



Postirradiation Examination Analysis and Capabilities

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ATR National Scientific User Facility Users Week
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Overview

PIE Flavors

PIE Capabilities

INL PIE Vision

Non-destructive techniques

Destructive techniques





PIE Flavors

Postirradiation examination (PIE) encompasses a variety of user definitions

LWR industry – poolside exams

Materials tests – TEM, FIB, tensile testing

Nuclear data – Isotopic analysis

Fuels development – Non-destructive and destructive examinations

Possible PIE Flavors

APPLE – Analytical Postirradiation Properties Laboratory for nuclear Energy

PECAN – Physics Experiments and Characterization on Atomic and Nano-scale

LEMON – LasEr-based Measurements of Nuclear material

MEAT – Mechanical / Environmental Analysis and Testing

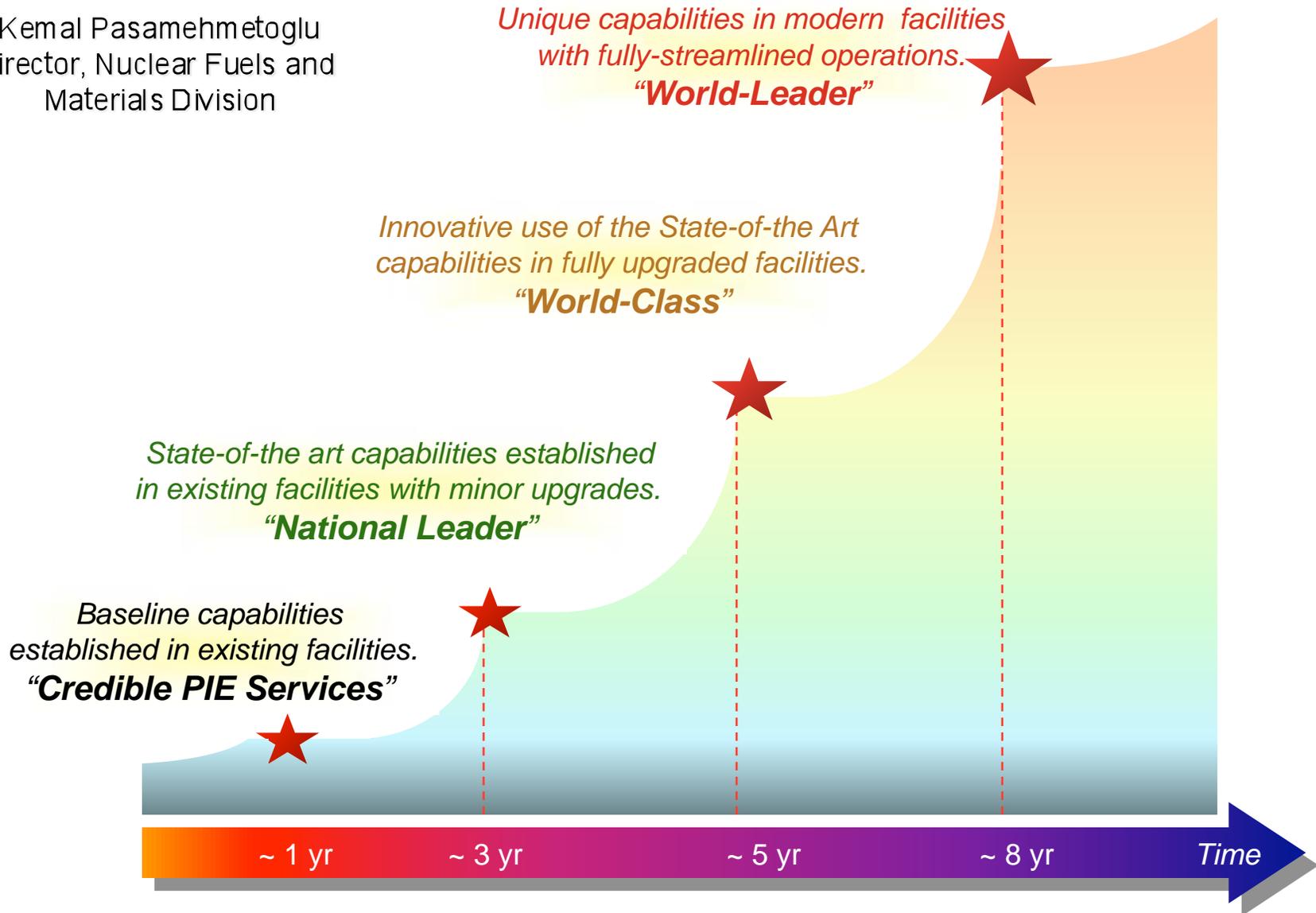
CREAM – CharacteRization by Electron Analytical Microscopy



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INL PIE Vision

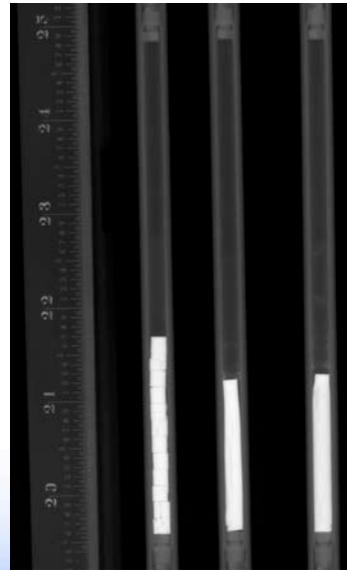
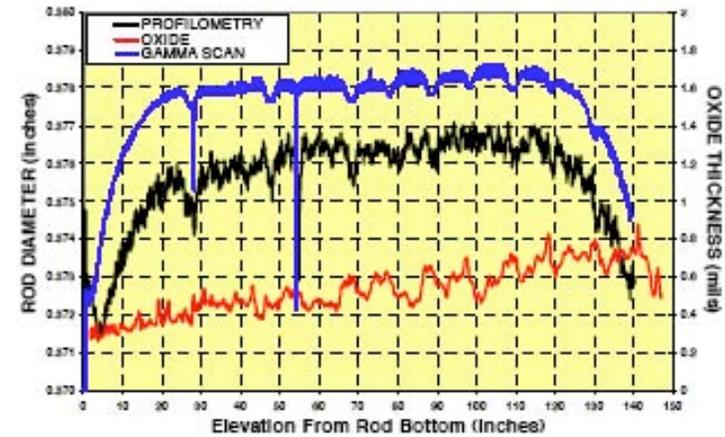
Kemal Pasamehmetoglu
Director, Nuclear Fuels and
Materials Division





Non-Destructive Techniques

- Visual Inspection
- Neutron Radiography
- Dimensional Inspection
- Gamma Scanning
- Eddy current Testing





