Current Status of the Irradiated Materials Characterization Laboratory at INL with Limited PIE Microstructural Characterization

Dr. B. D. Miller
Idaho National Laboratory
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Mito, Japan
Outline

• **Current Status of Irradiated Materials Characterization Laboratory (IMCL)**
  – Reason for IMCL
  – Sample Analysis Stations
  – Current IMCL layout and operational equipment
  – Future expansion and equipment

• **Limited post irradiation examination characterization at IMCL**
  – Focused Ion Beam Microscopy
  – Electron Probe Micro-Analyzer
  – Transmission Electron Microscopy
Reasons for IMCL

• With recent incorporation of high end characterization equipment on irradiated materials and fuels, INL needed a facility to properly house the equipment.

• Includes Focused Ion Beams, Electron Probe Micro Analyzers, Transmission Electron Microscopy, and a shielded sample preparation area (SSPA).
Design Basis of IMCL

• **Low vibration**
  – Floor designed as a single concrete slab with isolation pads for vibrating equipment

• **Temperature control**
  – Less than 1°C per hour

• **Low electromagnetic interference**
  – As to not interfere with operation of high-end electron microscopes
Current Layout of IMCL

- IMCL currently has 5 areas designated for characterization of irradiated materials
  - SSPA, TEM, FIB/PFIB SAS, and Thermal Properties SAS
  - Electron Probe Micro-Analyzer (EPMA) SAS
  - Plasma and Focused Ion Beam SAS (FIB/PFIB)
  - Thermal properties SAS
  - Transmission Electron Microscopy (TEM)

- Room for future expansion
**IMCL Shielded Cask (ISC)**

- Two specially designed casks for IMCL use
- Incorporate La Calhéne mating system
- Shielding equivalent of ~21 cm of steel using lead
- Compatible with various facilities at INL
- Compatible with the flying pig being developed

*IMCL Shielded Cask (ISC)*

ISC awaiting docking with the Shielded Sample Preparation Area

*Look. It is a flying pig. Oink.*

Flying Pig

*IMCL shield cask (ISC)*

ISC awaiting docking with the Shielded Sample Preparation Area
Shielded Sample Preparation Area-SSPA

• The SSPA's primary focus is sample preparation of highly radioactive materials/fuels
• All portions of the system are connected allowing easy transfer of samples
• Three shielded bays
  – CRL Manipulators
    • Type L-HD
    • Lead equivalent of 21 cm steel
• Radiological glovebox
  – N₂ inert
  – Sample preparation of low dose samples
• Radiological fume hood
  – Decontamination activities
  – Sample preparation

Relaxing after a long day at the SSPA

Various images of the SSPA line
Shielded Sample Preparation Area Cont’d

• Three shielded bays
  – CRL Manipulators
    • Type L-HD
  – Sample preparation bay
    • Autopolisher, low speed saw, ultrasonic cleaner, etc
  – Optical microscopy bay
    • Keyonce VHX-5000 microscope
    • 100-1,000x magnification
  – Shielded transfer cell
    • Transfers and radiation level measurements
**Sample Analysis Stations (SAS)**

- IMCL uses a variable “hot cell” design designated as Sample Analysis Stations (SAS)
- Instruments are coupled to a glovebox through a loading/unloading port
- Shielded steel walls enclose the glovebox and instrument, providing shielding
  - 21 cm steel walls
- Manipulators are attached to the gloveboxes, operated outside the shielded walls
- Flexible design to meet future equipment needs
- With only the loading port attached the glovebox, instrument maintenance is simplified as the outside of the instrument is not contaminated
- Designed to shield a 2 Ci-Co\textsuperscript{60} source

SAS layout for the Focused Ion Beam Microscopes

Steel wall sections awaiting assembly
Focused Ion Beam SAS’s

• Duel SAS structure installed for two Focused Ion Beams (FIB) microscopes
  – FEI Helios Plasma FIB
  – FEI Quanta 3D FIB
    (radioactively contaminated)

• SAS currently undergoing readiness review for operational status

• Fully operational on irradiated fuels and materials in late spring 2018
EPMA SAS

• Similar to the FIB/PFIB SAS setup but with extra manipulator for EPMA operations
• EPMA is a CAMECA SX-100R with 4 wavelength dispersive spectrometers
• Currently operational handing irradiated fuel samples
Thermal Properties SAS

- Currently fabricated and unassembled at IMCL
- Planned for installation in 2018
- Operational in 2019
- Planned equipment include:
  - Differential Scanning Calorimetry
  - Thermal Conductivity Microscope
  - Laser Flash
Transmission Electron Microscopy-TEM

• IMCL is equipped with a FEI Titan 200 keV CHEMI-Scanning Transmission Electron Microscope (STEM)
• Equipped with 4 Energy Dispersive Spectrometers (EDS) for fast elemental mapping
• Located in acoustic sound lowering room to improve resolution
• Able to perform sub-nanometer chemical analysis
• Currently operational to characterize irradiated materials and fuels
Future Expansion?

• Options include:
  – Atom Probe Tomography
  – Shielded Scanning Electron Microscope
  – Mechanical Properties Cell
  – Additional FIB’s?

http://toonut.com/dog-house-expansion/
Post Irradiation Examinations-FIB

• **FIB/PFIB** offer site specific characterization of materials on micron scale and below

• **Capabilities of the FIB/PFIB include**
  - TEM lamella preparation
  - Cube preparation for serial sectioning and chemical profiling of specific regions
  - Cross-section milling for visualization of the microstructure under the polished surface
  - Electron backscatter diffraction surface preparation
  - and many others…….
Post Irradiation Examination-EPMA

- EPMA characterization performed on an irradiated TRISO particle irradiated at the Advanced Test Reactor (ATR) at INL
- Uranium Oxycarbide surrounded by C buffer layers and a SiC confinement
- Focus on fission product migration across the fuel particle into the TRISO particle

Cracked TRISO particle

WDS maps of a TRISO fuel particle
Post Irradiation Examination-TEM

- U-Mo fuels being studied for use in research and test reactors throughout the world
- Forms ordered bubble superlattice at fission densities typically lower than $4.5 \times 10^{21}$ fissions/cm$^3$
- It was assumed that the bubbles were stabilized by Xe fission gas
- Proof that Xe is indeed present in the fission gas pores

EDS maps of the bubble superlattice in U-Mo fuels
Post Irradiation Examination-TEM

- HT-9 has been used as a cladding material for advanced fuels
- Rare earth elements diffuse into the cladding
- Diffusion can weaken the mechanical properties of the cladding
- Include:
  - Nd, Ce, Pr, Mo, and La

Scale Bar is 1 µm
Concluding Remarks

• Post-irradiation examination of irradiated fuels and materials has commenced at IMCL

• The EPMA, FIB, and TEM are radiologically operational with the FIB/PFIB coming online in early spring

• The thermal properties cell has been fabricated with anticipation of being installed in 2018 and operation in 2019

https://blogs.ams.org/mathmentoringnetwork/2014/08/04/math-talk-preparing-your-conference-presentation/
Questions!