates, dislocation loops**
  - **Identify crystal structure information Determine composition by energy dispersive spectroscopy**
- Description:
  - **JEOL 2010F TEM with EDS and STEM**
  - **Resolution: 0.23 nm**
  - **Double tilt high temp holder: 800˚C**
  - **Digital camera: 2k x 2k**







Oxide layer analyzed

Desired location for hole to form during ion milling

3 mm Disc

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Fuel annealing furnace for fission gas release studies

ATR National Scientific User Facility

- Purpose: **Measure temperature-driven release of condensable fission products and fission gases from irradiated fuel**
- Application:
  - **Heat irradiated fuel in helium sweep gas (T ≤ 2000°C)**
  - **Condense fission products during annealing on water-cooled cold plate for subsequent measurement**
  - **Collect and measure released fission gases (Kr, Xe) in cryo traps**
- Description:
  - **Max temp: 2000°C**
  - **Graphite heating element**
  - **Helium atmosphere (@ ambient pressure)**
  - **Hot zone: ~ 3.25" diameter x ~6" high**
  - **Computer controlled operation**
  - **Automated cold plate exchange**

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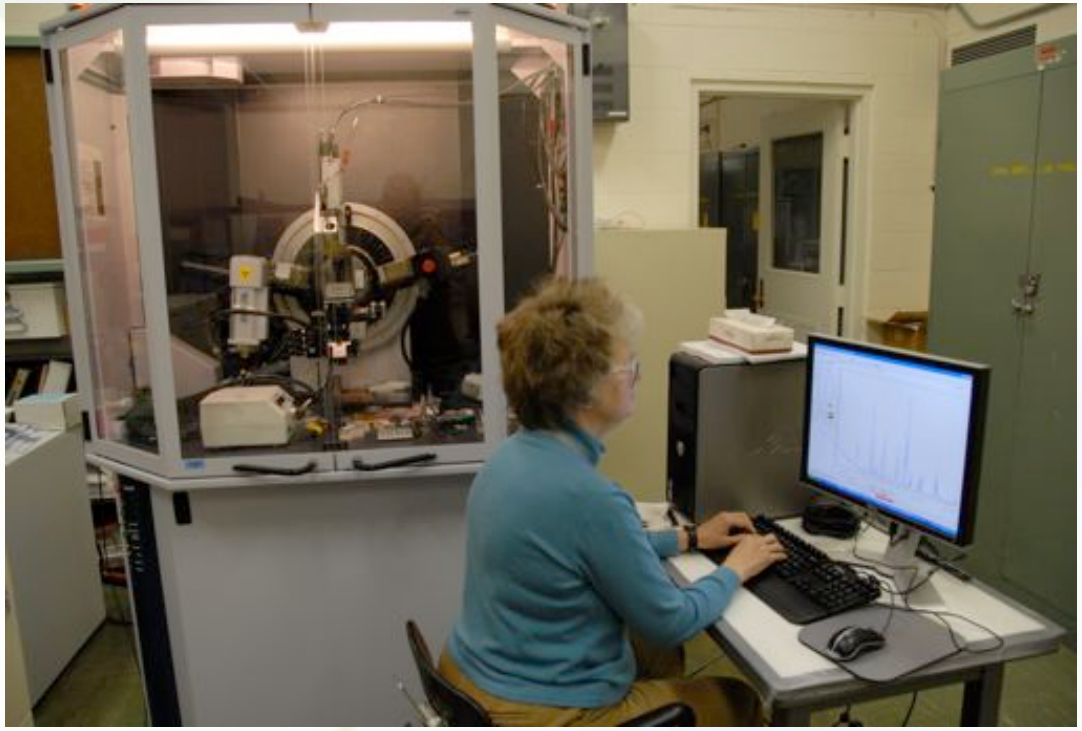
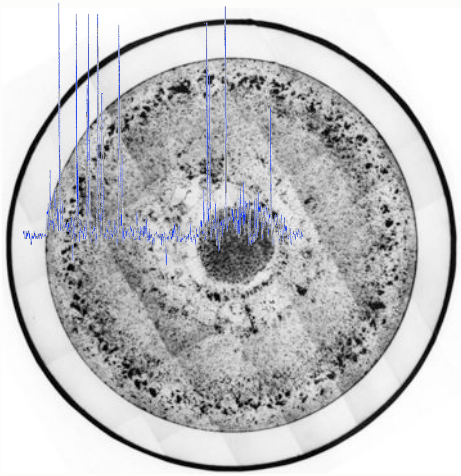
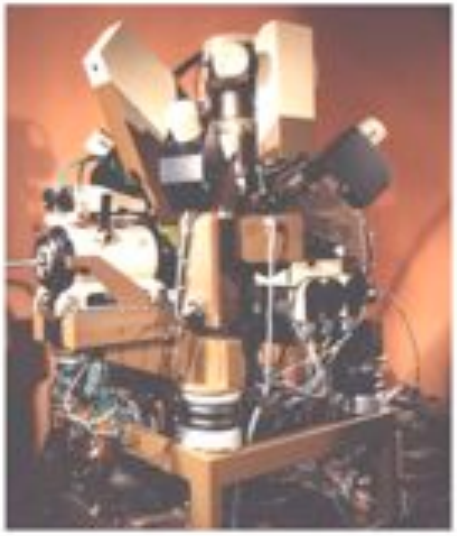
Micro X-ray Diffraction

