# NSUF Partner Facility Capabilities at the Pacific Northwest National Laboratory

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# Overview

MSTL

- PNNL's NSUF Partner Facility contributions include three broad capability areas
  - Irradiated fuel and high-activity structural materials post-irradiation examination and testing – Radiochemical Processing Laboratory (RPL)
  - Irradiated low-activity structural materials post-irradiation examination and testing – Materials Science and Technology Laboratory (MSTL)
  - Irradiation experiment design, analysis, and fabrication

RPL

# Receipt

- PNNL has recent experience with a number of US NRC licensed casks, including the NAC LWT, GE-2000, and PAS-1
- If activity levels permit, sample receipt in Type A shipping containers is very straightforward and far less expensive

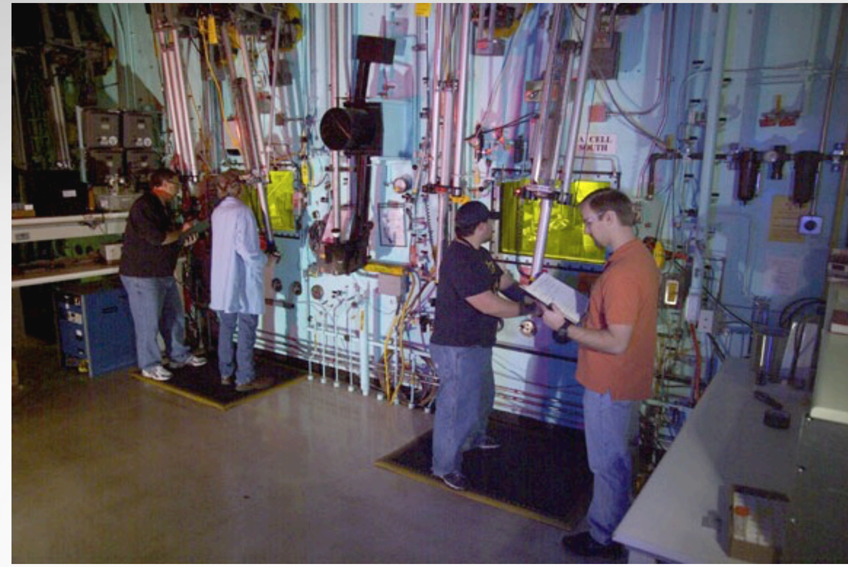
PAS-1

NAC LWT


GE-2000

# Cask Receipt and Unloading

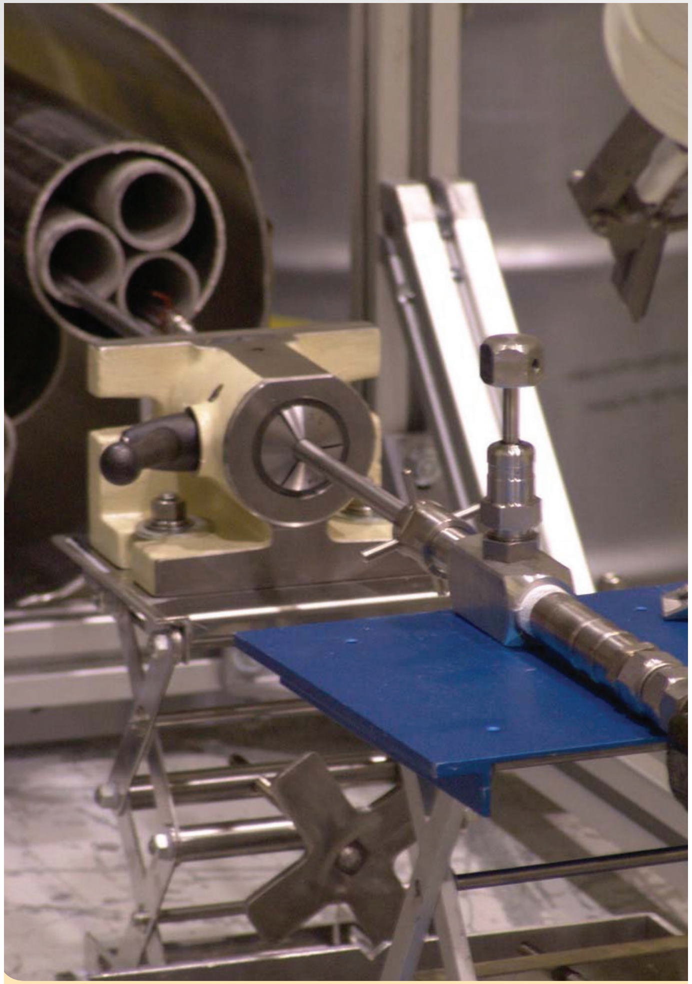
- Casks are typically received and unloaded at the RPL's High Level Radiochemistry Facility (HLRF)
  - High-density concrete shield walls (1.2 m thick)
  - Viewing provided by six lead-glass windows (total thickness 1.2 m) optically coupled with mineral oil
  - Each window has a pair of heavy-duty Model E manipulators
  - A-cell – Used for receiving – 4.5 m wide x 2.6 m deep x 5.2 m tall
  - Horizontal loading through 0.5 m diameter port
  - B-cell and C-cell – Used for size reduction – 1.8 m wide x 2.6 m x 5.2 m
  - All three cells interconnected for easy transfer