Visual Inspection

Detailed Visual Inspection

Thru-cell wall periscope 2X, 10X, 25X

Digital Still Camera Photography

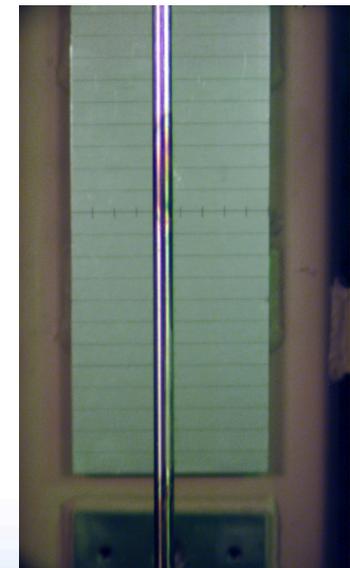
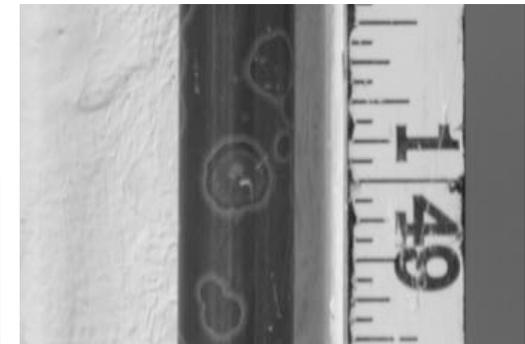
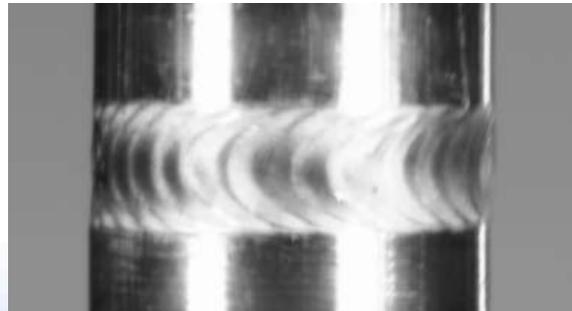
Macro photography Inspection

Thru Hot Cell window

Digital Video Camera Photography

Digital Still Camera Photography

Full color with grey balance



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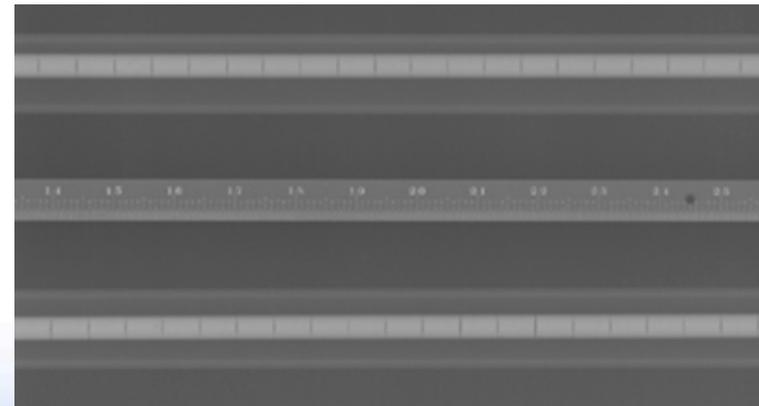
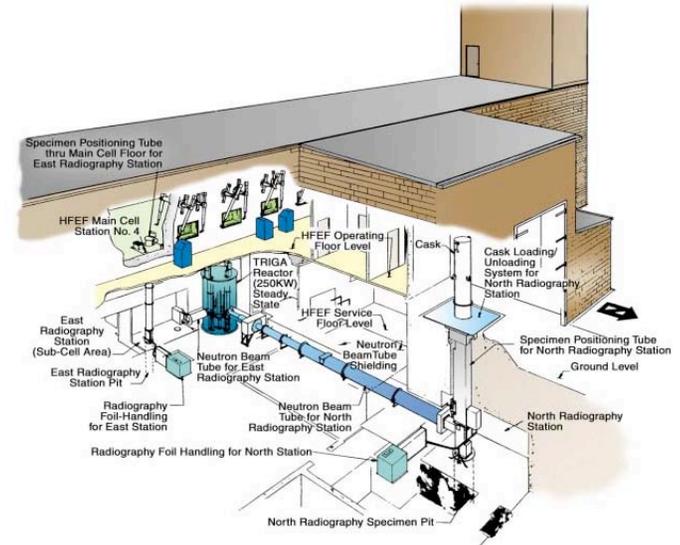
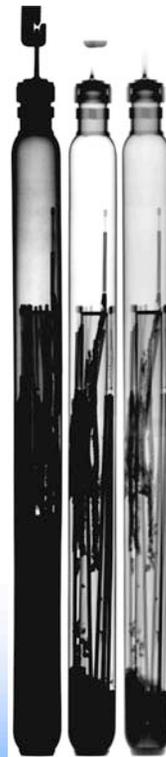
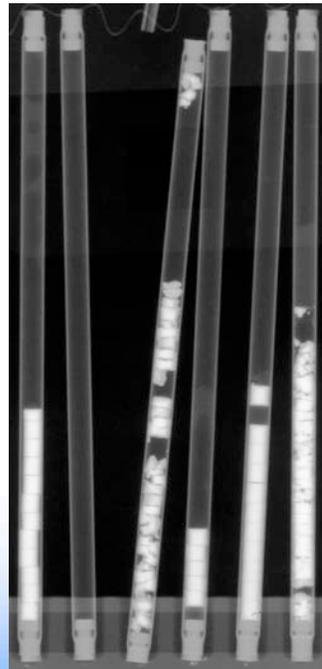
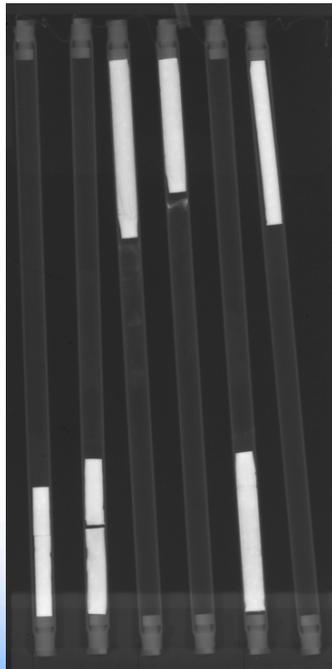
Neutron Radiography

Purpose: Non-destructively interrogate internals

Application:

Evaluate fuel integrity and movement

Hydridding in LWR cladding





Gamma Scan Analysis

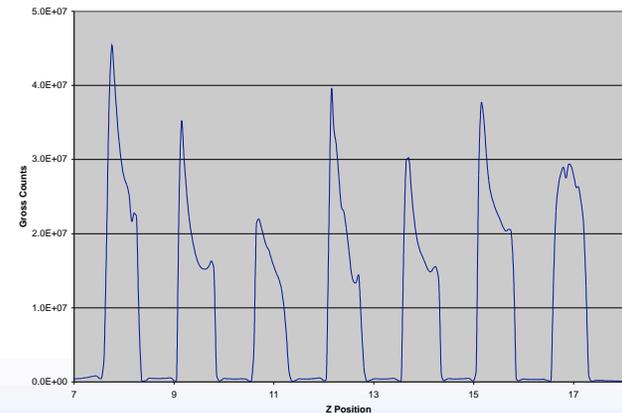
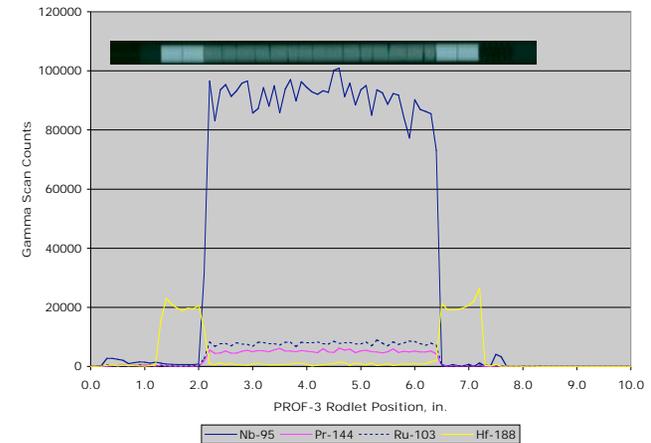
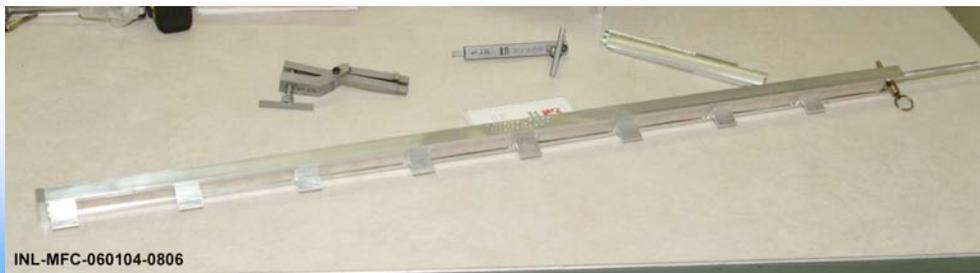
Purpose: Measure gross and isotopic profile of irradiated fuels

Application:

- Relative fuel burnup profile
- Pellet-pellet interfaces
- Relative distribution of various isotopes of interest in fuel

Description:

- Multi-channel Analyzer w/ software Library
- Vertical Step Travel Limit 0.01 in./step (0.05 - 0.1 in. typical)





Dimensional Inspection

Purpose: Measure diameter / plate thickness and bow and length

Application:

Cladding creep down in-reactor service and creep out during dry storage

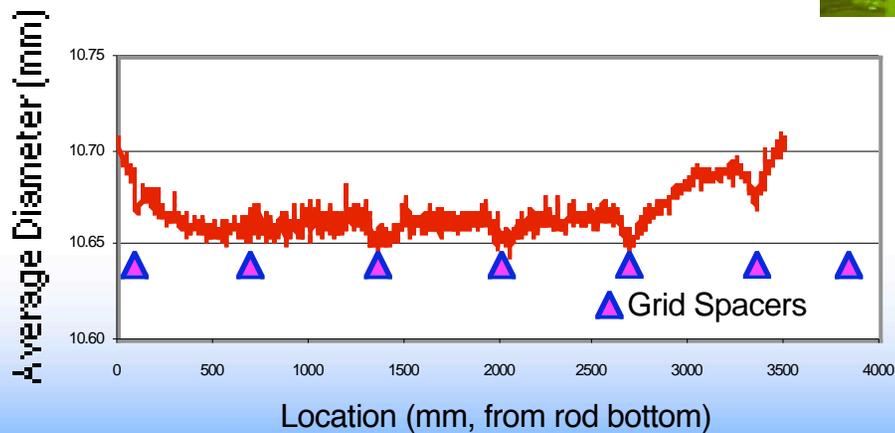
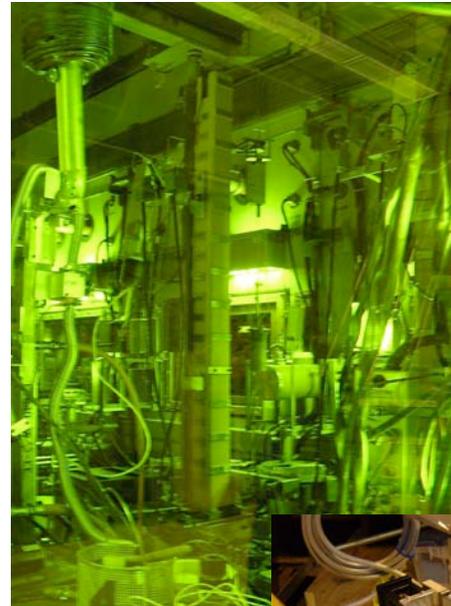
Irradiation induced swelling

Fuel rod growth

Description:

Element contact profilometer: diam. ± 0.0002 in. (± 0.0051 mm)

Bow and Length: ± 0.02 in. (± 0.51 mm)





Eddy current measurement system

Purpose: Non-destructively evaluate the structural performance of nuclear fuel cladding

Application:

Detect and characterize material defects

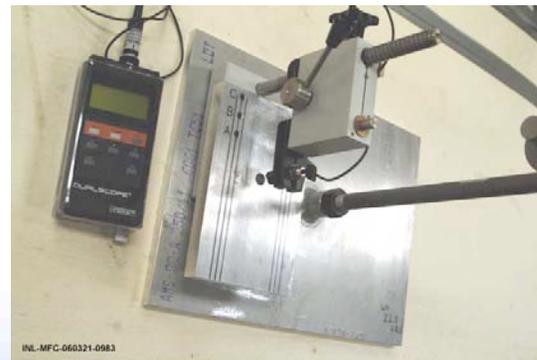
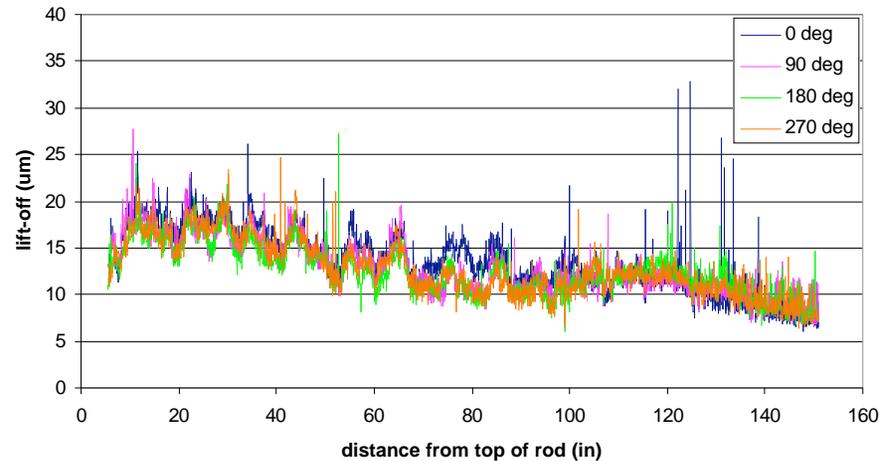
Non-destructively measure oxide layer thickness