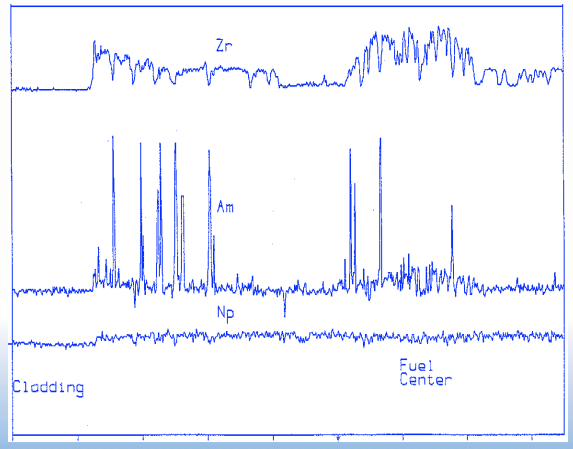
- Bruker D-8 Discoverer
- LynxEye or scintillation detectors
- Post specimen graphite monochromator
- Microdiffraction with 100 μm spot size
- Bragg-Brentano mode
- Pole figure capability



Shielded Electron Probe MicroAnalyzer



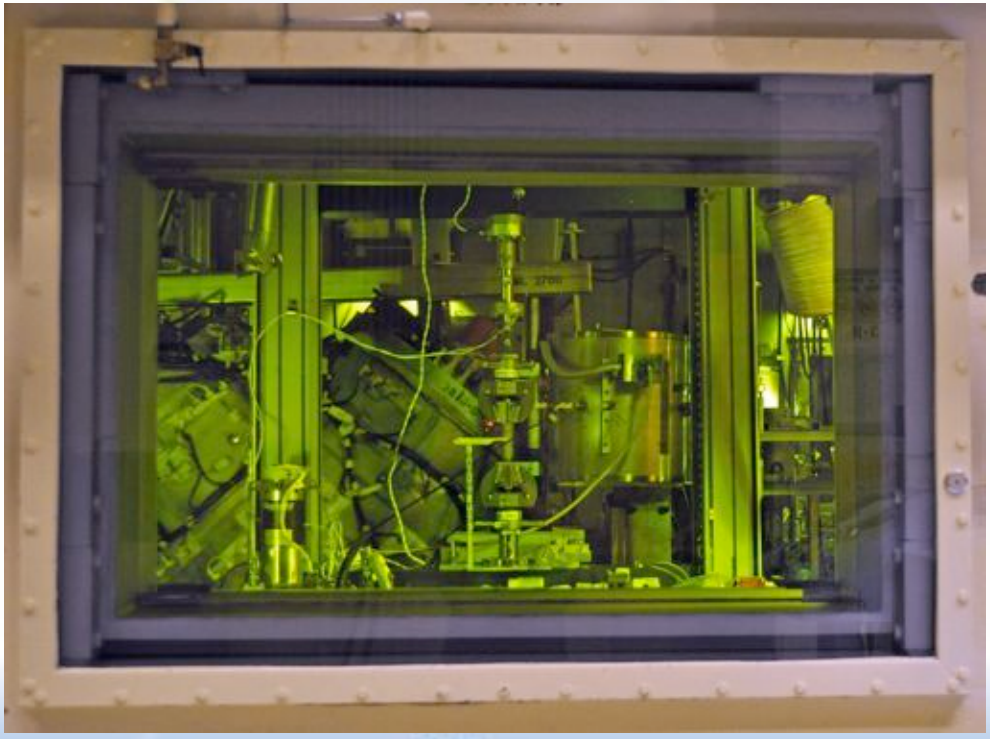




- Purpose: **Measure the localized micro-scale chemical composition of whole transverse cross-sections of irradiated fuels and materials and perform electron imaging of these samples.**
- Application:
  - **Characterize compositional homogeneity of as-fabricated actinide-bearing transmutation fuels**
  - **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**
- Description:
  - **4 WDS spectrometers (4 crystals)**
  - **SE detector for imaging**
  - **Be to actinides sensitivity: 20-100 ppm**
  - **Software: User input data.**

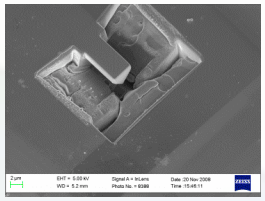
Hot Cell Tensile Testing

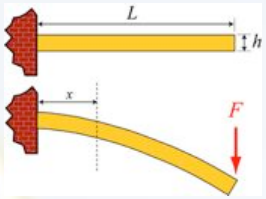
- 50 kN Instron tensile testing system installed in HFEF and qualified
- Provides engineering data on ASTM standard specimens



FY 2010 - FY12: New PIE Capabilities

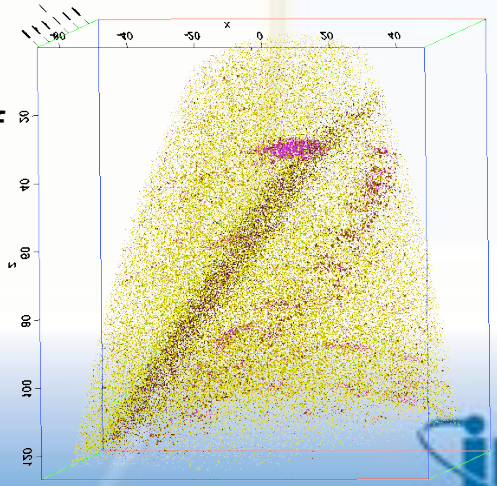
- **Mechanical properties**
  - Nanoindenter/AFM
  - Small sample mechanical testing
- **Analysis at micro- and nano-scale**
  - FEG-STEM-chemical and structural analysis at the nano-scale
  - Atom Probe- maps single atoms in 3 dimensions (few in world available for use on radioactive material, none on fuel)
  - Raman spectroscopy-light scattering to understand atomic structure
  - SEM hot stage-real time analysis of changes in structure due to temperature and strain
- **Sample Preparation**
  - Dual-beam FIB #2