# Rod Puncture, Gas Analysis, and Gamma Spectroscopy



- Mechanical rod puncture
- Gas collection and online analysis system
  - Gamma energy analysis for radioactive gases
  - Mass spectrometry for non-radioactive gases
  - Can also quantify beta-emitting gases (e.g. tritium)
  - Total pressure determination
- Gamma spectroscopy with translating table
  - Axial resolution 2.5 mm
  - Maximum segment length 1.5 m



# Visual Examination



- During receipt or subsequent handling activities in HLRF, visual examinations are routinely conducted using high-resolution photography and videography
- Cameras mounted in-cell or positioned with manipulators

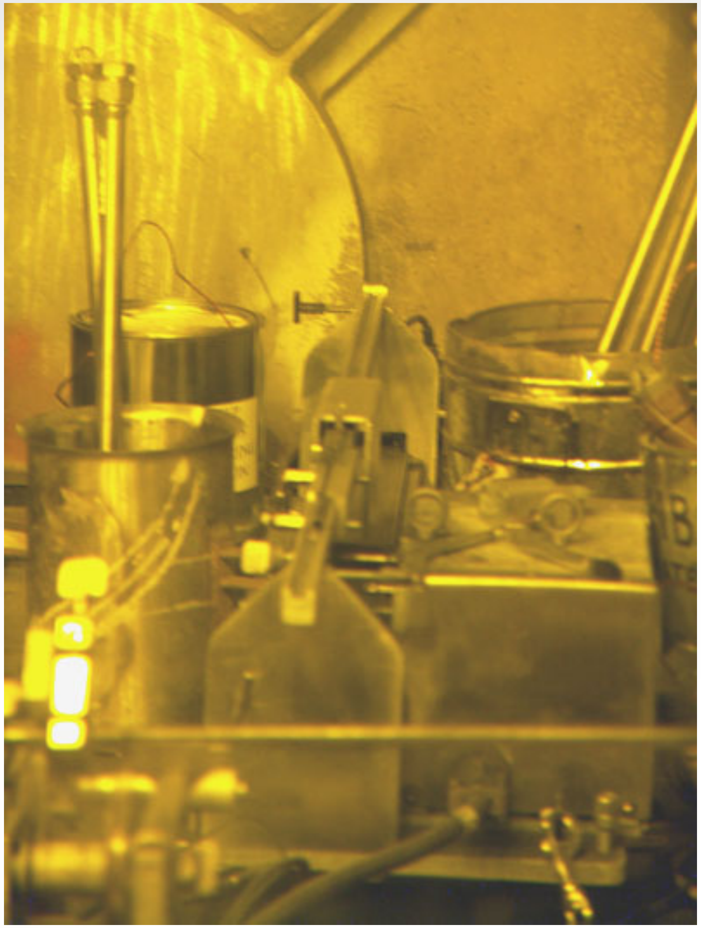
High-resolution in-cell photograph of failed fuel rodlet with centerline thermocouple


High-resolution video capture of a zircaloy-clad fuel rodlet with Pb-Bi eutectic on the exterior, held in manipulator fingers

# Initial Size Reduction

- Conducted in the HLRF hot cells
  - Cutting with low-speed metallographic saws or tubing/pipe cutters
  - Core drilling
  - Other similar size reduction operations with modified tabletop or hand tools

