Commissioning of the Irradiated Material Characterization Laboratory

HOTLAB 2018
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Presented by Mitch Meyer

www.inl.gov

Idaho National Laboratory

IMCL Team

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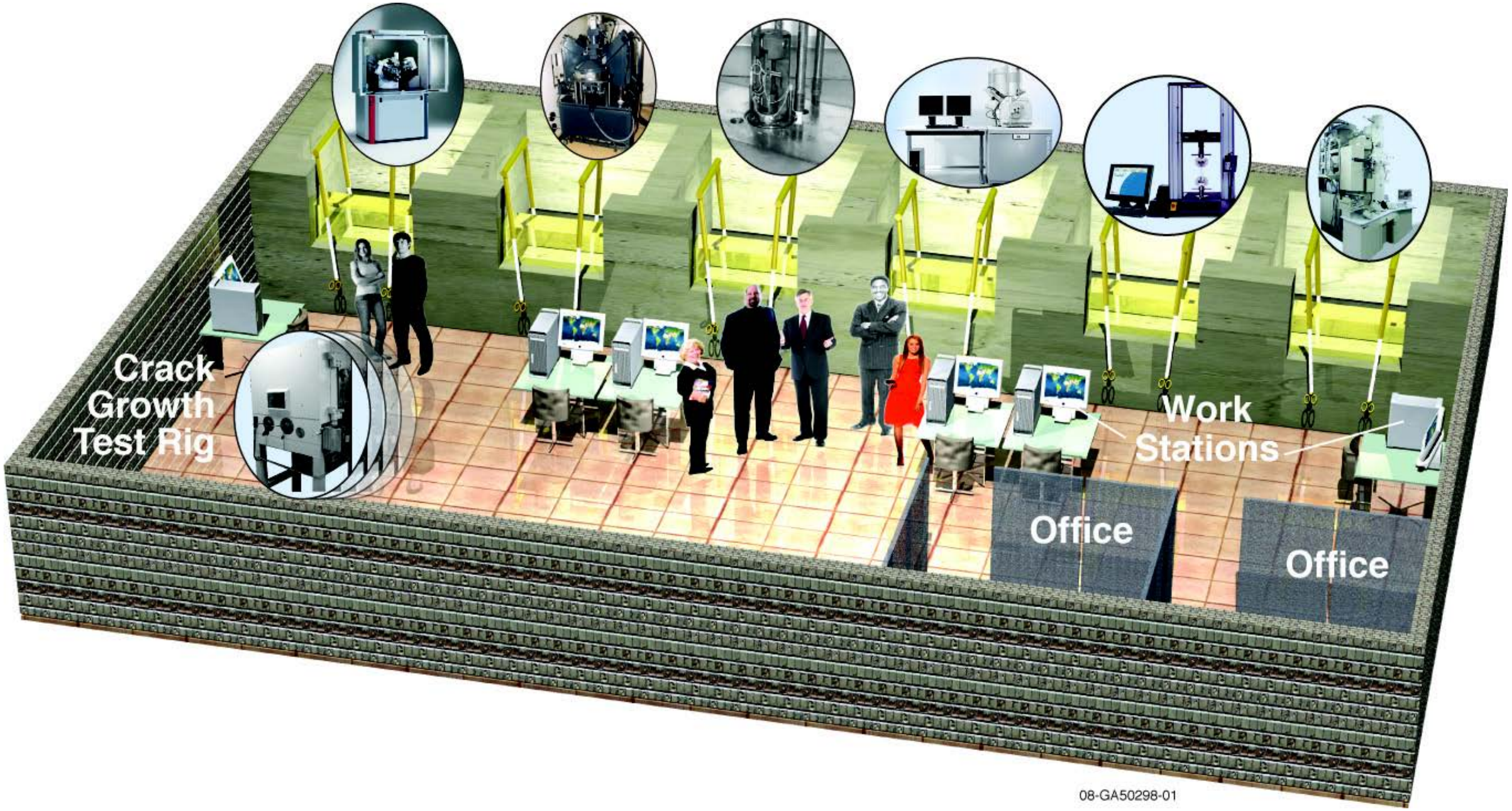
Topics

- 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)

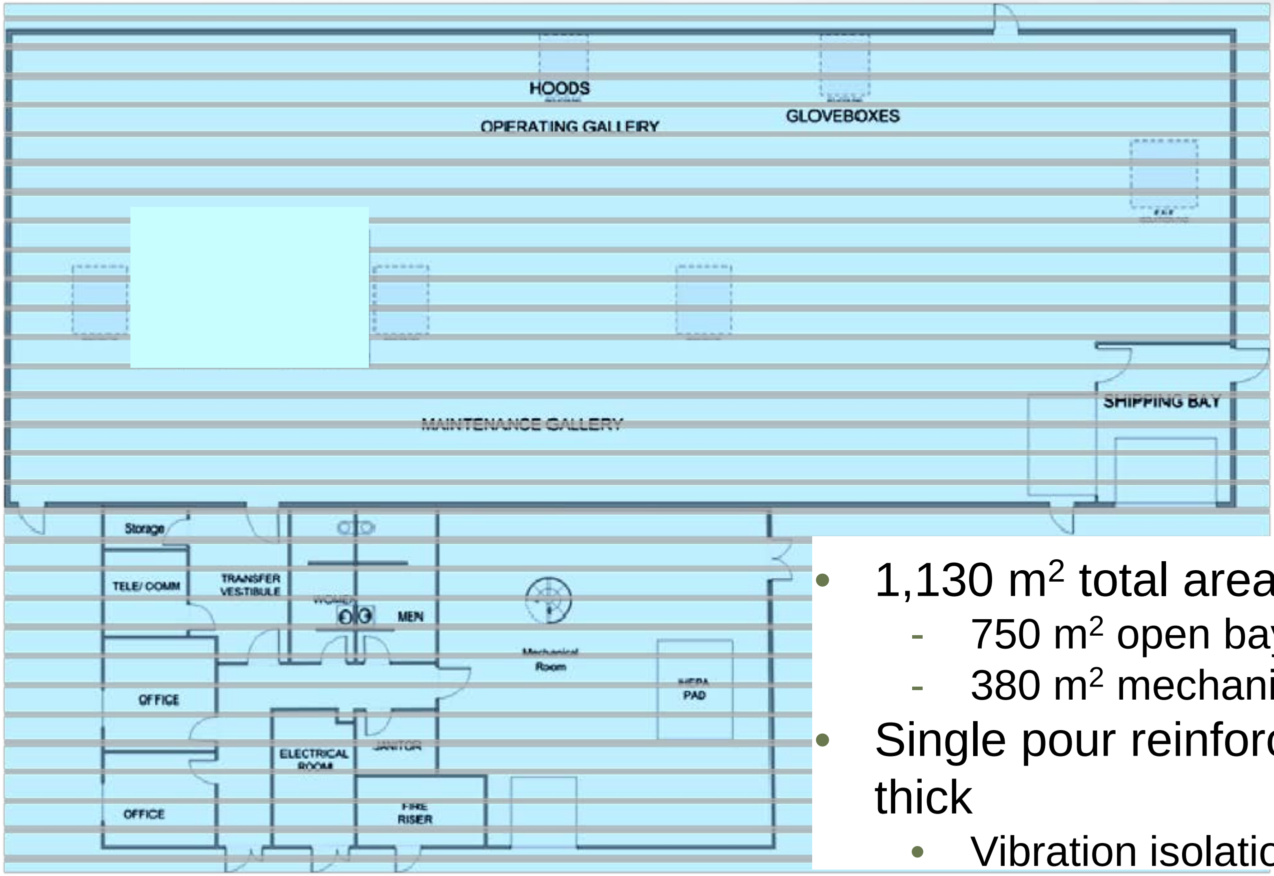


- Idea first presented in 2008 to support Nuclear Science User Facility
- Micro XRD, EPMA, IASCC, FIB, mechanical testing, and TEM