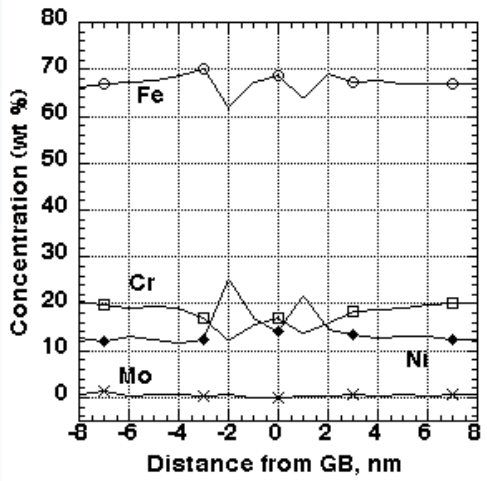




Nano-indenter, in combination with the FIB, can perform mechanical analysis on the nanometer scale

FEG-STEM can perform chemical analysis on the nanometer scale

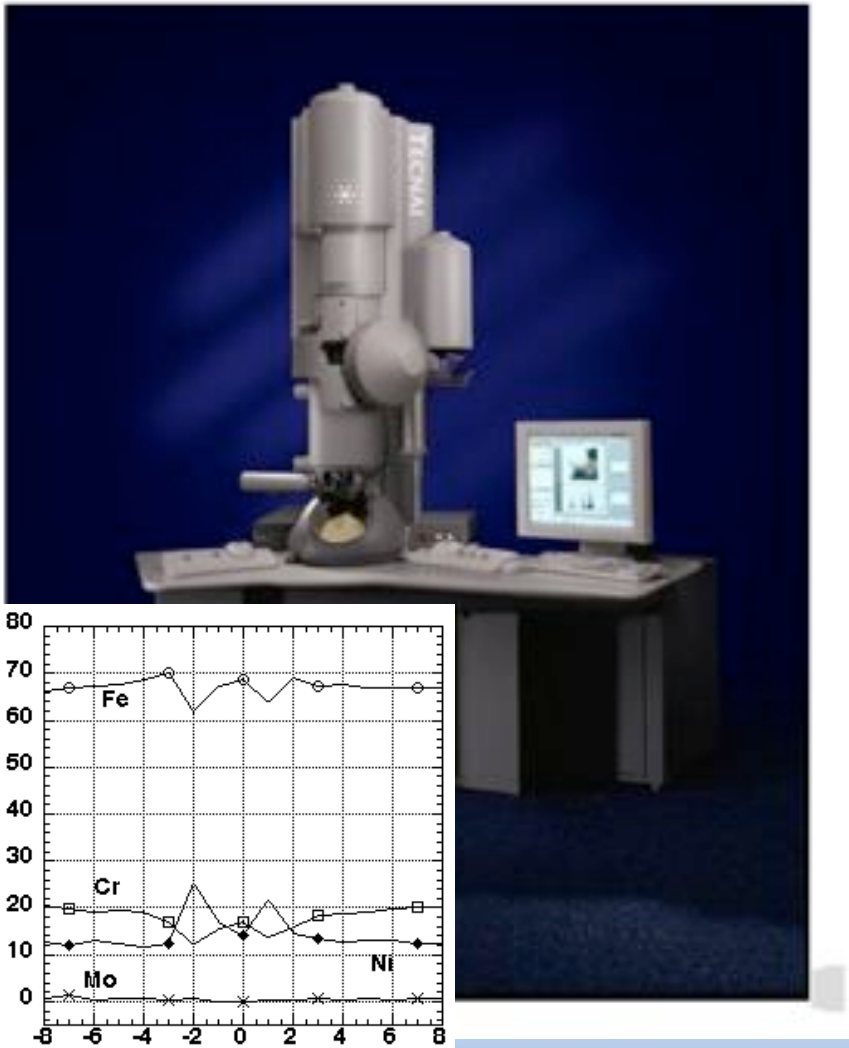




Atom Probe maps material composition atom-by-atom in 3D

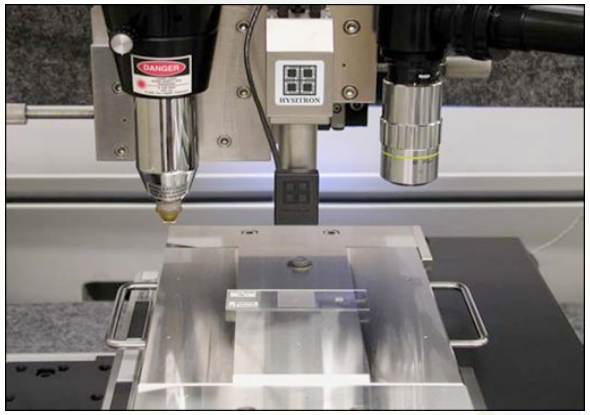
FEG-STEM

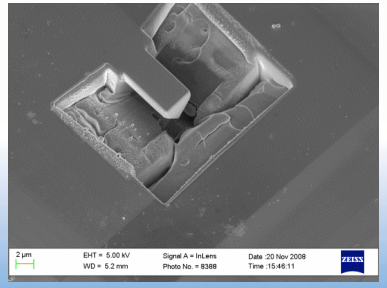
- FEI Tecnai F30 S-TWIN FEG-STEM
- Nanometer scale chemical and structural analysis
- 300 KeV (allows easier fuel sample preparation)
- Gatan Tridiem 863 P GIF
- HAADF detector
- EDS
- Tomography holder
- Delivery in September 2010
- Installation in CAES
- Will be moved to IMCL in 2012

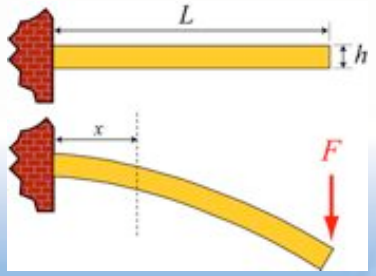


Nanoindenter

- Hysitron TI-950
- 400°C hot stage
- AFM head
- MFM capability
- In-situ SPM imaging
- Dual heads for high and low loads(10mN -5N)
- Delivery July 2010
- Installation in CAES