Cutting open a Ti capsule with modified pipe cutter



Longitudinal slitting of a tritium-producing burnable absorber rod

# Precision Sectioning

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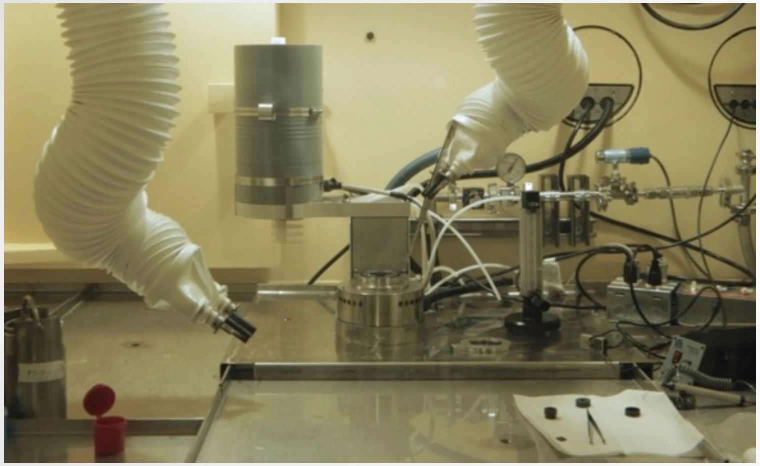
- Precision sectioning to extract samples is conducted in either the Shielded Analytical Laboratory or a modular hot cell, depending on activity and availability
- Shielded Analytical Laboratory (SAL) →
  - Six interconnected hot cells
  - 1.7 m wide x 1.7 m deep x 5.2 m tall
  - Shield walls are 1 m thick concrete and steel
- Modular hot cells
  - Two cells 1.7 m wide x 1.5 m deep x 3.7 m tall →
  - One cell 3.0 m wide x 1.5 m deep x 3.7 m tall
  - One cell 2.1 m wide x 1.5 m deep x 3.7 m tall
  - Shield walls are 30 cm steel
  - Large access doors for easy equipment installation/removal

# Thermal Properties



- Thermal property measurements are routinely conducted, with individual capabilities located in SAL and modular hot cells
- Thermal diffusivity ($\alpha$), specific heat ($c_p$), and density ($\rho$) data are combined to provide thermal conductivity ($k$)

$$k = \alpha c_p \rho$$



Thermal Diffusivity via Laser Flash Analysis
Netzsch LFA 457

Specific Heat via DSC
Perkin Elmer Pyris 1

Density via He Pycnometry
Micromeritics AccuPyc 1300

# Mechanical Properties



- Bulk tensile properties of high-activity materials obtained using a load frame installed in one of the modular hot cells
  - Instron 8800
  - 9800 N and 98,000 N load cells
  - Designed for miniature sample testing

- Hardness and Elastic Modulus
  - Nanosurf LensAFM attachment on a Nikon E400 POL microscope
  - Existing capability for topographic analysis
  - New capability being developed for micron-scale hardness and elastic modulus mapping

# Mechanical Properties



- Tensile properties of low-activity materials obtained using a load frame in a walk-in fume hood
  - Centorr 2500°C W-mesh furnace
- Fracture toughness of low-activity materials
  - Benchtop Instron 8801 servo-hydraulic load frame with 800°C tube furnace