Measures electrical current induced when a conductor is placed in a region of shifting magnetic flux

Description:

Max. sample size: 1 in. D x 154 in. L

Oxide thickness uncertainty: $\pm 5 \mu\text{m}$

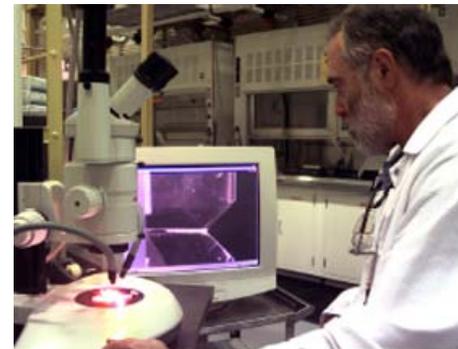
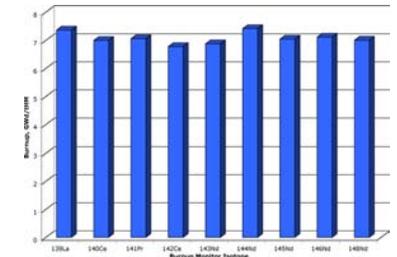
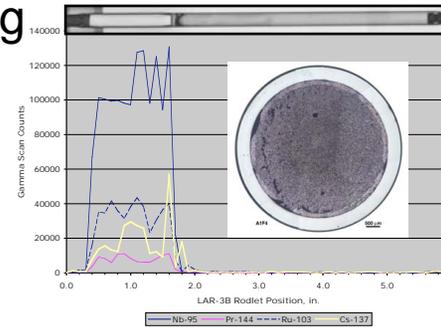


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Destructive Techniques

- Test disassembly and sample harvesting
- Fission Gas Puncture & Analysis
- Isotopic & Burnup Analysis
- Microstructure
- Microchemistry
- Physical properties
- Mechanical properties



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Test disassembly, sample retrieval and furnace testing

Purpose: Disassemble irradiation experiments and retrieve samples for testing. Perform blister anneal test.

Application:

Disassemble irradiation experiment capsules

Retrieve reduced size samples for SEM / TEM analyses in EML

Evaluate irradiated plate fuel residual bond strength

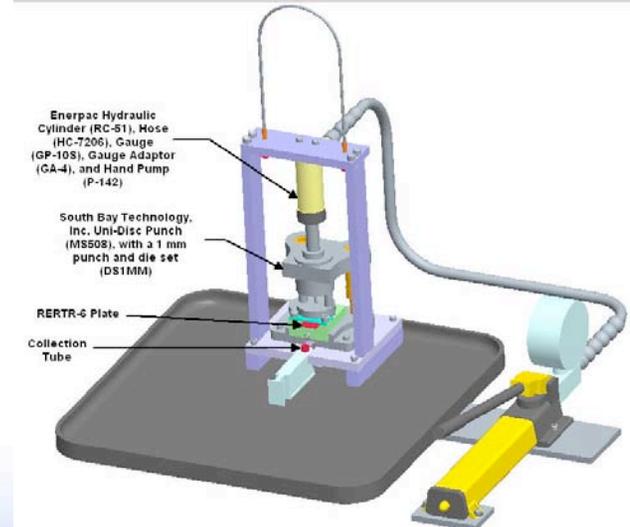
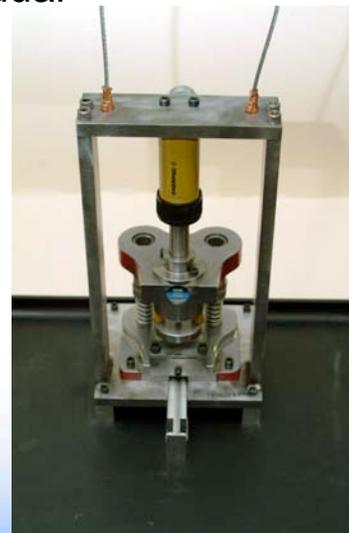
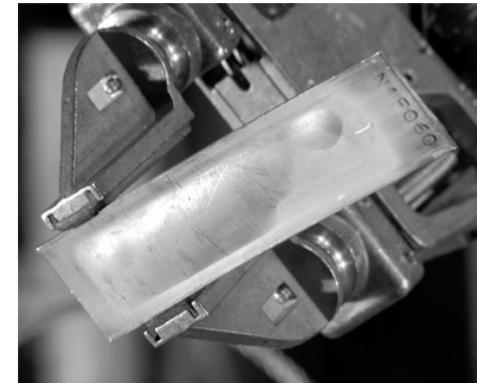
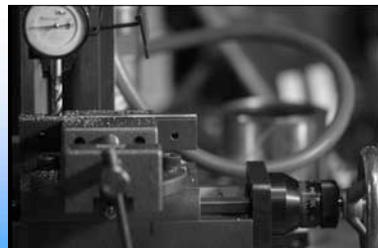
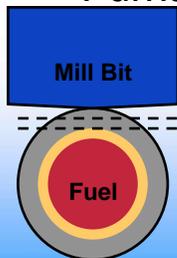
Description:

Table top Mill

SEM Punch

Furnace temp: 1200°C

Furnace cavity: 6" D x 24" H





Fission Gas Puncture & Analysis

Purpose: Puncture fuel rod and analyze fission gas pressure/ internal void volume and chemical/isotopics

Application:

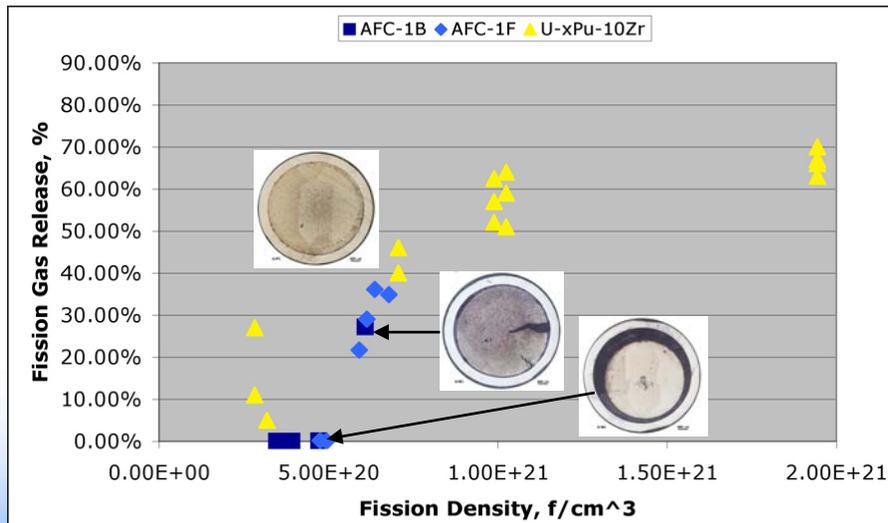
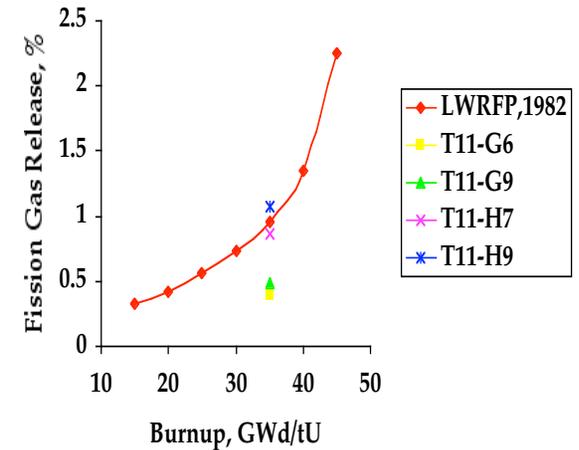
Determine fission gas and helium release

Description:

Laser puncture system

Fuel rod internal void volume and gas pressure, $\pm 5\%$

Plenum gas batch sample chemical analysis





Fuel annealing furnace for fission gas release studies

Purpose: Measure temperature-driven release of condensable fission products and fission gases from irradiated fuel

Application:

Heat irradiated fuel in helium sweep gas ($T \leq 2000^{\circ}\text{C}$)

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





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: $<\pm 1.0\%$

Fission product isotopes: $\pm 2\%$

Elemental analysis: $\pm 2-5\%$

NIST traceable standards





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

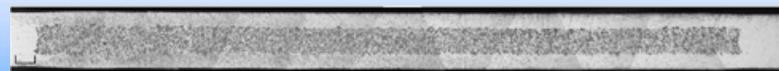
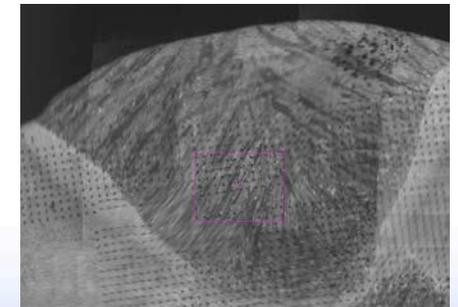
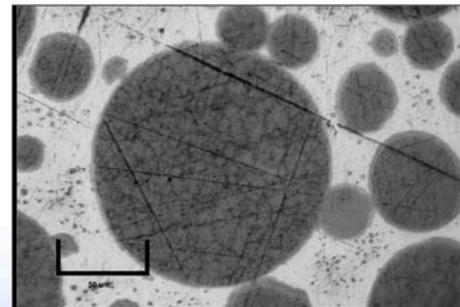
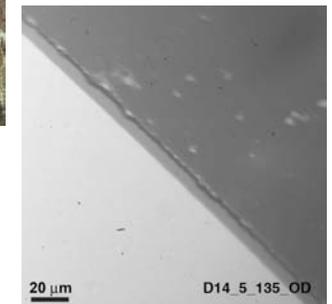
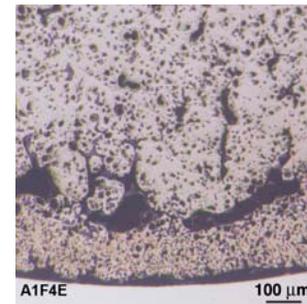
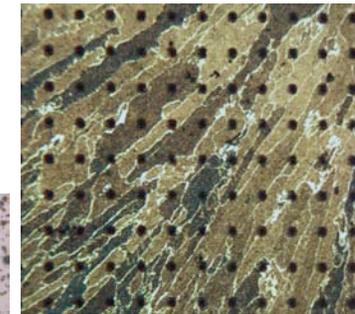
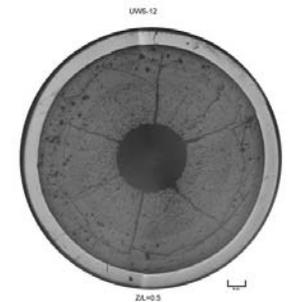
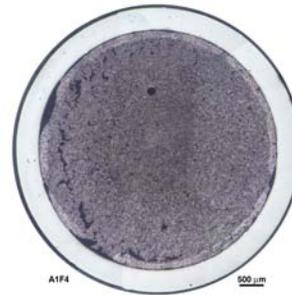
Leitz MM5 RT Metallograph (80X to 800X)

Microindenter Hardness Tester

Automatic stage control

Integrated data collection and analysis

Digital Still Photographic Image





Micro Gamma Scan Analysis

Purpose: Measure radial isotopic profile

Application:

Determine radial burnup profile

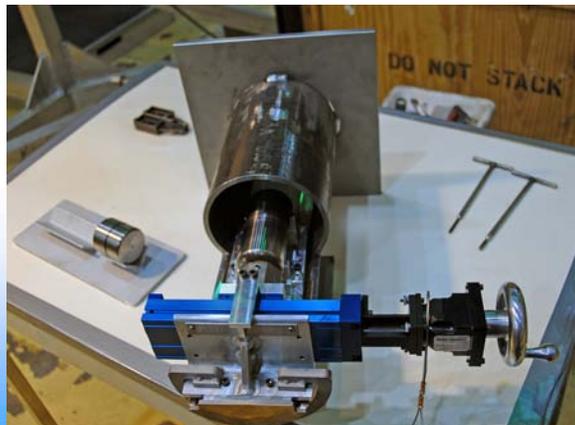
Investigate constituent migration in irradiated fuels

Description:

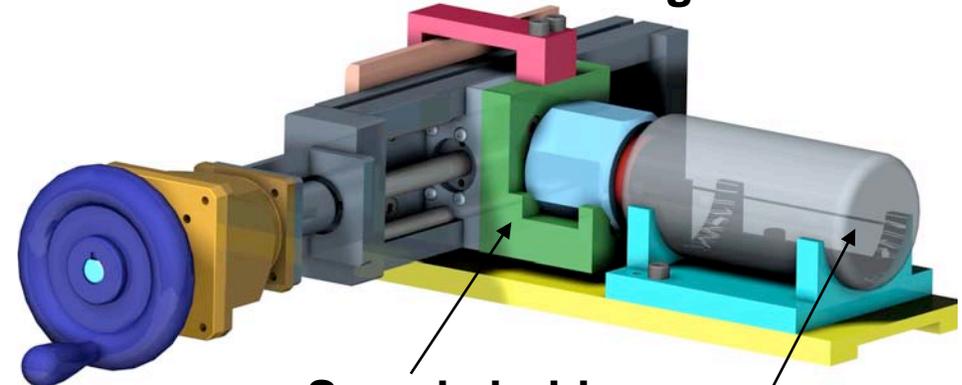
Enhancement of Analytical Laboratory Gamma Scan System

Precision linear stage and integral sample holder

Tungsten Collimator

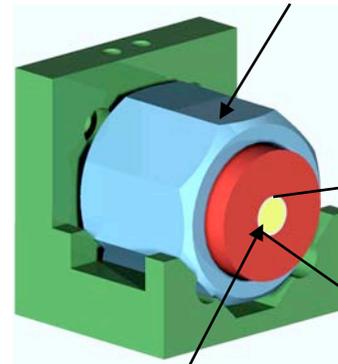


Precision linear stage

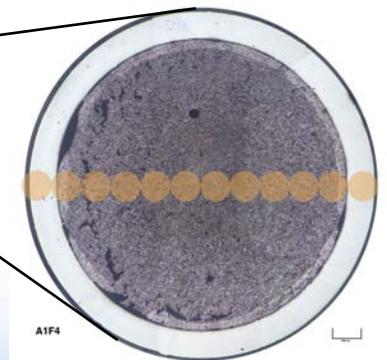


Sample holder

Collimator



Fuel Sample



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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 spectroscopy

Identify phases using crystal structure information by electron backscatter diffraction

Study fuel cladding chemical interaction

Description:

JEOL 7000F SEM with EDS, WDS, EBSD detectors.

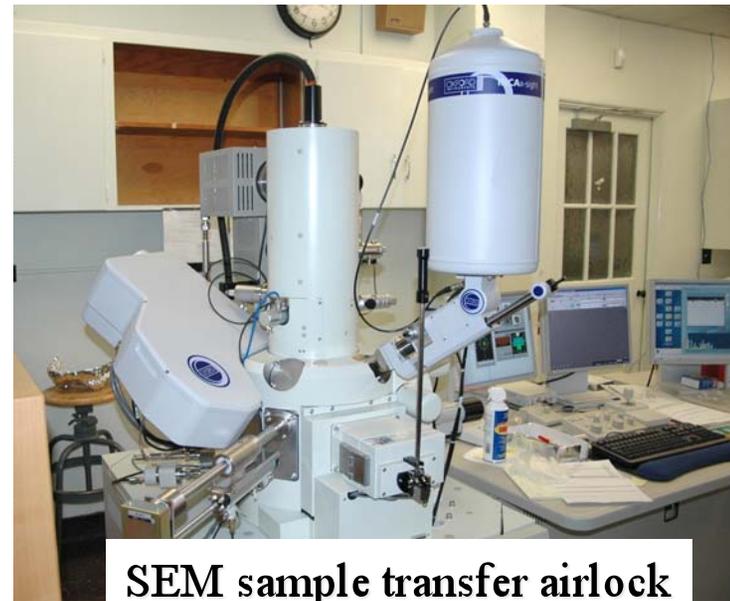
Mag: 200,000x (4 nm resolution)

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



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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

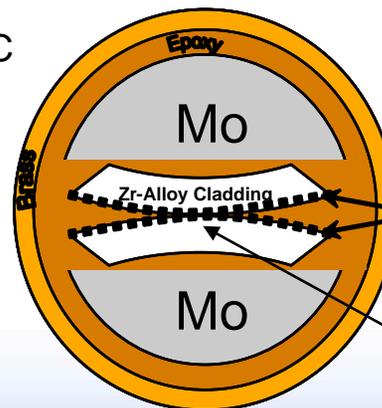
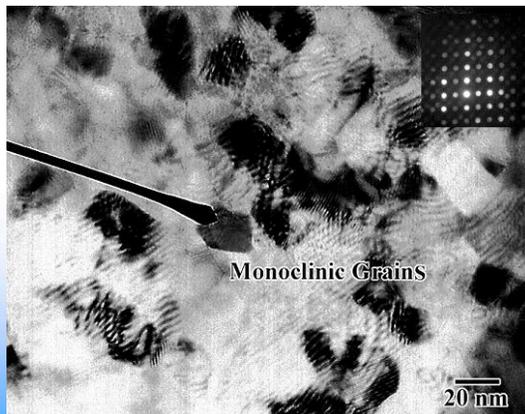
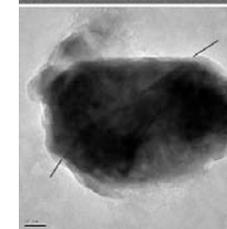
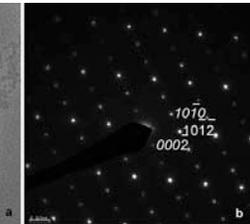
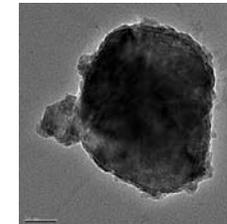
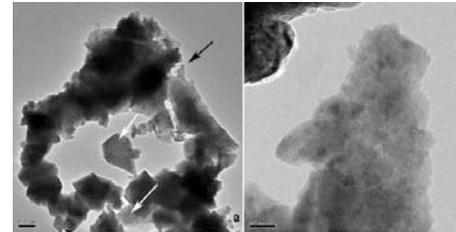
Description:

JEOL 2010 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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Dual Beam Focused Ion Beam

Purpose: Investigate damage that occurs at the submicron level in irradiated fuels and materials. Prepare small volume samples of highly activated materials for subsequent examination

Application:

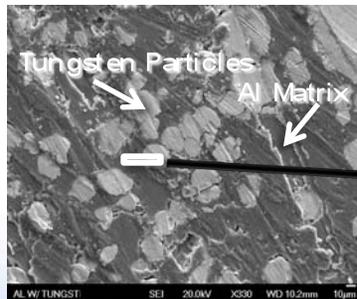
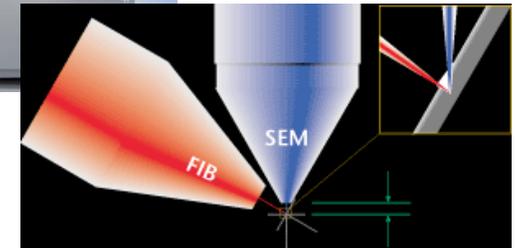
- Electron and ion imaging
- 3D image and chemical reconstruction of submicron features
- Site-specific microsectioning for TEM membrane preparation