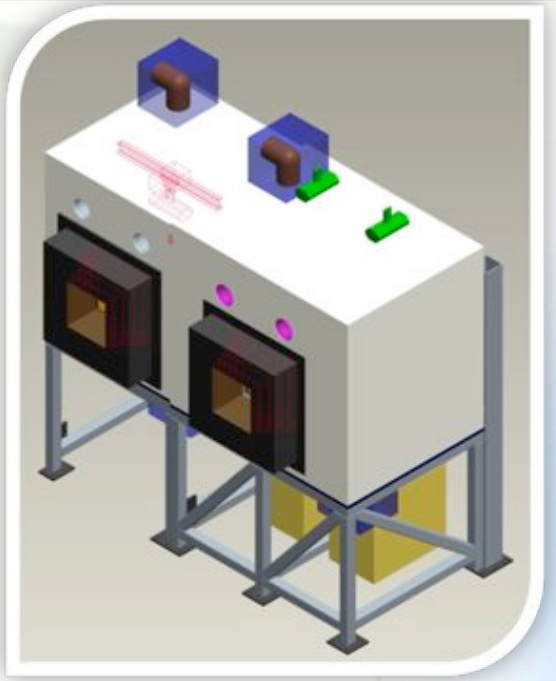


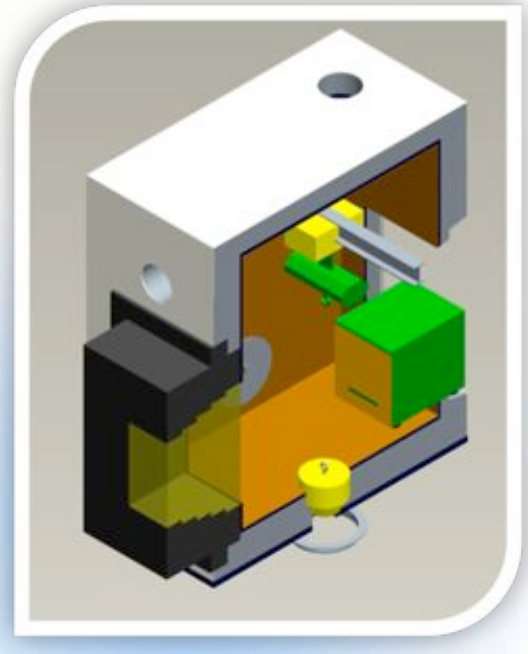


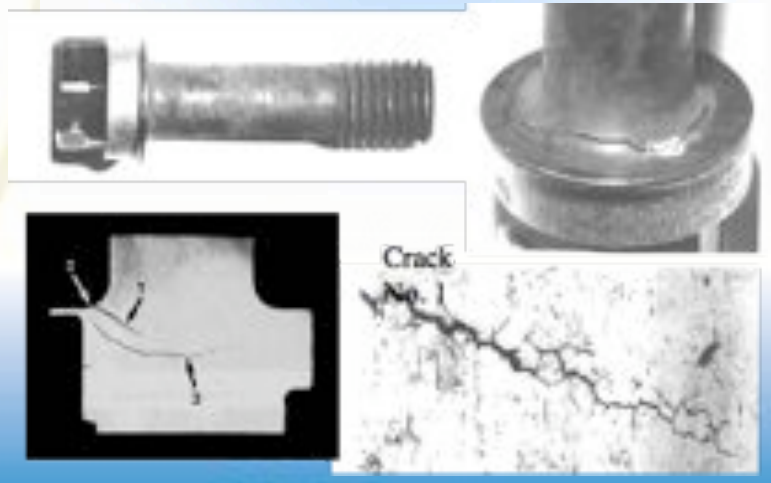


Irradiation Assisted Stress Corrosion Cracking

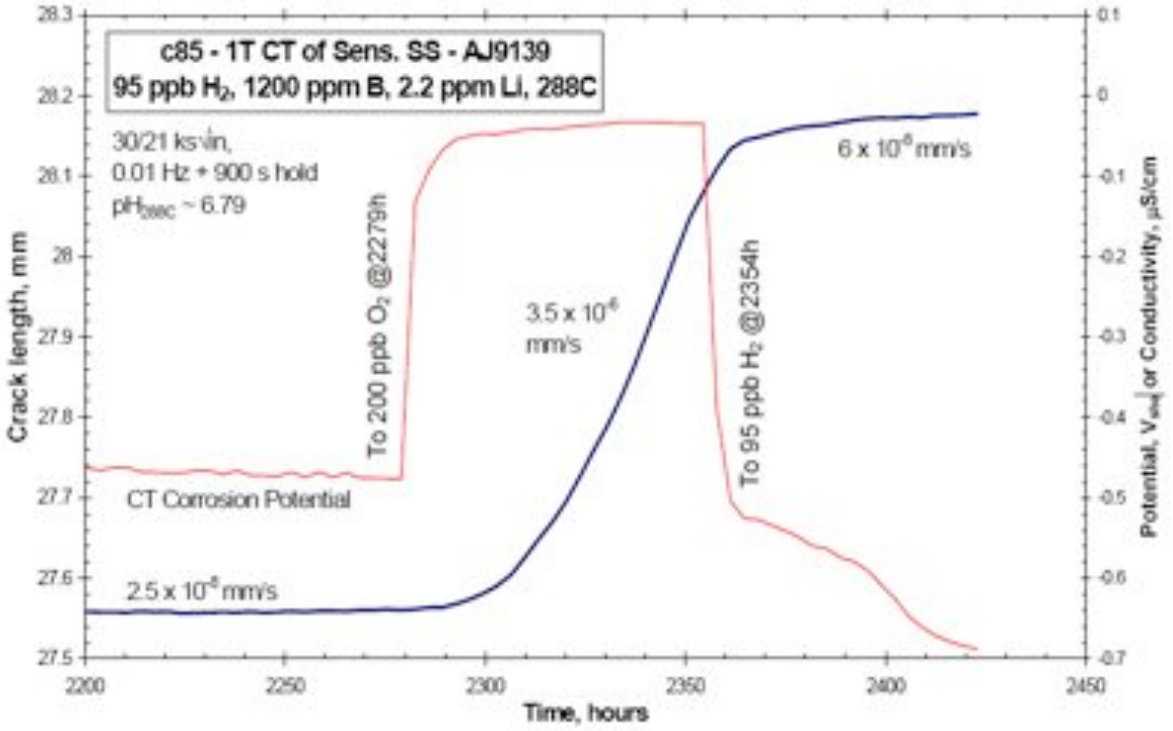
- IASCC Test Rigs
  - IASCC is a huge issue for LWRs
  - Rigs allow testing of material crack growth rates at BWR/PWR temperatures, pressures, and water chemistry on irradiated materials
  - Lead shielded cells (9 inches)
  - SEM in-cell
  - Servohydraulic testing systems (100 kN)
  - Activation in summer 2011