# Optical and Scanning Electron Microscopy

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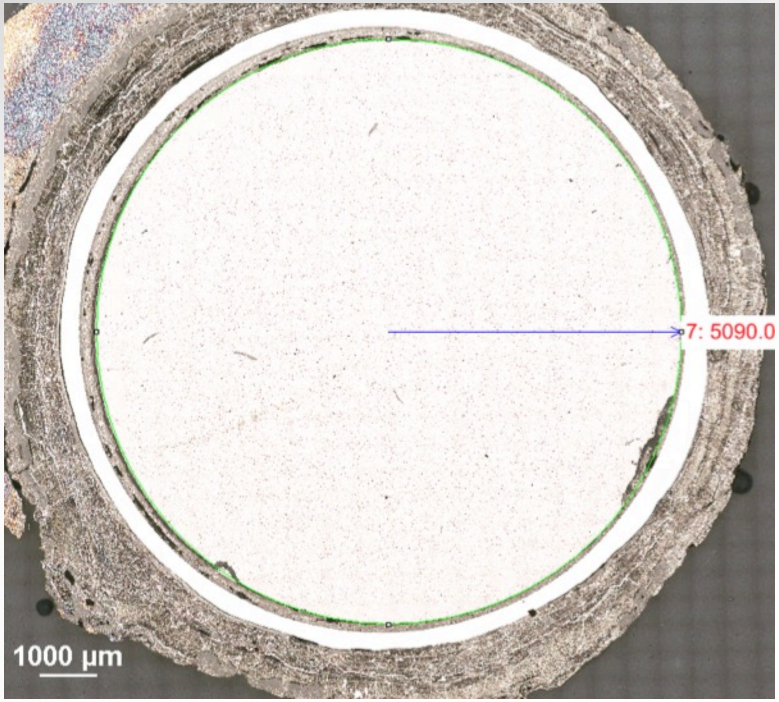
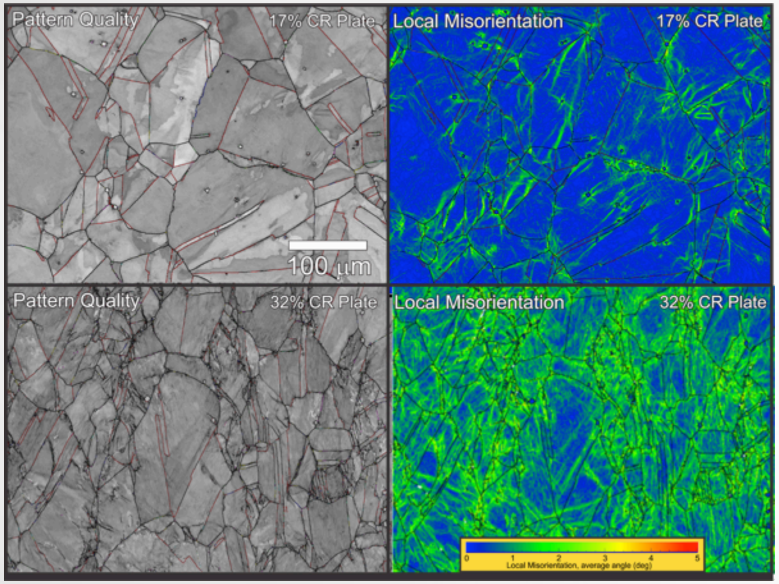
- Fully-automated and remote-operated Nikon 200MA optical microscope
- FEI Quanta250 FEG SEM with EDS/ WDS/EBSD
- JEOL 7600 SEM with EDS/WDS/EBSD
- Powder x-ray diffraction




Full-round UZrHx fuel rodlet showing fuel pellet, inner Pb-Bi bond, Zircaloy-4 cladding, and outer Pb-Bi bond. Mosaic composed of over 500 individual high-magnification images