Description:

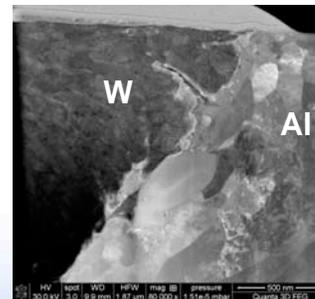
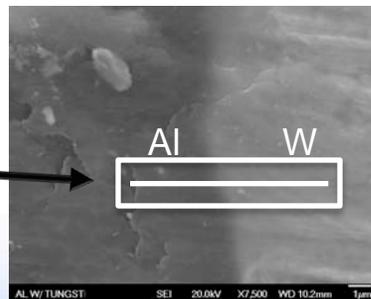
- FEI Quanta 3D FEG Dual Beam FIB with EDS, WDS, EBSD and STEM
- Imaging resolution: <3 nm e-beam, <7 nm ion
- Ga ion source



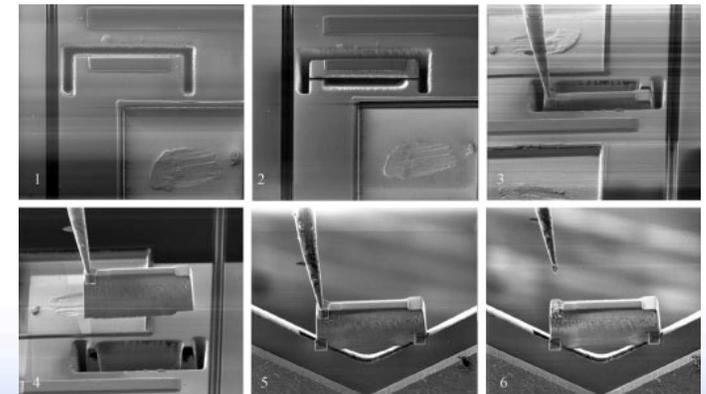
Two columns for simultaneous imaging and microscale milling and sectioning



W-Al Surrogate Dispersion Fuel



STEM Image





Shielded Electron Probe MicroAnalyzer (EPMA)

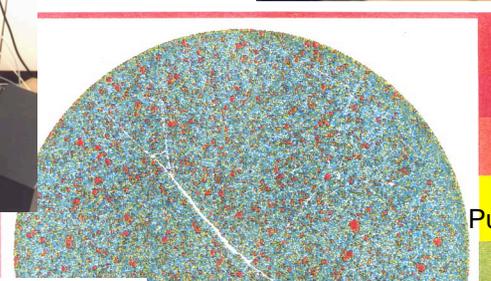
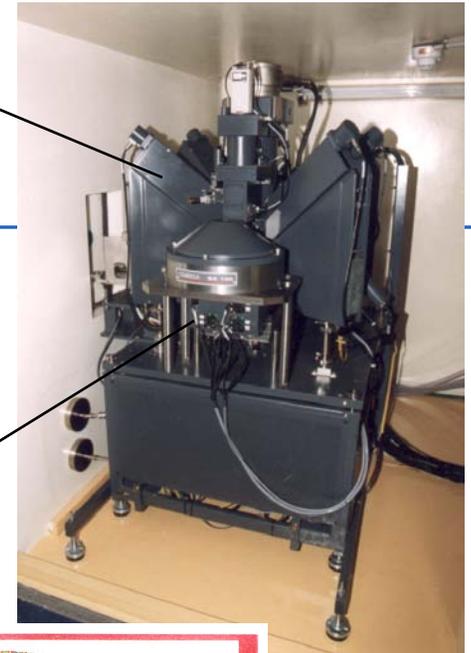
Purpose: Measure the localized micro-scale chemical composition of whole transverse cross-sections of irradiated fuels and materials (<3 Ci) and perform electron imaging of these samples.

Application:

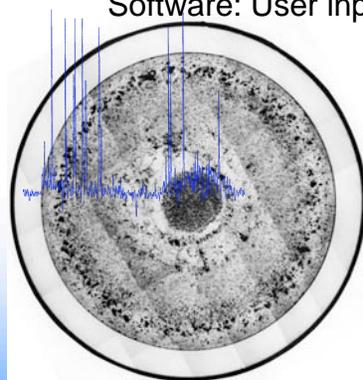
- Characterize compositional homogeneity of actinide-bearing transmutation fuels
- Analyze migration and redistribution of fuel constituents and fission products
- Quantify radial burnup profile
- Microstructure imaging

Description:

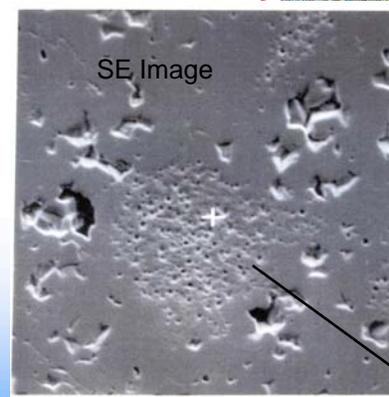
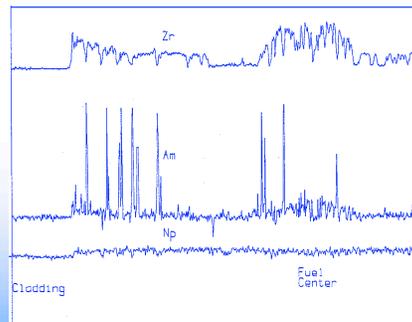
- CAMECA SX100R EPMA
- 4 WDS spectrometers (4 crystals)
- SE detector for imaging
- Be to actinides sensitivity: 20-100 ppm
- Software: User input data.