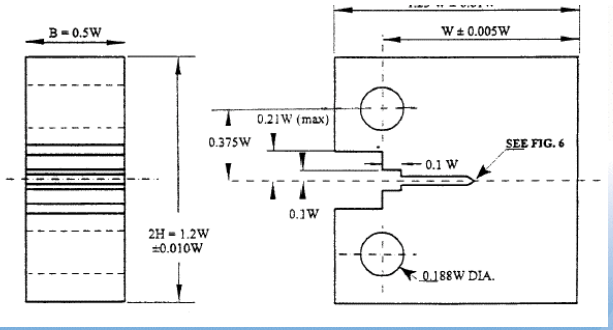






Stress Corrosion Crack Growth Rate





- IASCC systems measure crack growth rates and fracture toughness of material as a function of:
  - Irradiation dose (dpa)
  - Temperature
  - Water chemistry

Raman Spectroscopy

- Horiba LabRam HR
- Light scattering to understand atomic structure
- Free space stage allows installation of autoclave for in-situ analysis of corrosion under high pressure water
- Fiber optic port for potential in-cell installation
- Will be installed in Idaho Research Center laser lab for development

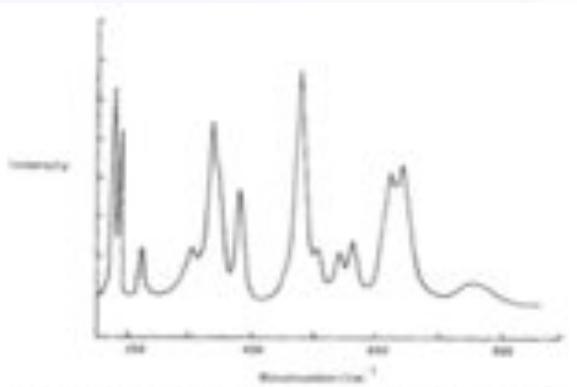


Figure 3. Remote Raman microprobe spectrum of Zircaloy-4 cladding oxidized in lithiated-boronated water at 360°C. Conditions as in Fig. 2.

Miniature Specimen Mechanical Property Testing

- 30 kN load frame (6750 lb) for tensile testing
-1200°C tube furnace

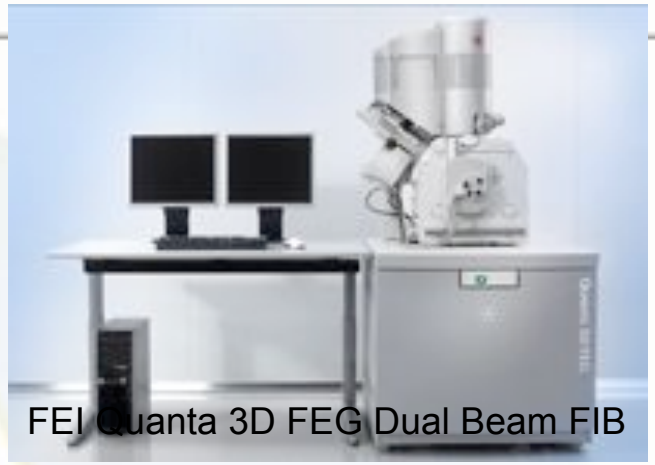
- 5 kN load frame (6750 lb) for shear punch testing

- MiniTower impact testing system
- Energies of up to 18 J (13.2 ft-lb)
- Velocities up to 3 m/s (9.8 ft/sec)

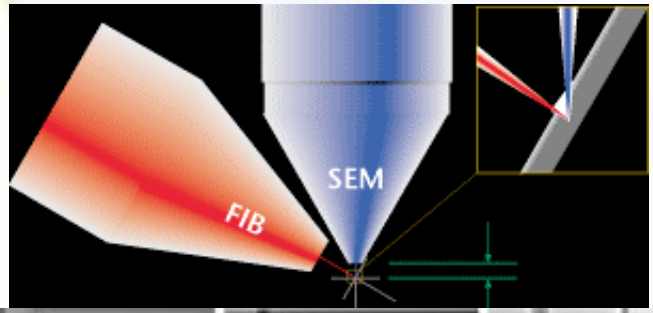
- Testing of small specimens can be done outside of a hot cell