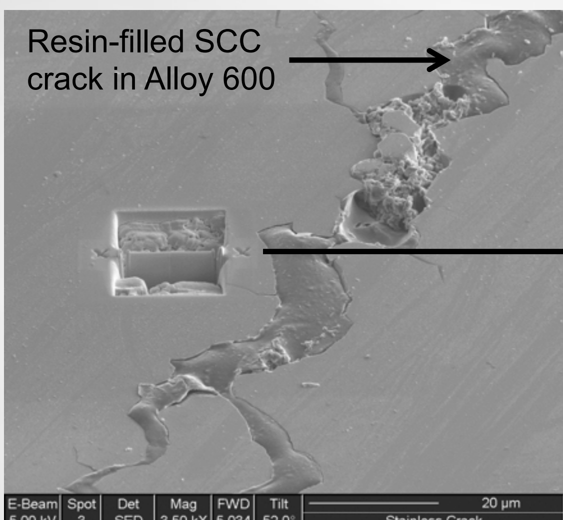


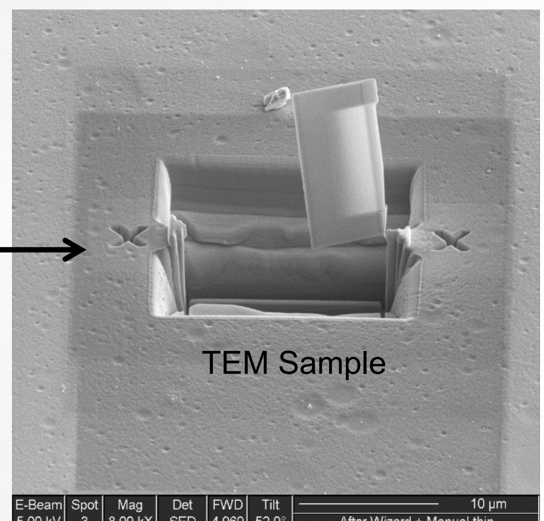

Using EBSD to image strain gradients and twins due to local misorientation in cold rolled Ni-base alloy

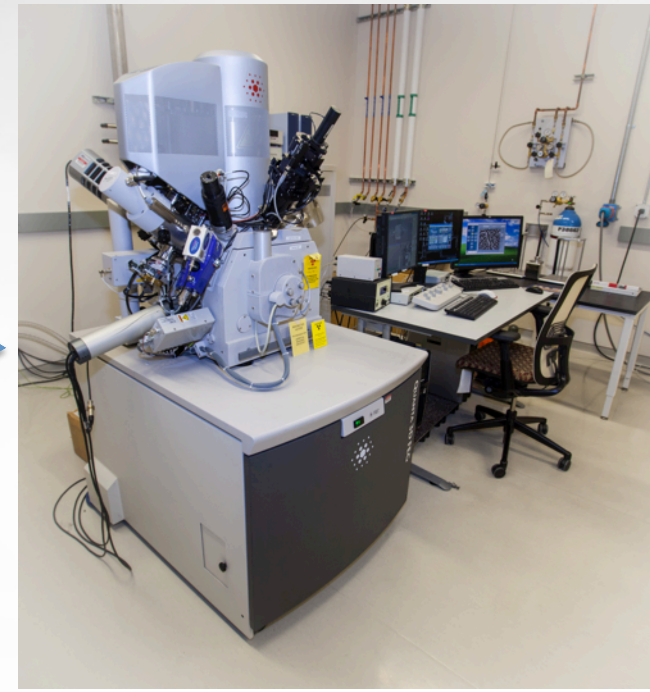
# SEM with Focused Ion Beam



- FEI Helios 660 Nanolab dual beam focused ion beam (FIB) SEM
  - For use on fuel, high-activity, and dispersible radioactive materials
- FEI Quanta 3D FEG dual beam FIB SEM
  - For use on low-activity, non-dispersible materials