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 m^2 total area
  - 750 m^2 open bay laboratory
  - 380 m^2 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 August 2018

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)

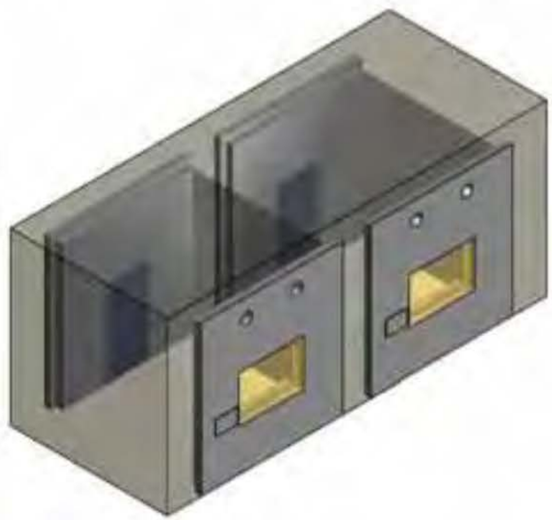
x Acoustic noise higher than instrument specification
  - Noise level above 55 dB below 2 kHz caused by ventilation system
  - Slight impact on instrument performance
  - Some impact on human performance

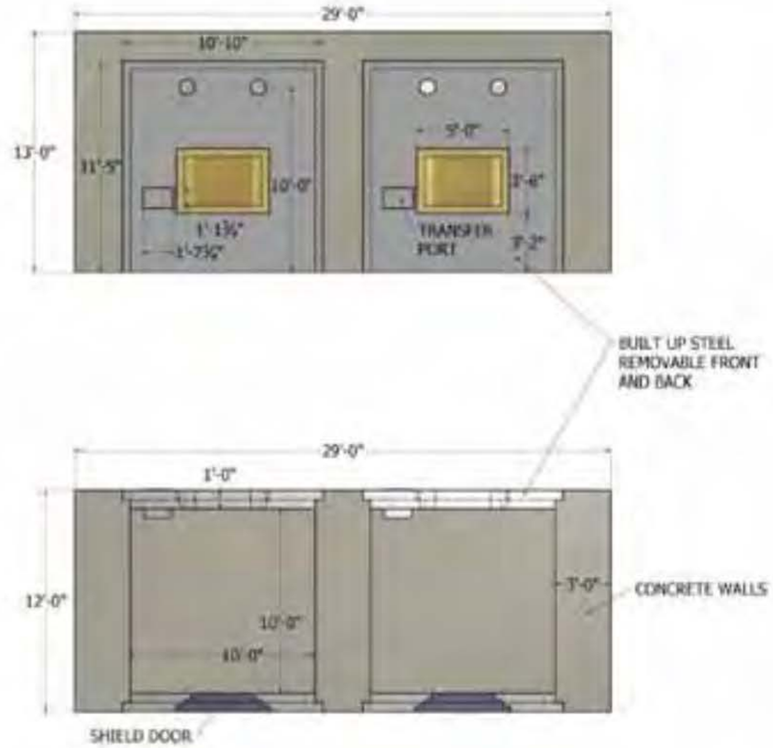
\- Asynchronous electromagnetic noise slightly out of limits

- 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

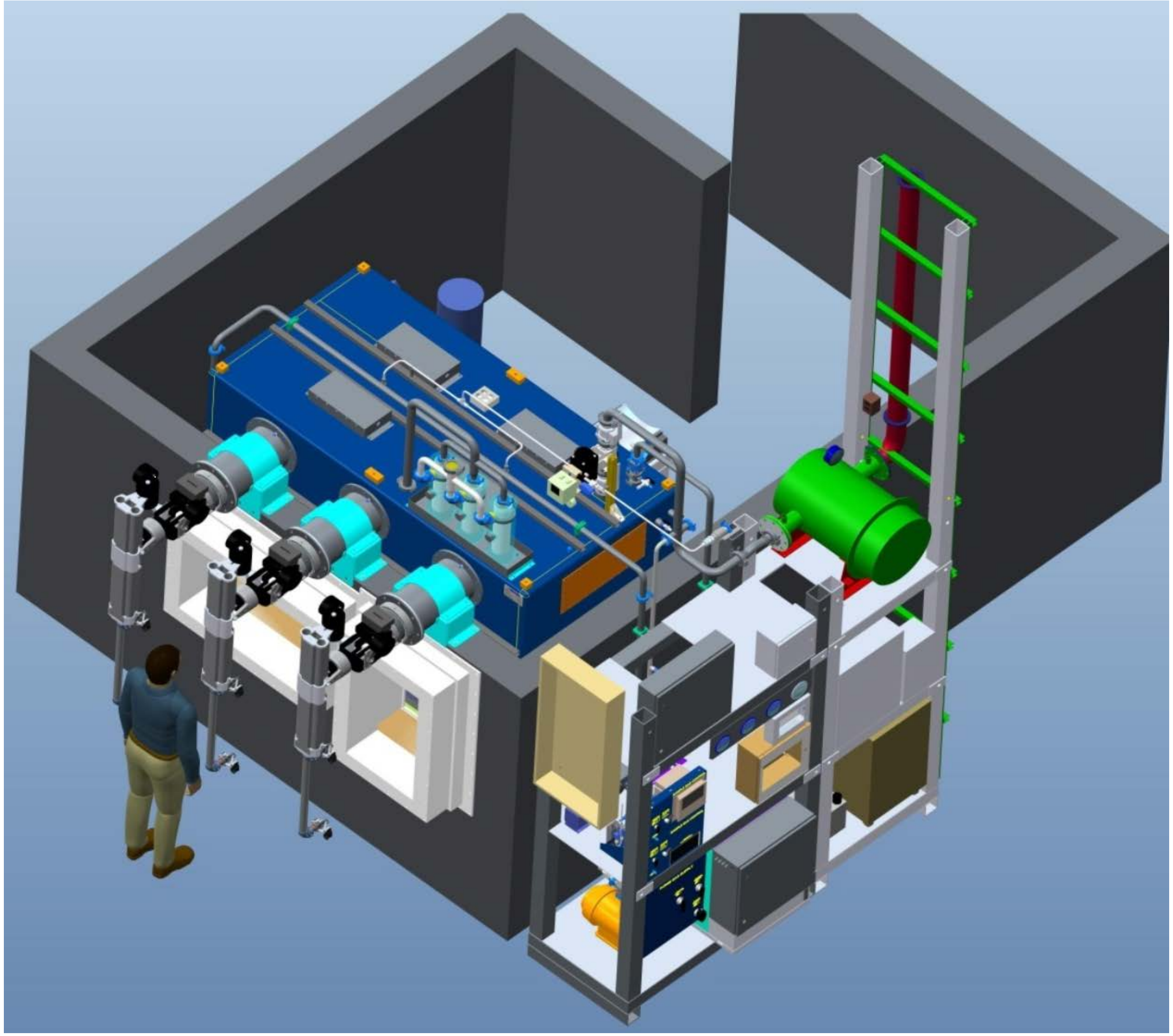
Shielded Cell Design Concept (2010)