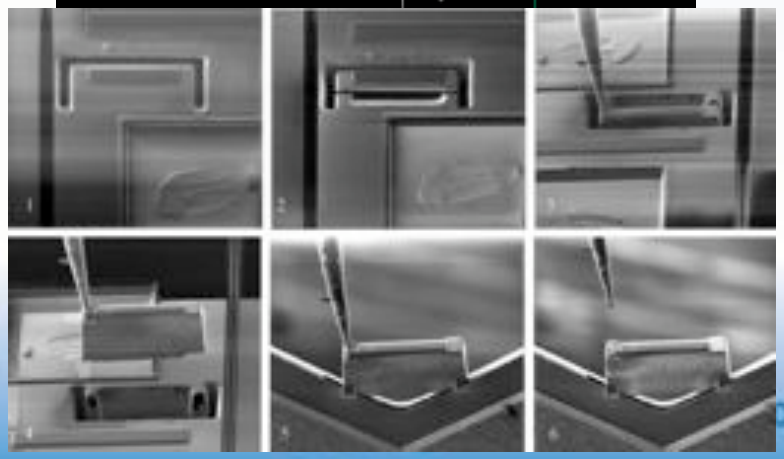
Dual Beam FIBs (2)

- FEI Quanta 3D FEG 600
- Analytical capabilities: (EDS [2], WDS [1] and EBSD[2])
- Sample Probe, Pt and C deposition
- Scanning transmission electron microscope (STEM) capability
- Environmental capability
- 3D image and chemical reconstruction of submicron features
- FIB #1
  - Currently set up in the Electron Microscopy Laboratory (EML) at MFC
  - Will be moved to IMCL once construction completed and used for work on specimens with high activity (fuels)
  - CSM Faculty Student team working techniques for fuel sample prep
- FIB #2 is will be installed in the Center for Advanced Energy Studies for use on lower activity specimens in May 2010



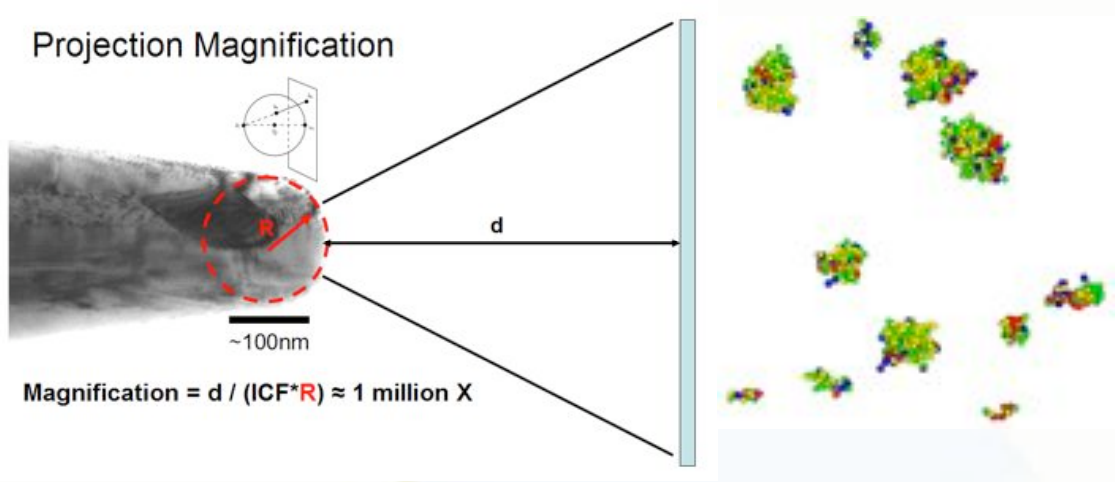
FEI Quanta 3D FEG Dual Beam FIB

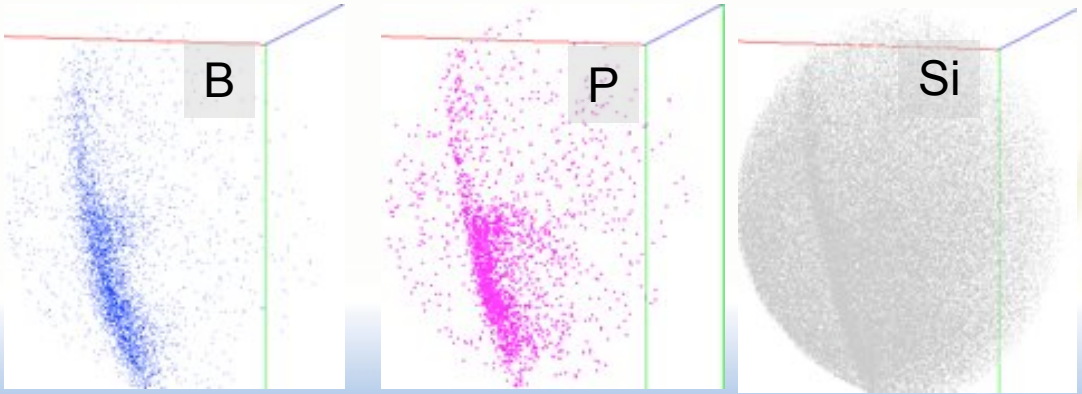




Atom Probe Tomography

- IMAGO 4000X HR
- Maps composition atom-by-atom in 3D
- Will be used on irradiated materials and fuels
- Located in CAES
- Delivery August 2010