# Transmission Electron Microscopy

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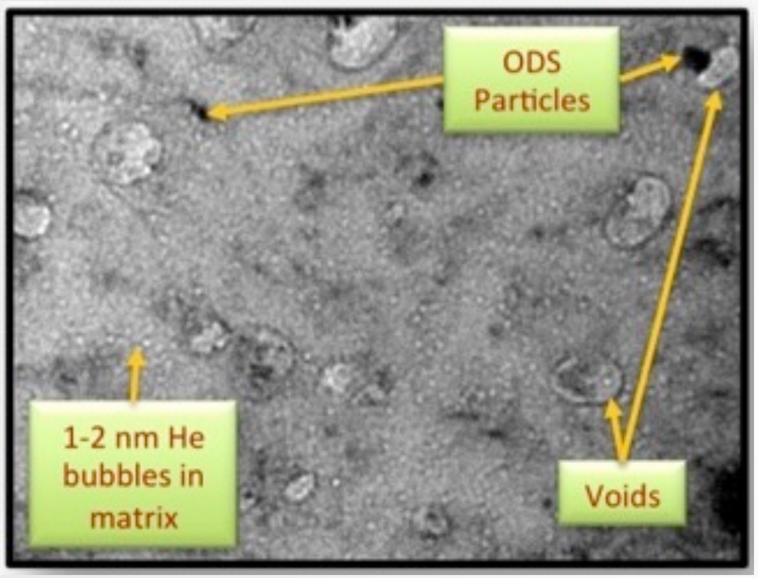
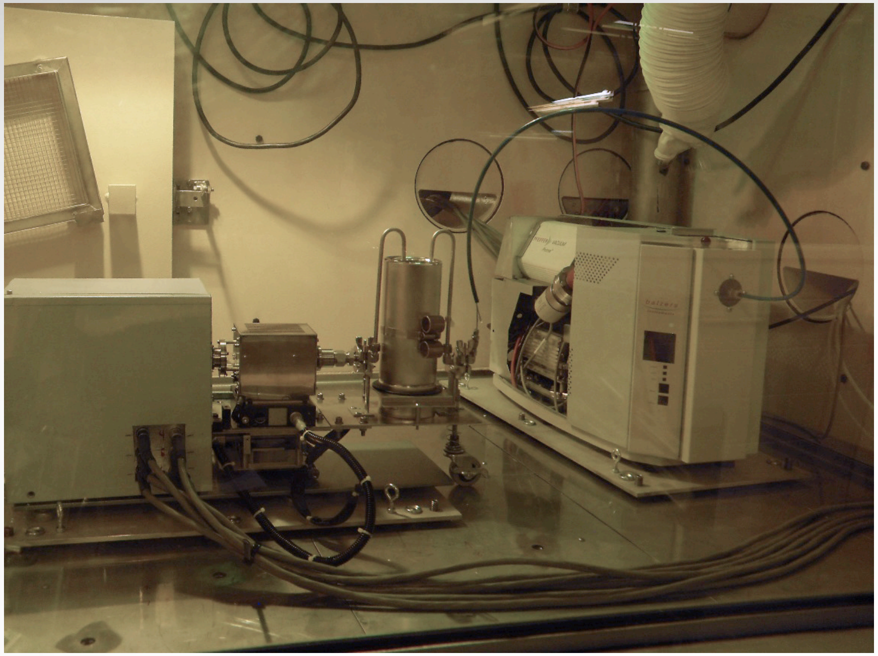
- FEI Tecnai 330 keV TEM with EDS and Gatan ORIUS digital camera
- JEOL ARM 200CF aberration-corrected TEM with EELS

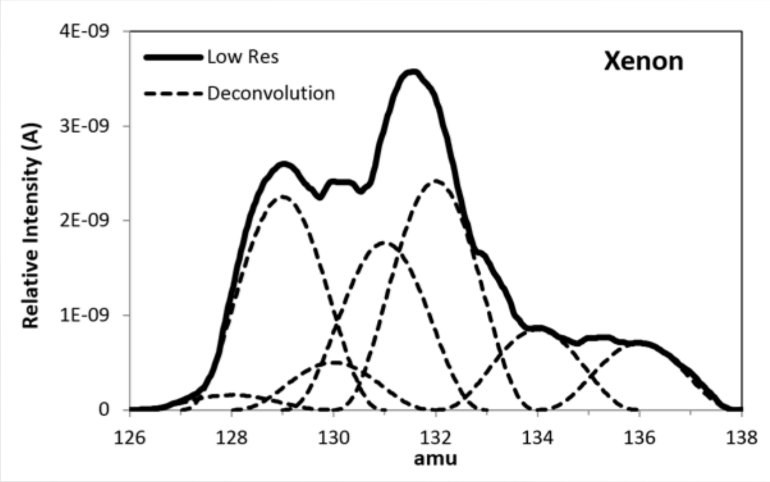



Nanostructured ferritic alloy simultaneously neutron irradiated and helium injected, imaged by the JEOL ARM 200CF TEM

# Fission Gas Analysis

- Thermo-gravimetric/differential thermal analysis
  - Seiko 320 TG/DTA
  - Pfeiffer Thermostar mass spectrometer (1 amu resolution)
  - Both modified for in-cell use






| Stable Xe Isotopes amu | Natural Abundance % | Measured % |
|---|---|---|
| 128 | 1.9 | 1.8 |
| 129 | 26.4 | 26.0 |
| 130 | 4.1 | 5.8 |
| 131 | 21.2 | 20.4 |
| 132 | 26.9 | 27.9 |
| 134 | 10.4 | 9.8 |
| 136 | 8.9 | 8.2 |