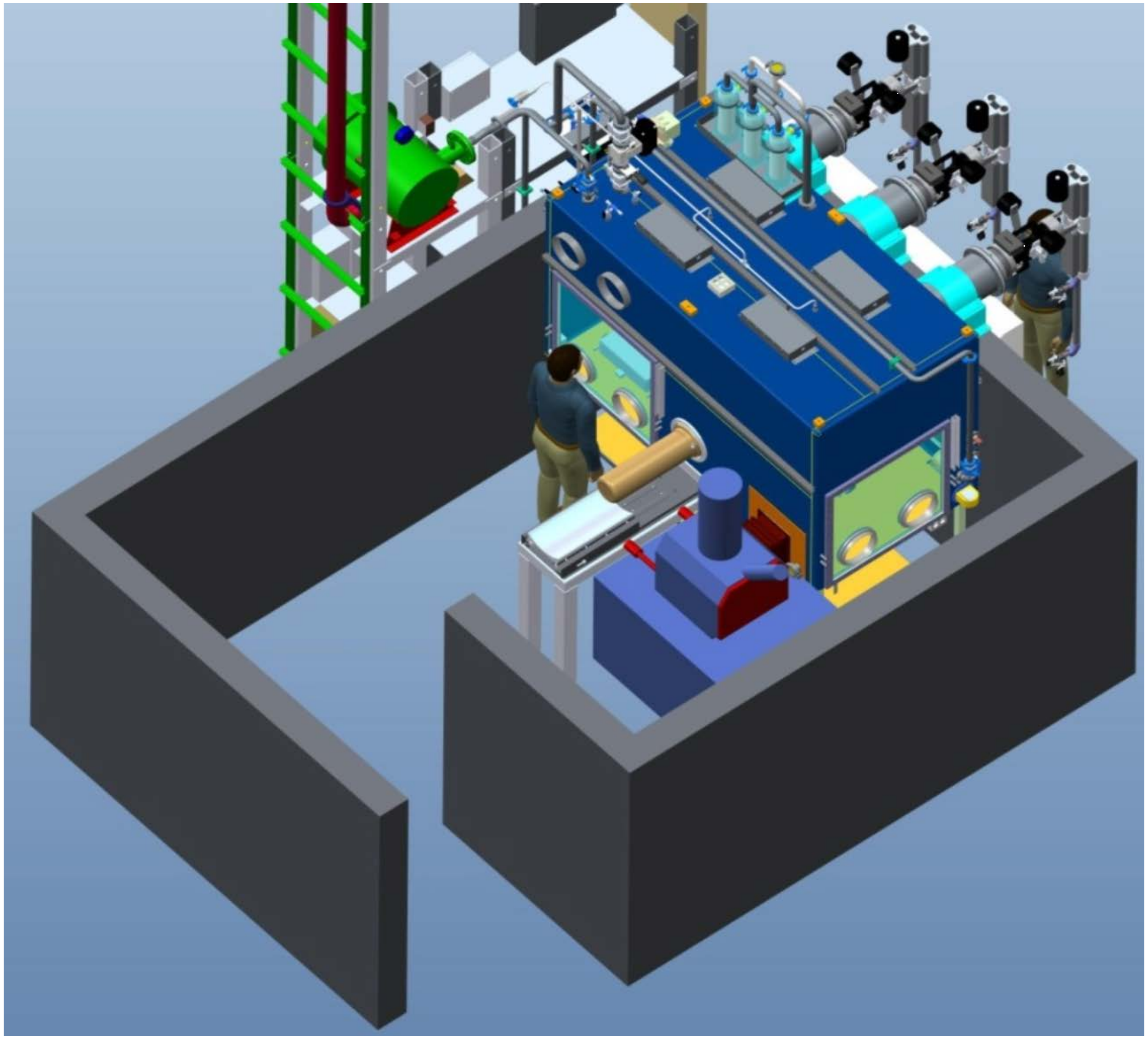
- Conventional concrete cell ~1 m (3') wall thickness with removable steel front and back walls and rear entry door
- Typical of 1960's cell design currently in use in U.S.
- Difficult to maintain modern instruments using this type of cell

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 γ shield walls
- Leaded glass windows
- Manipulators penetrate glovebox front wall
- No roof on cells
  - Simplify installation of piping, electrical, and instrumentation
  - Seismic issues