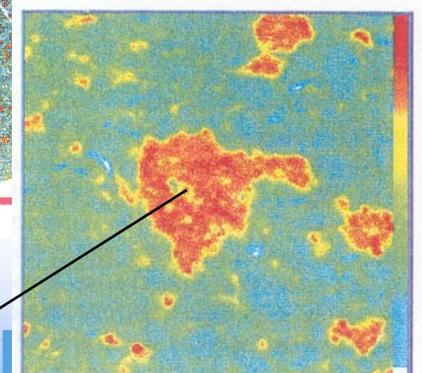
Pu X ray maps



U-Pu-Am-Zr metallic alloy (EBR-II)



SE Image



Pu agglomerate in PWR MOX
Higher local porosity corresponding to higher burnup



Micro X-ray Diffractometer (MXRD)

Purpose: Identify and quantify compositional phases of actinide-bearing samples. Provide phase data which is link to thermodynamic and kinetic database used in modeling and simulation of fuel behavior

Application:

Determine crystal structures and lattice parameters with high precision (e.g., quantifying swelling)

Estimate degree of crystallinity (recognizing amorphous materials)

Identify crystalline phases by their structures and quantify proportion in mixtures

Quantify crystallographic preferred orientations (e.g., understanding cladding failure mechanisms)

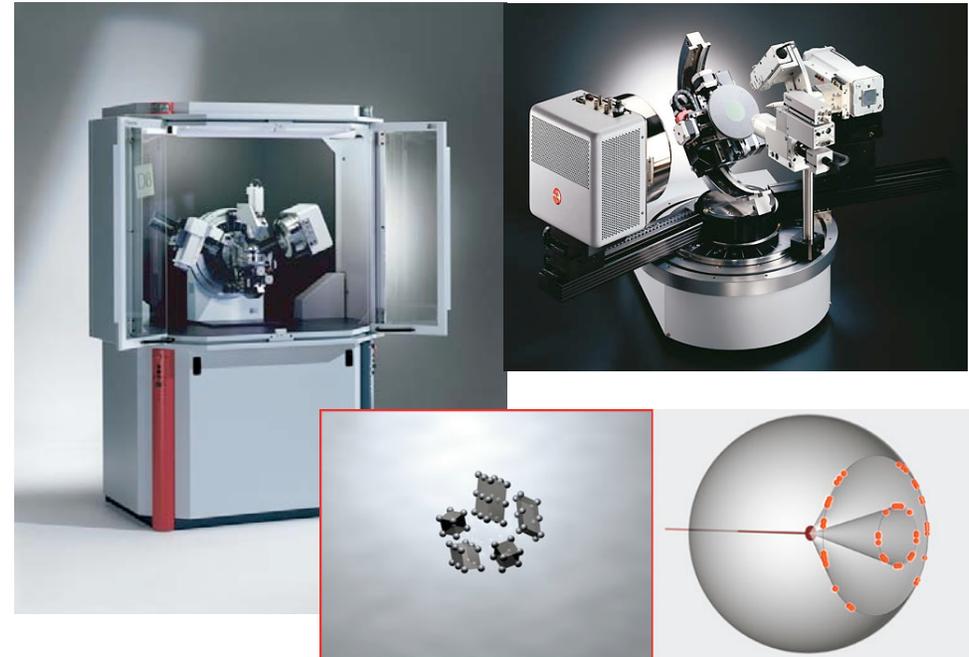
Description:

Bruker D8 Discover MXRD

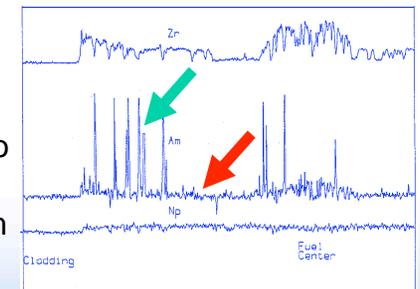
Bragg-Brentano geometry: lattice parameter refinement to 1 picometer (<0.5%) for powders

Micro-diffraction beam: 100 μm at sample surface (analytical volume: $\sim 0.006 \text{ mm}^3$, vs $\sim 20 \text{ mm}^3$ for conventional XRD), options allow beam to 10 μm

Laser / video imaging system for beam positioning to 10- μm



MXRD will identify Am phases correlated to chemical profiles from EPMA





Physical Properties Testing

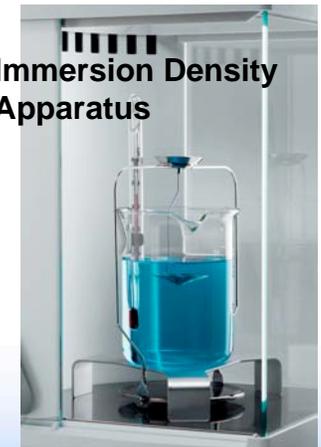
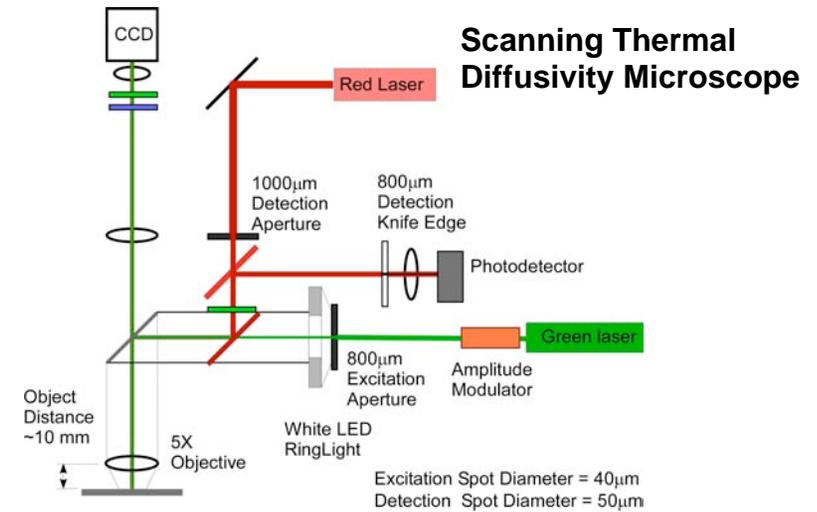
Purpose: Characterize effects of irradiation on basic physical properties. Provide data necessary to identify fundamental mechanisms and develop predictive models

Application:

- Thermal diffusivity to quantify conductivity changes with irradiation
- Immersion density measurements using Archimedes Principle
- Density to quantify irradiation swelling
- Density as input to derive thermal conductivity
- Hydrogen absorption and hydrogen embrittlement

Description:

- Scanning Thermal Diffusivity Microscope
- Immersion density: ≥ 0.25 g sample, 1RSD: $\leq \pm 1\%$
- Gas pycnometry: ≥ 1 cm³ sample, 1RSD: $\leq \pm 1\%$
- LECO RH-400 H₂ Determinator modified for remote operation
- Hydrogen content: 0.2 g sample, 1RSD: $\pm 2.5\%$



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Mechanical Properties Testing

Purpose: Characterize effects of irradiation on basic mechanical properties and mechanical / environmental behavior. Provide data necessary to develop predictive models

Application:

Determine tensile properties/ductility for axial and circumferential directions at room temperature and elevated temperature prototypic of reactor operation or storage

Finite element analysis for deriving properties from specialized geometries

Machine complex specimen geometries from irradiated materials and cladding from LWR and advanced reactors

Investigate mechanical / environmental behavior

Description:

RAM Electric Discharge Machining (EDM)

Instron Model 5869: 50 kN, 0.001-500 mm/min

Furnace: 1200°C, Inert atmosphere

Inspection tolerance ± 0.0005 in.

IASCC Rigs