# Analytical Chemistry

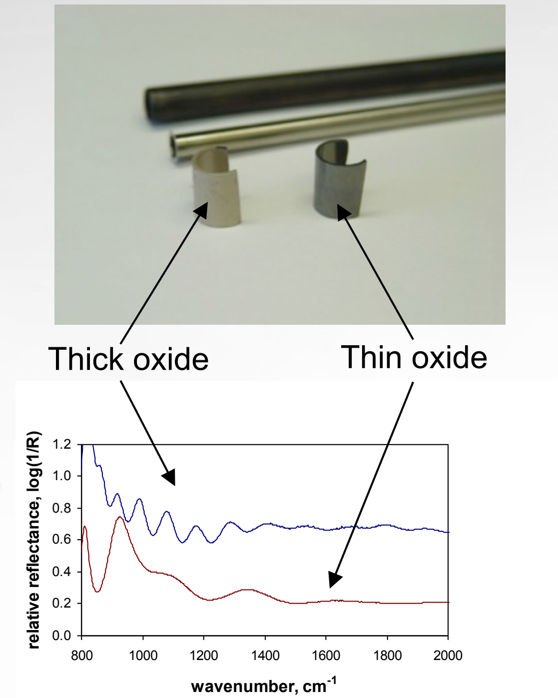


- Helium mass spectrometry
  - Measures $^{3}He$ and $^{4}He$ in parts-per-trillion concentrations in steels and other materials (~$10^{8}$ atoms detection limit)
- Hydrogen isotope mass spectrometry
  - Measures individual hydrogen isotope concentrations to ppm levels
- TIMS
  - Useful for measuring isotopic abundance to determine burnup
- ICP-OES
- ICP-MS
- Gas mass spectroscopy
- Ion chromatography
- NMR spectroscopy (300 MHz)
- Raman spectrophotometry

# Surface Science



- Four Physical Electronics Systems
  - Auger Electron Spectroscopy
  - X-ray Photoelectron Spectroscopy
  - Secondary Ion Mass Spectroscopy
  - Scanning capability
  - Spatial resolution from 0.1 to 50 μm
- Fourier Transform Infrared Spectroscopy