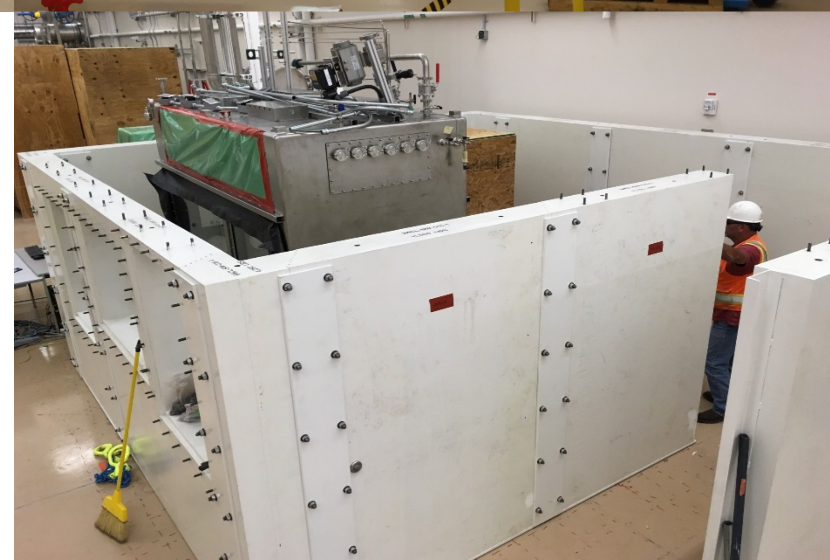
Final Shielded Cell Design



- 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

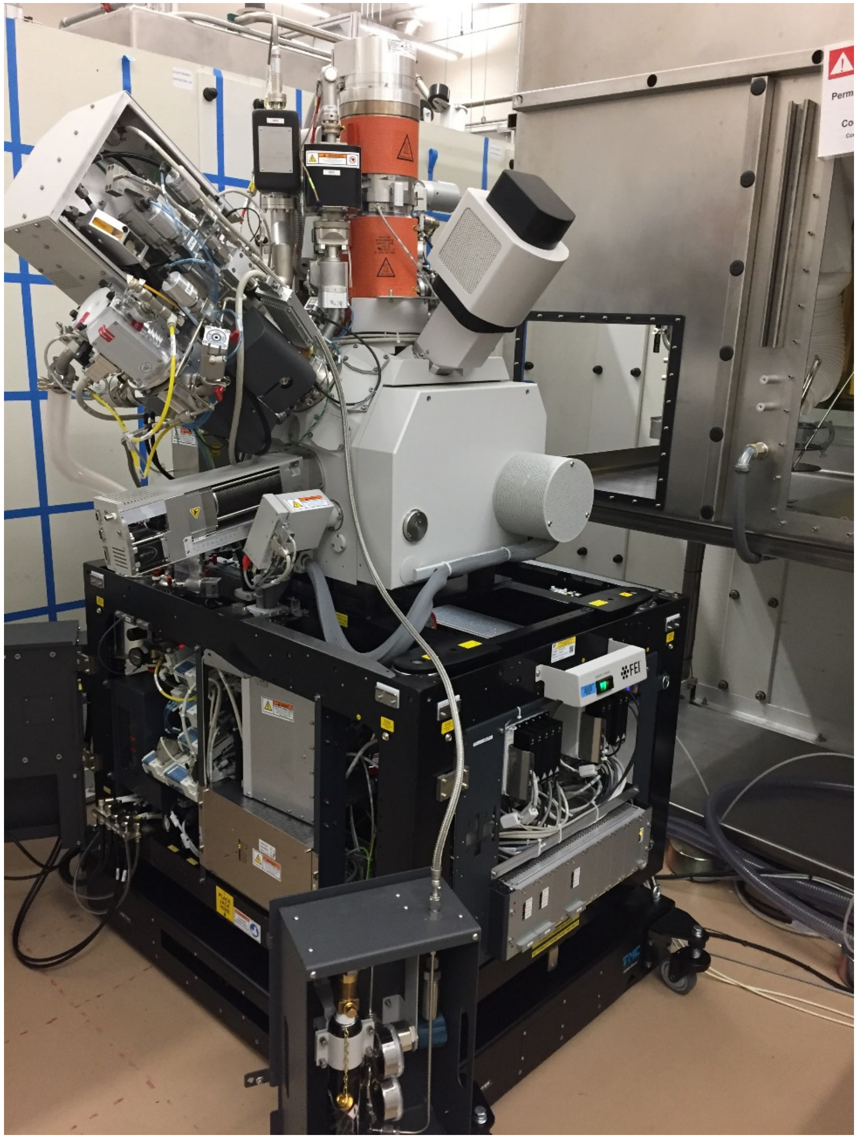
Shielded Cell Design

Shielded Cell Installation

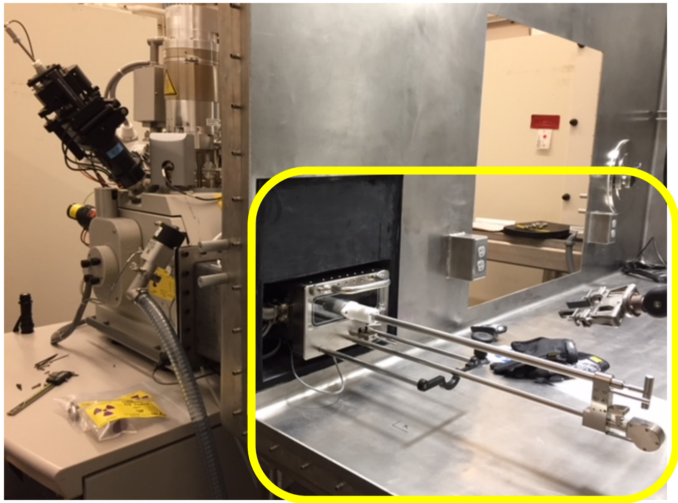


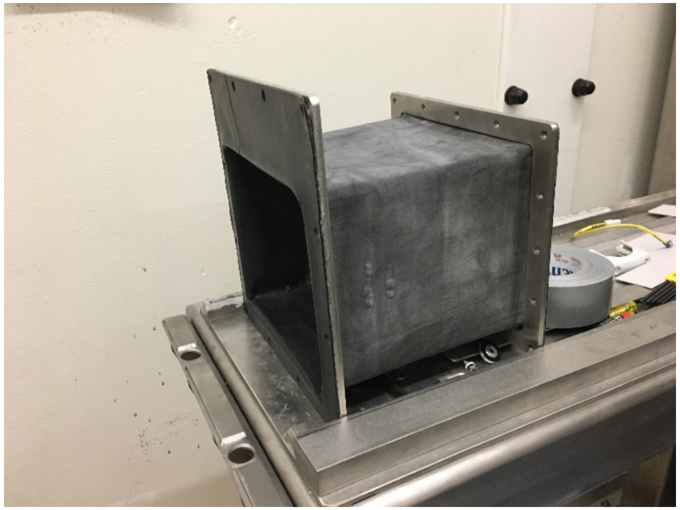
- Overhead crane not included in facility design (affordability)
- Shield panels (3~4 MT) installed using 20 ton mobile crane
- Future cell reconfiguration will require portable gantry crane