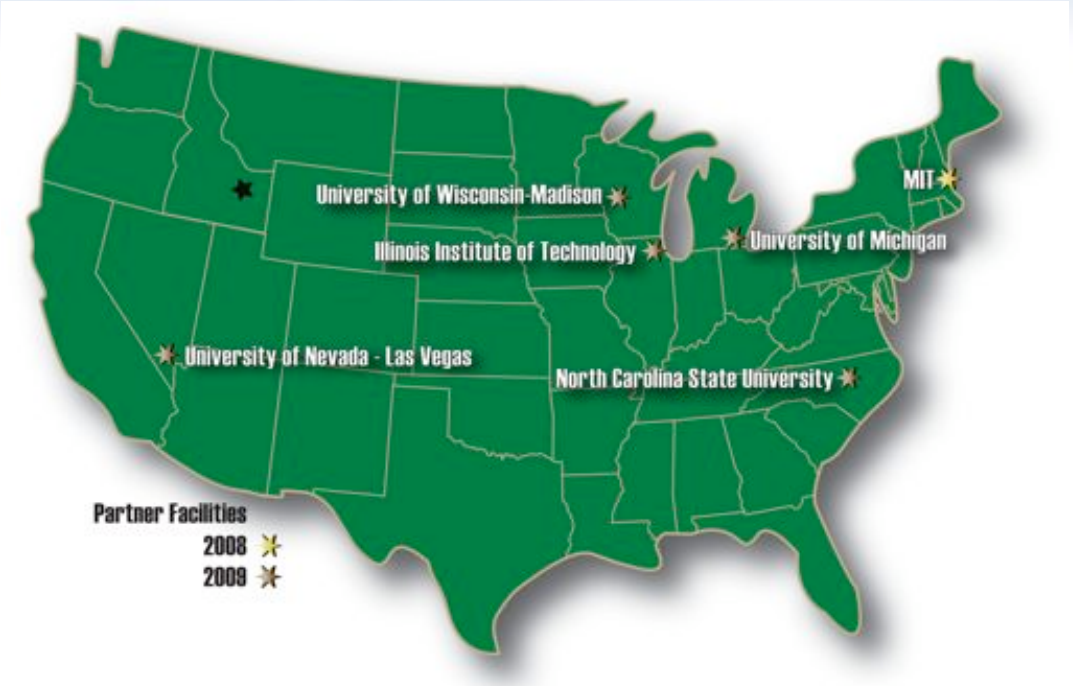


Alloy 600 grain boundary

Collaboration

- University Partner Facilities
  - MIT (MIT Reactor)
  - Illinois Institute of Technology (Advanced Photon Source)
  - University of Wisconsin (Characterization Lab for Irradiated Materials)
  - University of Michigan (Irradiated Materials Testing Lab)
  - NCSU (Pulstar Reactor)
  - UNLV (Electron Microscopy Laboratory)

  - **Collaboration with Oxford University**

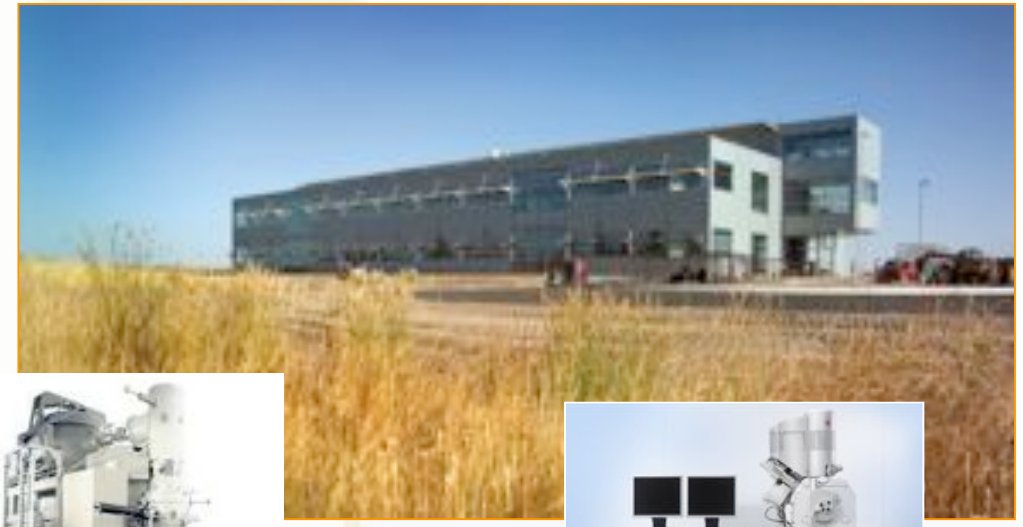


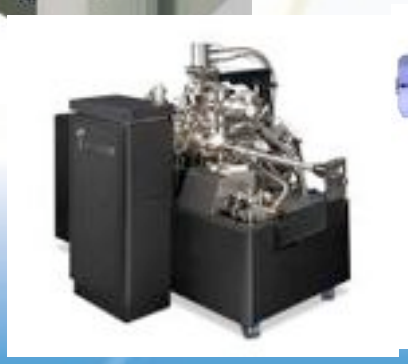
National User Facilities:

- **Current**
  - **Advanced Photon Source**
  - **SHaRE**
- **Possible: LANSCE, MAI, ORNL**

Center for Advanced Energy Studies (CAES)

