Commissioning of the Irradiated Material Characterization Laboratory

HOTLAB 2018
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Presented by Mitch Meyer
IMCL Team

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Topics

- IMCL Facility Design and Performance
- Shielded Cell Design and Performance
- IMCL Shielded Transfer Cask Design and Performance
- Current IMCL Capabilities
- Future IMCL Capabilities
- Looking Forward
IMCL Concept (2008)

- Idea first presented in 2008 to support Nuclear Science User Facility
- Micro XRD, EPMA, IASCC, FIB, mechanical testing, and TEM
IMCL Design Goals

- Meet DOE and industry program needs
- Broadly accessible as a national/international user facility
- Routine application of state-of-the-art microstructural characterization capability for analysis of irradiated nuclear fuels and materials
- Reduce (dramatically) the time required to complete PIE of irradiated fuels and materials
- Reconfigurable over 40+ year lifetime
- High material quantity limits
IMCL Facility Design (2010)

- 1,130 m² total area
  - 750 m² open bay laboratory
  - 380 m² mechanical and office space
- Single pour reinforced concrete floor 45 cm thick
  - Vibration isolation pads near each instrument 15 cm thick
- Mechanical room on separate foundation
- Reinforced concrete walls (seismic PC-3)
- Single HEPA filtered ventilation system serves HVAC and instrument needs

- IMCL is a Hazard Category II nuclear facility with a material envelope of 300 Ci $^{239}$Pu equivalent (3.4 g $^{244}$Cm)
- 700 g $^{239}$Pu from perspective of criticality
IMCL in 2015

- IMCL building construction was completed in August 2012
- Instrument cell installation began in 2016
IMCL FIB and TEM were heavily used during TPC construction
IMCL Facility Performance

- Facility design based on vibration, EMI, temperature stability, and acoustic noise criteria required for high-resolution TEM

✓ Vibrational performance of facility well within instrument specifications
  - All instruments installed with active vibration cancelling systems to protect against occasional transient vibration (heavy trucks)

✗ Acoustic noise higher than instrument specification
  - Noise level above 55 dB below 2 kHz caused by ventilation system
  - Slight impact on instrument performance
  - Some impact on human performance

- Asynchronous electromagnetic noise slightly out of limits

- Temperature stability in open bay insufficient for TEM (but TEM room required anyway)

- Lesson learned:
  - Isolate room HVAC from hot cell ventilation system if possible (but this is costly)
  - Safety basis/operational tradeoffs
Shielded Cell Design Concept (2010)

- Conventional concrete cell ~1 m (3’) wall thickness with removable steel front and back walls and rear entry door
- Typical of 1960’s cell design currently in use in U.S.
- Difficult to maintain modern instruments using this type of cell
Final Shielded Cell Design

- Shielding and confinement boundaries are separated for ease of access to instrument
- Inert glovebox provides containment of $\alpha + \beta$ contamination
- 22 cm (8.5”) thick steel $\gamma$ shield walls
- Leaded glass windows
- Manipulators penetrate glovebox front wall
- No roof on cells
  - Simplify installation of piping, electrical, and instrumentation
  - Seismic issues
Final Shielded Cell Design

- Instrument connected to glovebox with reinforced ‘rubber’ bellows
- Instrument is part of $\alpha + \beta$ confinement boundary
- Access to glovebox interior though back glove wall
- Commercial (unshielded) entry door used to control access; labyrinth provides adequate shielding
- Instrument electronics/detectors are not ‘hardened’
- Shielded sample storage in glovebox
Shielded Cell Installation

- Overhead crane not included in facility design (affordability)
- Shield panels (3~4 MT) installed using 20 ton mobile crane
- Future cell reconfiguration will require portable gantry crane
FIB Loading System

- Instrument loading through custom sample transfer system
Shielding Performance

- 0.9 Ci $^{60}$Co source testing
- Contact dose rate at front face 5 $\mu$S/hr ($<0.5$ mrem/hr) per hour below 1.8 m (6 ft) height
- Absence of roof leads to slightly higher dose rates 20 $\mu$S/hr (2 mrem/hr) above 1.8 m
- General area dose rate on roof is 200 $\mu$S/hr (20 mrem/hr) – access control
Shielded Transfer Container

- Cask mates to back wall of glovebox
- Clean rapid transfer port (alpha/beta port) is mechanically interlocked to mating port on the cask. Neither container nor glovebox port will open unless the container is ‘locked’ to the glovebox
- Lightly shielded transfer containers also used
Shielded Transfer Container