FIB Loading System

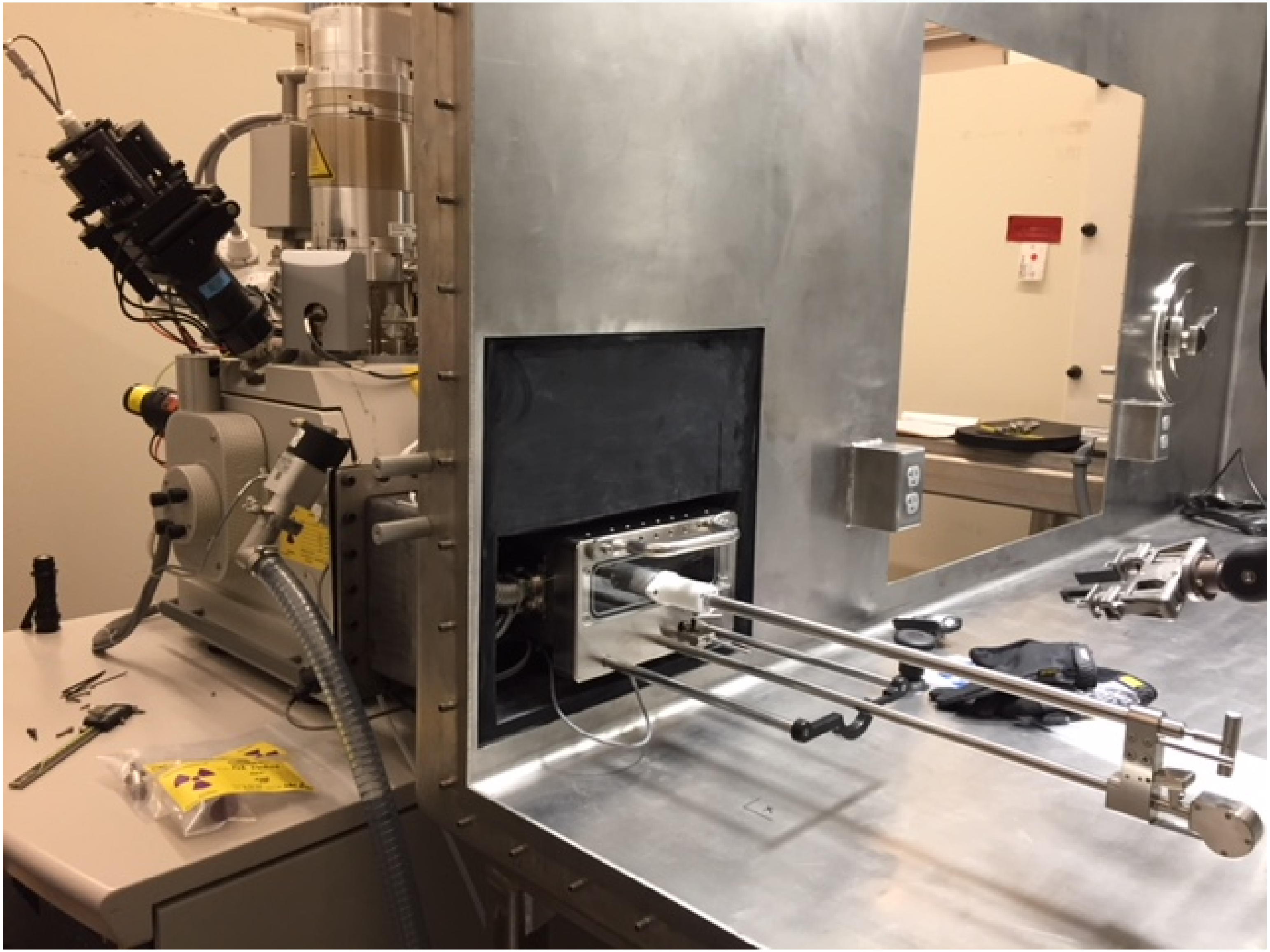


- Instrument loading through custom sample transfer system

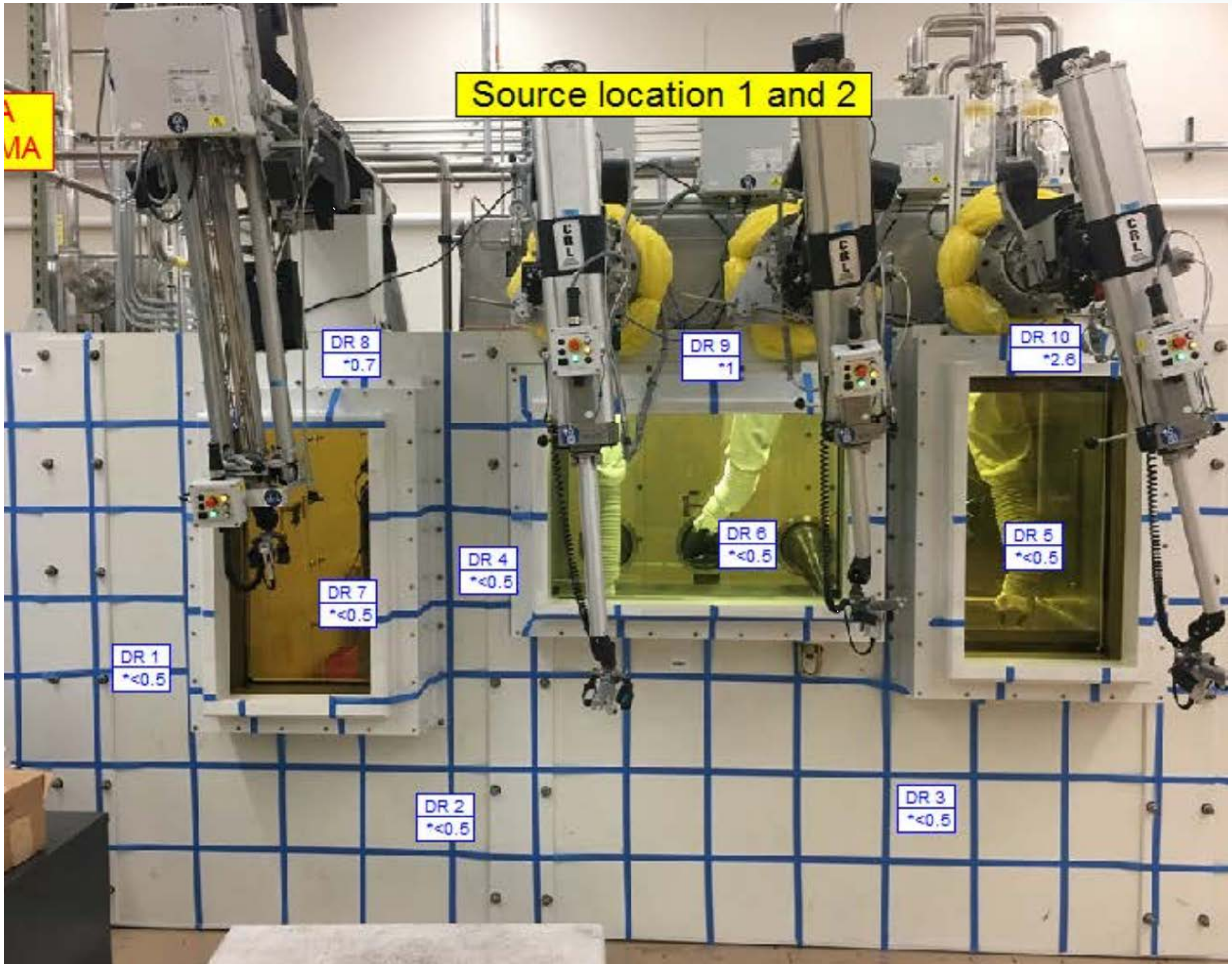




FIB Loading System

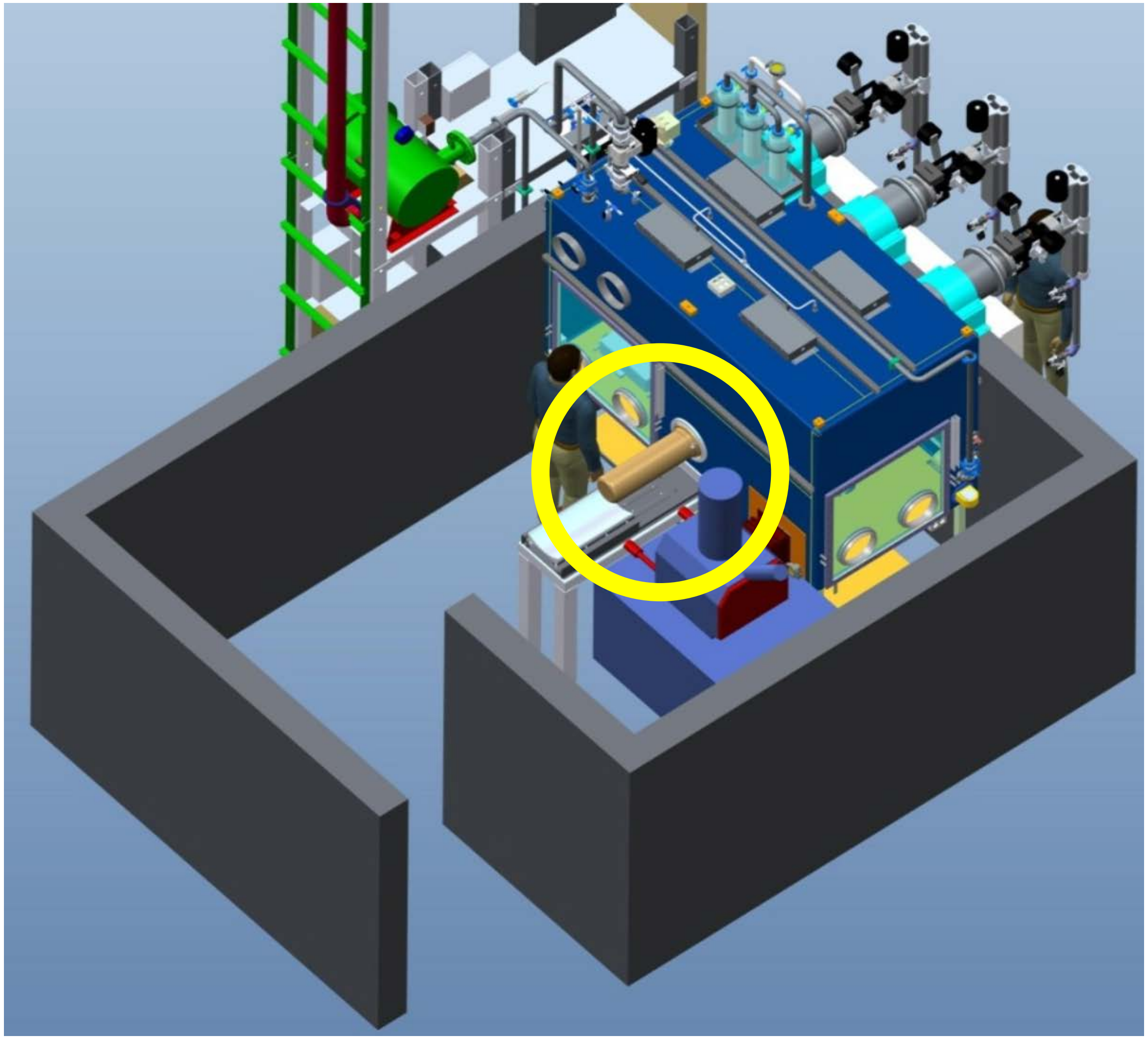


Shielding Performance



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

Shielded Transfer Container

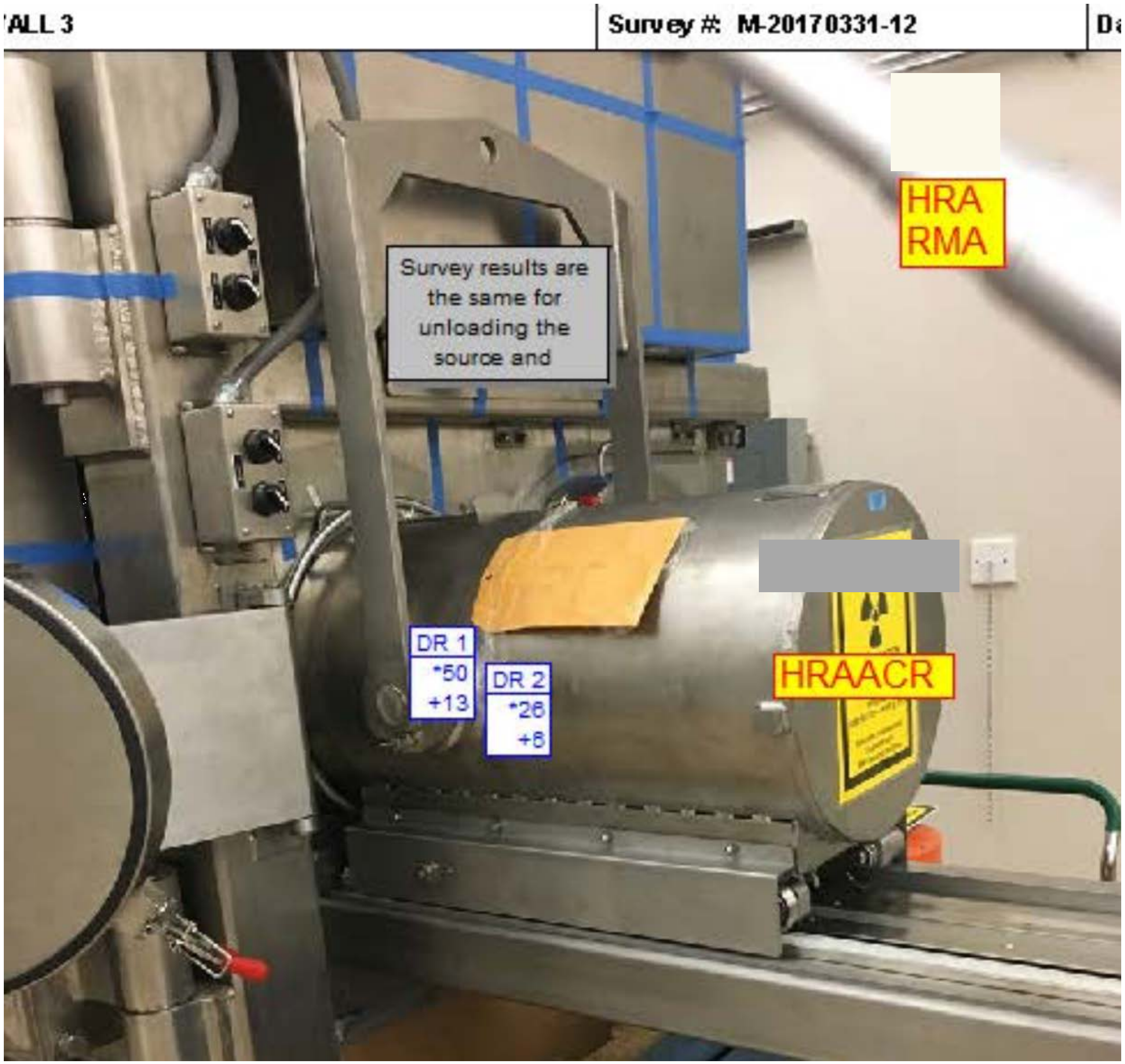


- Cask mates to back wall of glovebox
- Clean rapid transfer port (alpha/beta port) is mechanically interlocked to mating port on the cask. Neither container nor glovebox port will open unless the container is ‘locked’ to the glovebox