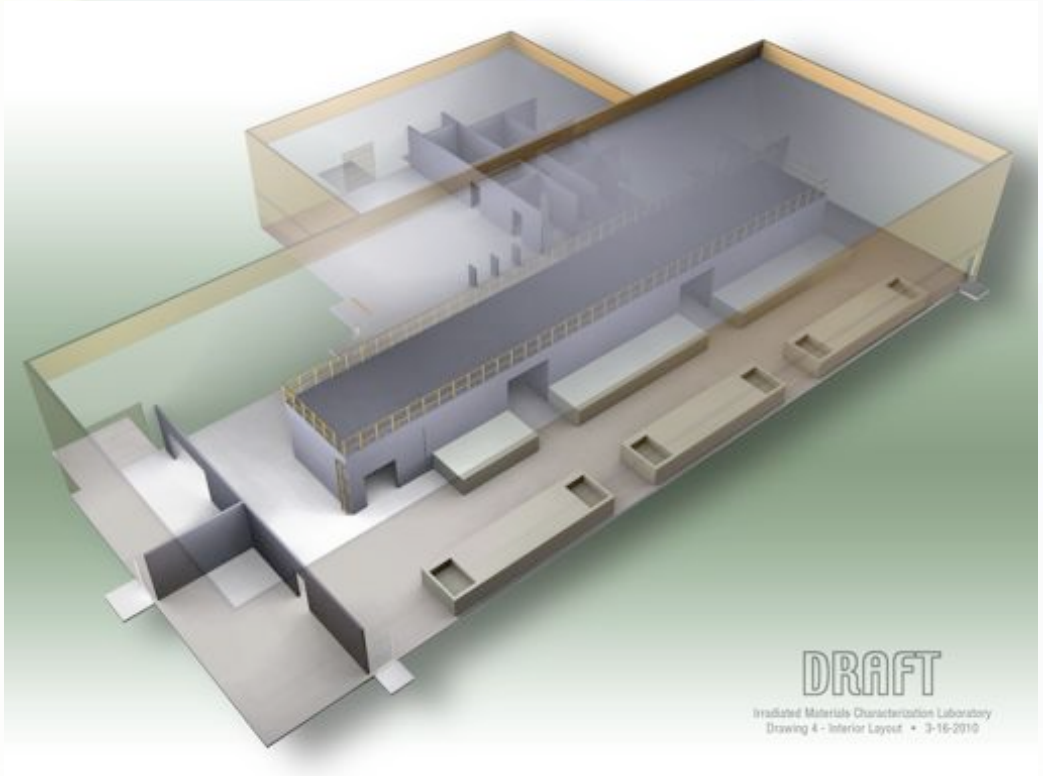
- CAES is a collaboration between INL and Idaho Universities
- CAES facility owned by State of Idaho
- CAES will house high-end equipment for use with lower activity samples
- Atom Probe, FIB, SEM, TEM, nano-indenter
- Small sample mechanical testing (30 kN Instron, 1000°C furnace, shear punch, drop tower)
- NRC licensed facility
- Material quantity limits sufficient for atom probe and TEM on fuel
- Outside the INL firewall, easier remote access





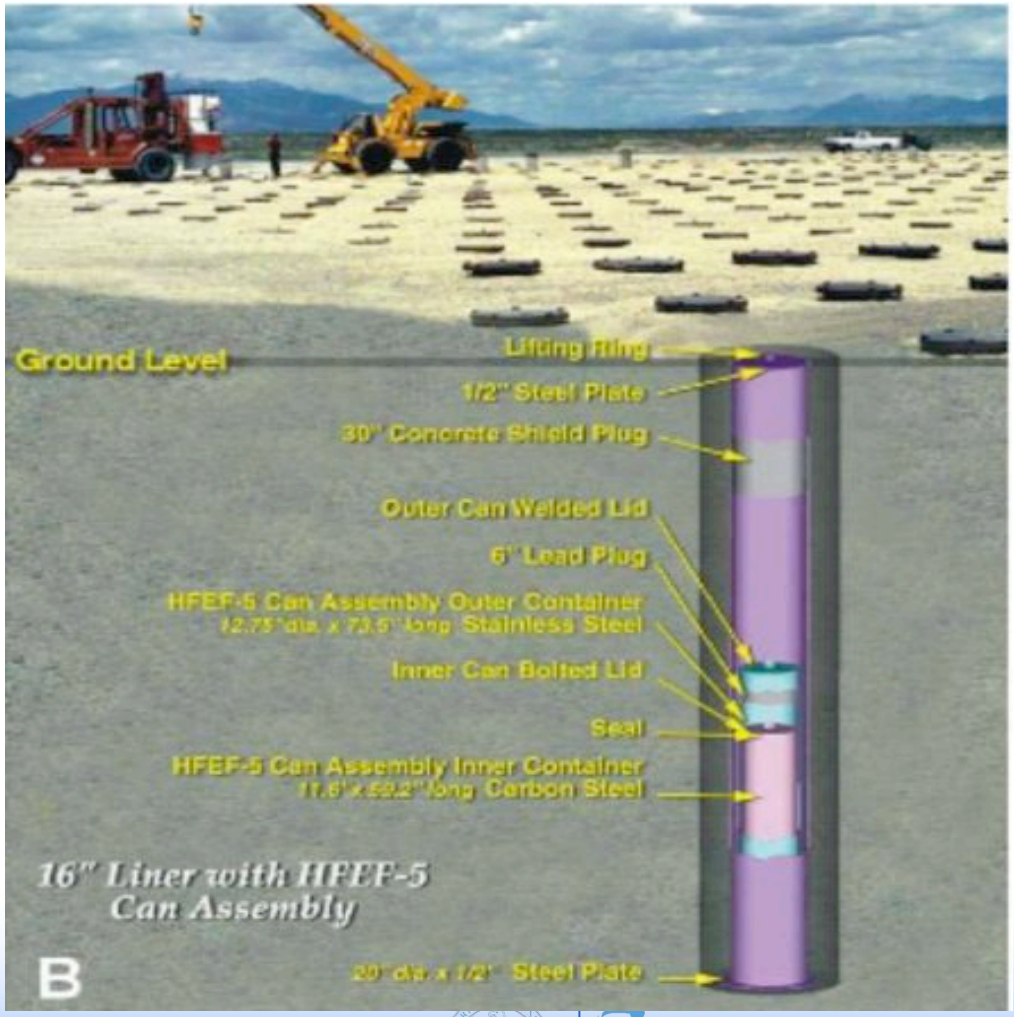
Irradiated Materials Characterization Laboratory

- Irradiated Materials Characterization Laboratory (IMCL) consolidates new post-irradiation analytical capability
- Shielded enclosures for handling multiple irradiated fuel samples
- Inert gloveboxes within enclosures and interfaced with instruments for contamination control
- Arrangement of shielded cells allows easy transfer between instruments
- Instruments can be serviced when radioactive material is removed
- EPMA, FIB, FEG-STEM, m-XRD, thermal analysis, mechanical testing, sample prep
- Occupancy in 2012



Waste Disposal

- Waste disposal is always an important consideration
- ‘Temporary’ disposal facility at MFC site



We're Here to Help

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