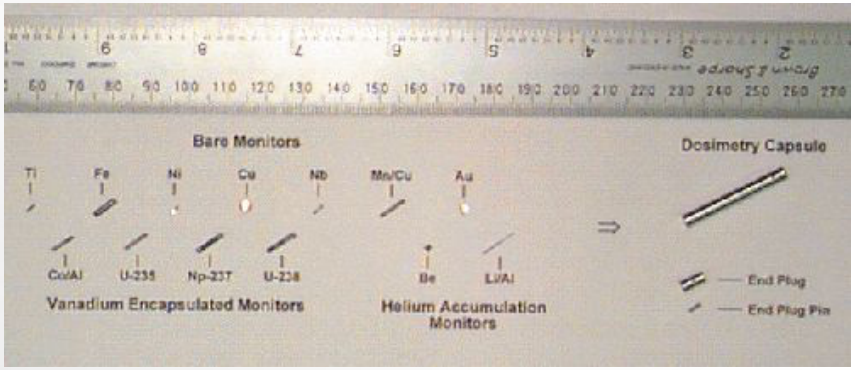



Measurement of oxide thickness on Zircaloy-4 tubes by FTIR

Physical Electronics 560 for scanning AES, XPS, and SIMS on radioactive samples

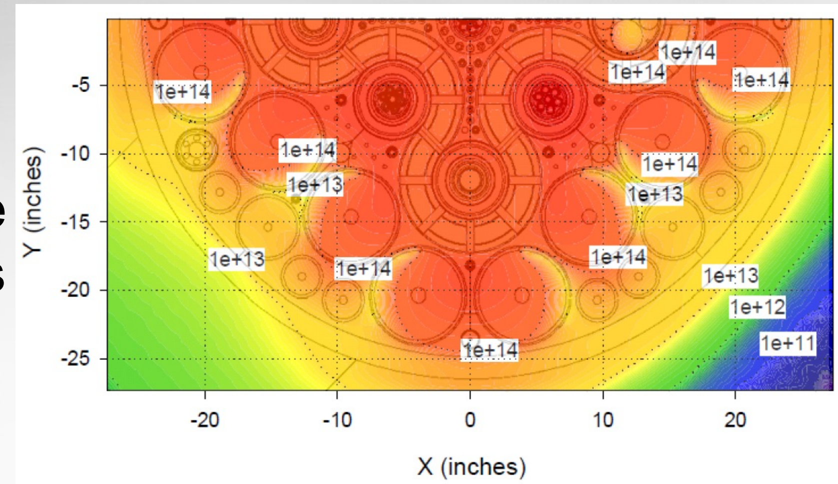
# Reactor Dosimetry

- Flux wires
  - A combination of mg-size pieces of very pure metals that have (n,$\gamma$) reactions
    - Distinct gammas
    - Covers the spectrum of interest
  - Typically encased in a low-activation capsule (e.g. V) so they can be counted via gamma spectroscopy without disassembly after irradiation
  - Using appropriate codes (e.g. STAYSL) along with good spectra, the energy-dependent fluence can be reconstructed from flux wire activation
  - Subsequent calculations (e.g. SPECTER) can be done to convert fluence to dpa
  - In the absence of flux wires (or in addition to them) sections of the irradiation capsule can be used to provide dosimetry data

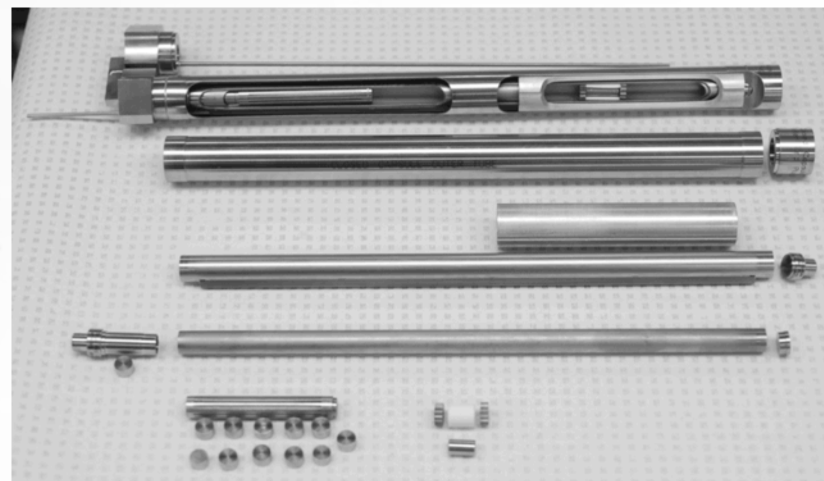


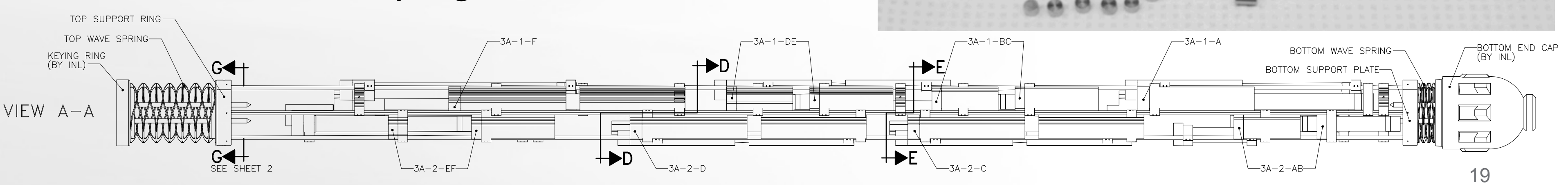
# Irradiation Experiment Design, Analysis, and Fabrication






- PNNL has capabilities and experience designing and fabricating experiments irradiation in ATR
  - Neutronics
  - Thermal-hydraulics
  - Structural engineering
  - Both lead and drop-in experiments
  - High precision machining and weld capabilities
  - NRC- and INL-approved 10 CFR 50 Appendix B and ASME NQA-1 quality assurance programs





# Possible Future Inclusions into PNNL Partner Facility Capability



- PNNL houses the Environmental Molecular Science Laboratory, a DOE Office of Science User Facility
- EMSL (including RadEMSL capabilities below) not currently included in PNNL NSUF Partner Facility, but potentially could be in future
  - Atom probe tomography
  - Electron microprobe
  - MicroXRD
  - X-ray photoelectron spectrometer
  - NMRs for solids and liquids (100 and 750 MHz)
  - Continuous wave x-band (9.5 GHz) electron paramagnetic resonance spectrometer
  - Analytical chemistry
    - ICP-MS
    - Ion chromatography