- Lightly shielded transfer containers also used

Shielded Transfer Container

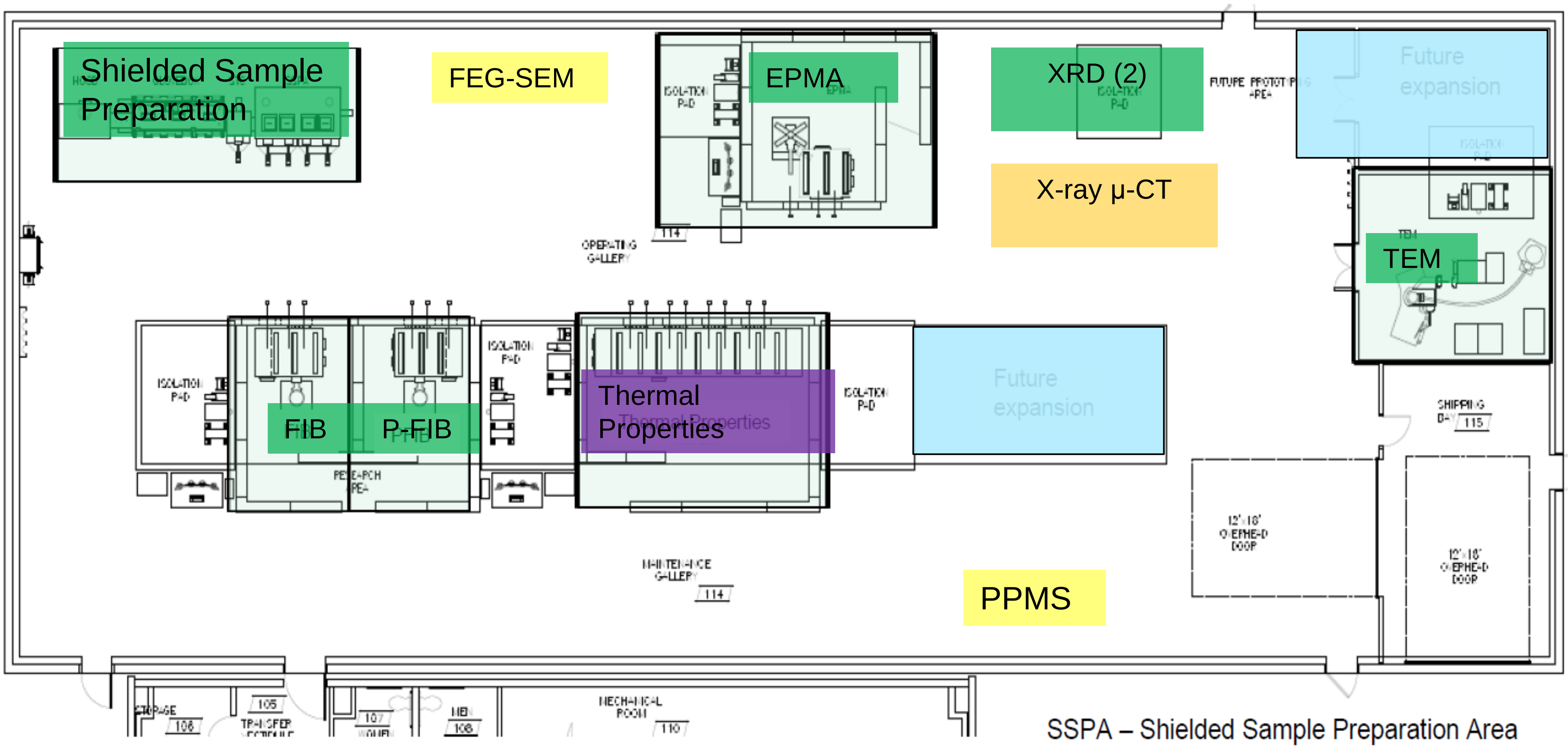
- Shielded Transfer Container (STC) mates to shielded instrument cells, Shielded Sample Preparation Area, and air hood
- 100 cm (4 “) of lead shielding
- Interior cavity ~10 cm dia. x 25 cm long
- Total weight ~1950 kg
- Hydraulic adjustment of STC height to match shielded cell cask stand height
- Will 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 occurence of alpha port sticking closed in several hundred uses