- Shielded Transfer Container (STC) mates to shielded instrument cells, Shielded Sample Preparation Area, and air hood
- 100 cm (4") of lead shielding
- Interior cavity ~10 cm dia. x 25 cm long
- Total weight ~1950 kg
- Hydraulic adjustment of STC height to match shielded cell cask stand height
- Will interconnect with HFEF (Hot Fuel Examination Facility, 2019) and SPL (Sample Preparation Laboratory)
- Not qualified for use on public roadways
**Shielded Transfer Container Performance**

- IMCL Shielded Transfer Container in position on the SSPA (Shielded Sample Preparation Area)
- Goal: < 2000 μS/hr at 30 cm (< 200 mrem/hr) with 2 Ci $^{60}$Co source term
- Contact dose rate on surface 500 μS/hr (50 mrem/hr) with 1.8 Ci $^{60}$Co source term
- One occurrence of alpha port sticking closed in several hundred uses
IMCL Capabilities

- Shielded Sample Preparation
- FEG-SEM
- EPMA
- XRD (2)
- X-ray μ-CT
- TEM
- FIB
- P-FIB
- Thermal Properties
- PPMS

Fully operational
In progress
Procured, awaiting installation
Future expansion
Shielded Sample Preparation Area

- Shielded sample preparation
  - Diamond saw
  - Autopolisher
- Optical microscopy
  - Keyence VHX
- Sample transfer cell between shielded box and glovebox
- Glovebox for preparation of reduced size/low dose rate samples
- Hood for transfer of low dose rate samples, waste
- Lessons: Argon atmosphere, waste handling

Operational in August 2017
Electron Probe MicroAnalysis (EPMA)

- Coater feedthroughs
- Pu, Am, Np standards

EPMA analysis of a full cross section of irradiated U-Pu-Am-Np-Zr fuel

Operational in August 2017
Transmission Electron Microscopy

Titan 200 KeV TEM with ChemiSTEM EDS system.
Operational in March 2017

Top: Microstructure of MOX fast reactor fuel irradiated to 112 GWd/THM. Bottom: Atomic resolution imaging of 5 metal precipitates and fission gas bubbles in UO₂.
Dual Beam Focused Ion Beams

- FEI Quanta Ga FIB
- FEI Helios Xe plasma FIB
- Lessons:
  - Sample transfer system
  - Cable lengths may impact performance
  - Helios has short working distance and in-lens detectors

Inverse pole figure map of irradiated U-Mo showing the formation of a fine grain (240 nm) structure. Surface cleaned with Helios P-FIB

Microtensile testing of U-Mo fuel at 500°C.

Operational March 2018
Thermal Properties Cell

- Shielded cell construction complete
- Currently in final acceptance testing
- Largest cell in IMCL
- Instruments:
  - Laser flash thermal diffusivity
  - Thermal conductivity microscope
  - Thermogravimetric Analysis, Differential Scanning Calorimetry, Mass Spectrometry
- Operational for radiological samples June 2019
**Additional IMCL Capabilities**

- **Currently installed**
  - FEG-SEM
  - XRD (2)
  - X-ray μ-CT
  - TEM
  - PPMS

- **In progress**
  - Shielded Sample Preparation
  - EPMA
  - FIB
  - P-FIB
  - Thermal Properties
  - Future expansion

- **Procured, awaiting installation**
  - Future expansion

- **Future expansion – user specified**

**Abbreviations:****
- SSPA – Shielded Sample Preparation Area
- EPMA – Electron Probe MicroAnalysis
- FIB – Focused Ion Beam
- TEM – Transmission Electron Microscope
- TPTC – Thermal Properties Test Cell
Additional Capabilities

Quantum systems
Dynacool-9 PPMS
- 2 - 400 K
- 9 Tesla field
- Electrical, magnetic, and thermal property measurements
- January 2019

Zeiss Versa 520
- 0.7 μm spatial resolution
- Diffraction contrast tomography
- In-situ heating and mechanical testing
- Flat panel detector
- March 2019

• Installation of probe corrector and EELS on Titan TEM (March 2019)
• JEOL-7600 FEG-SEM transferred from another facility (March 2019)
• One shielded and one unshielded space remain for future instruments

• Bruker D-8 μ-XRD, 50 μm resolution
• Panalytical Empyrean powder XRD with heated stage
• Unshielded
• Currently installed
• National and international workshops in 2011 defined needs for postirradiation examination.
• A combination of the Hot Fuel Examination Facility, Sample Preparation Laboratory, and the Irradiated Material Characterization Laboratory will meet those needs.
Come work with us!

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