IMCL Capabilities



SSPA – Shielded Sample Preparation Area
EPMA – Electron Probe MicroAnalysis
FIB – Focused Ion Beam
TEM – Transmission Electron Microscope
TPTC – Thermal Properties Test Cell

Fully operational

In progress

Procured, awaiting installation

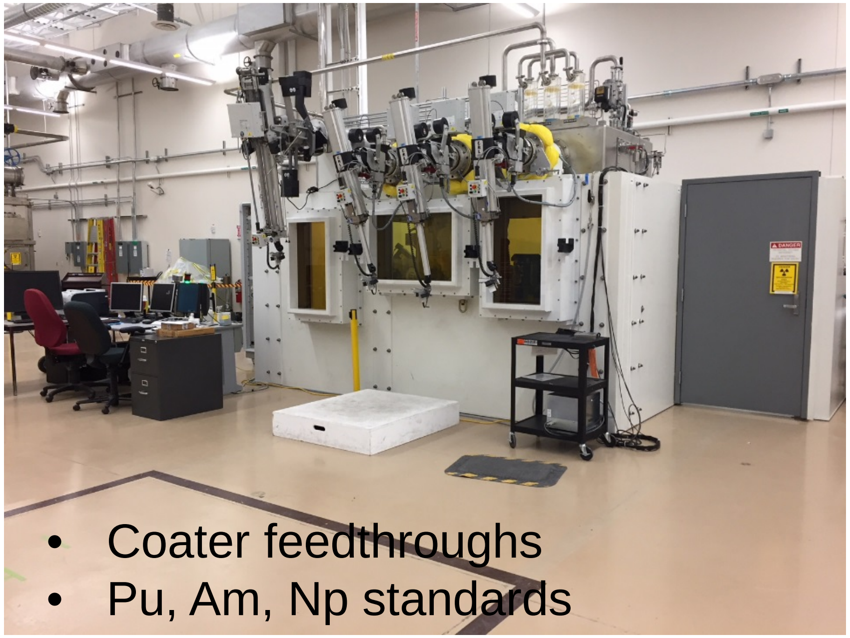
Future expansion

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

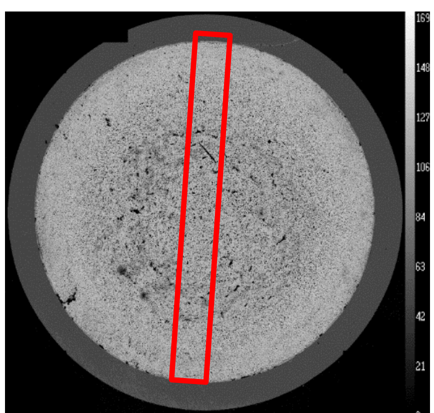
Operational in August 2017

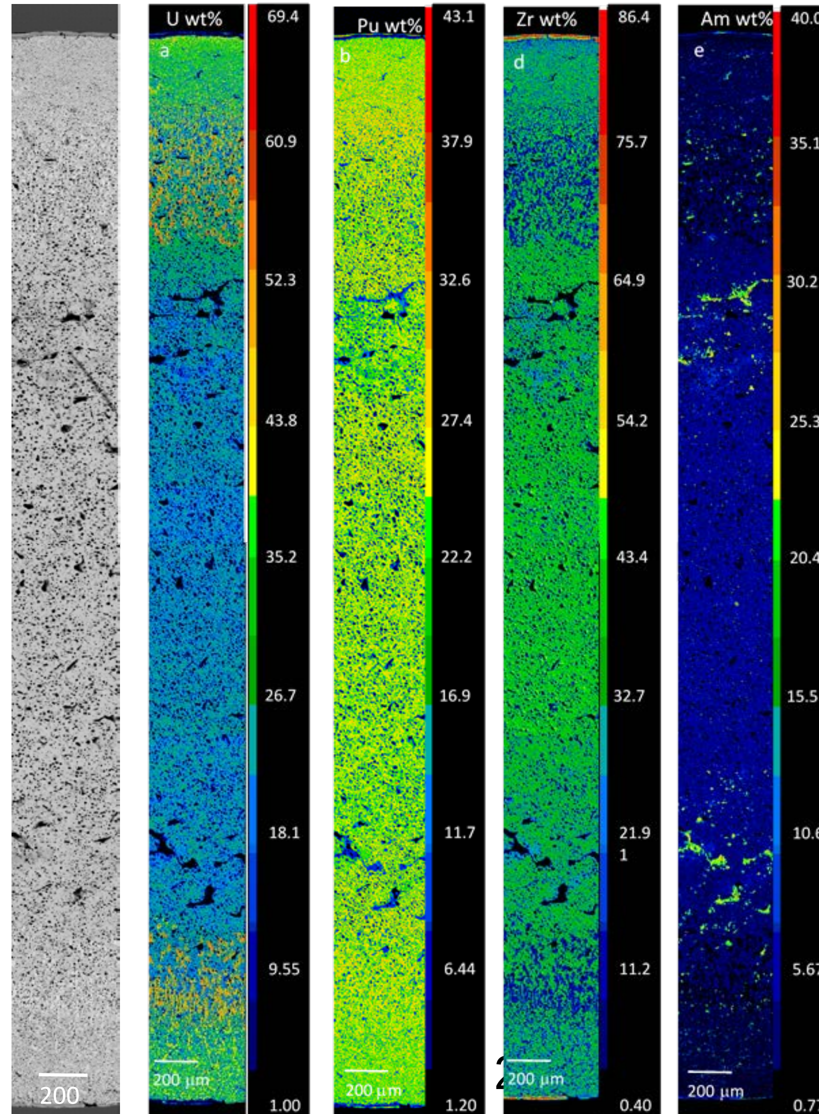
Electron Probe MicroAnalysis (EPMA)



- Coater feedthroughs
- Pu, Am, Np standards

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

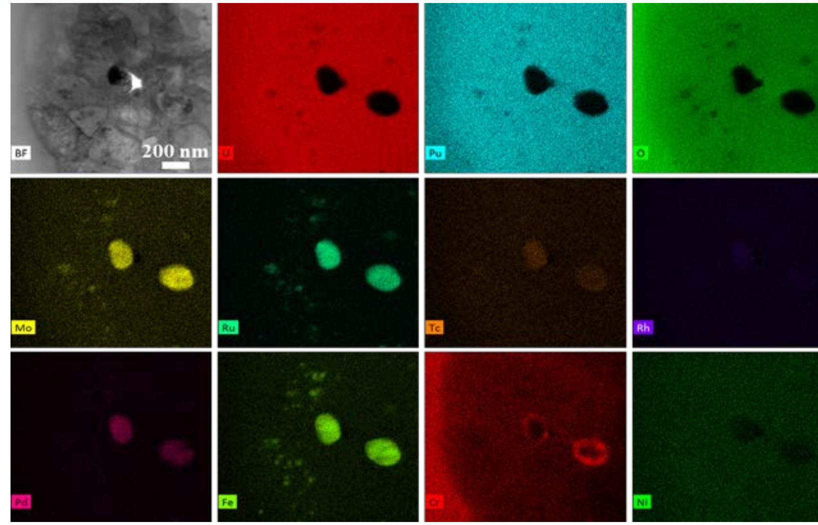




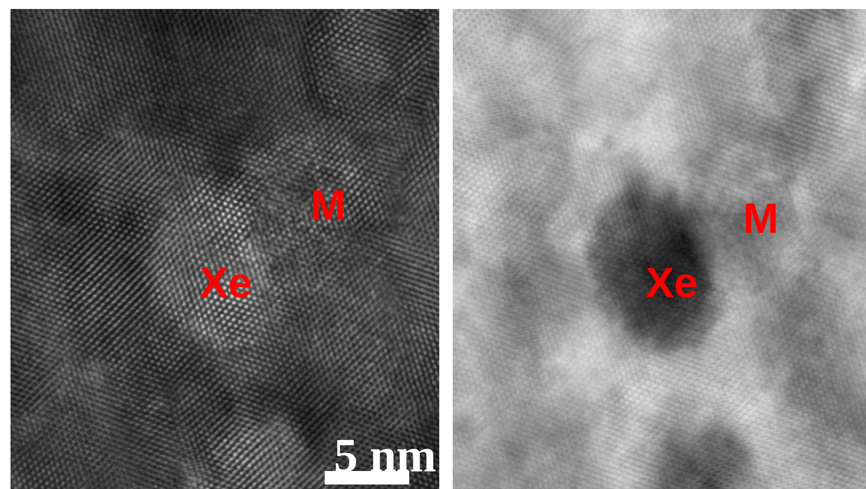
Operational in August 2017

Transmission Electron Microscopy

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

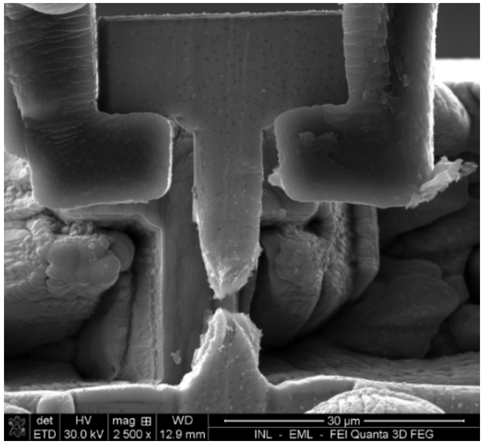


Top: Microstructure of MOX fast reactor fuel irradiated to 112 GWd/tHM. Bottom: Atomic resolution imaging of 5 metal precipitates and fission gas bubbles in UO_2.

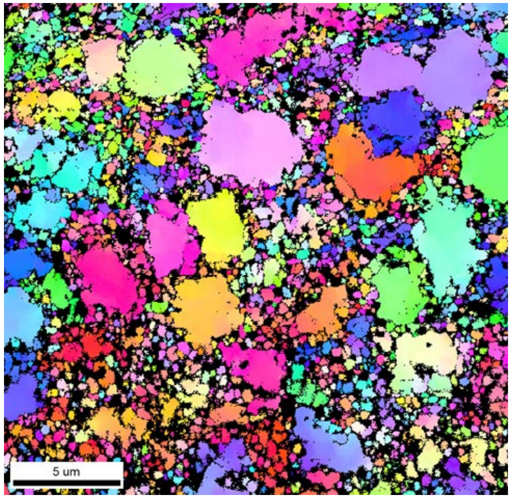


Dual Beam Focused Ion Beams

- 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



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



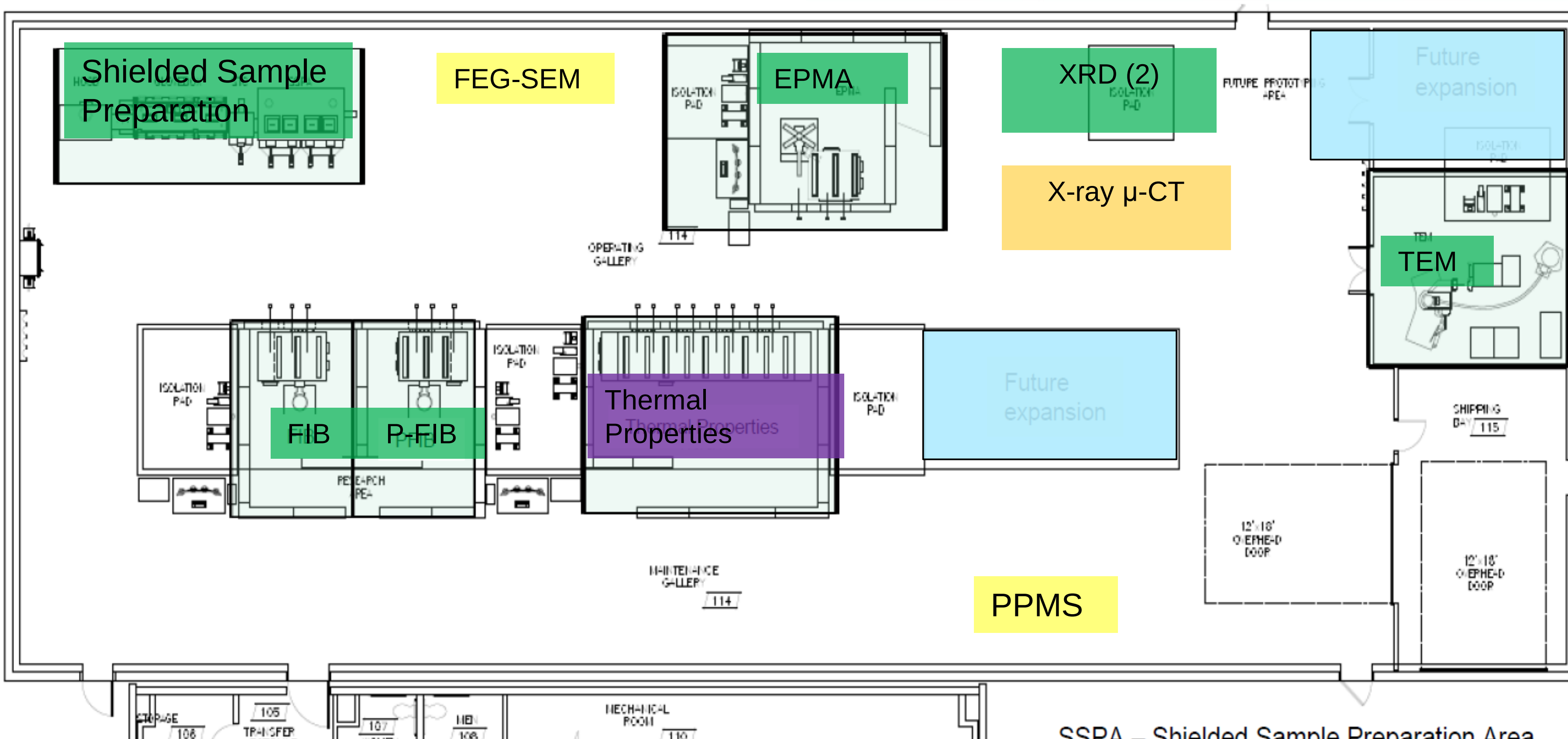
Inverse pole figure map of irradiated U-Mo showing the formation of a fine fine grain (240 nm) structure. Surface cleaned with Helios P-FIB

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

In progress

Procured, awaiting installation

Future expansion – user specified

SSPA – Shielded Sample Preparation Area
EPMA – Electron Probe MicroAnalysis
FIB – Focused Ion Beam
TEM – Transmission Electron Microscope
TPTC – Thermal Properties Test Cell

Additional Capabilities

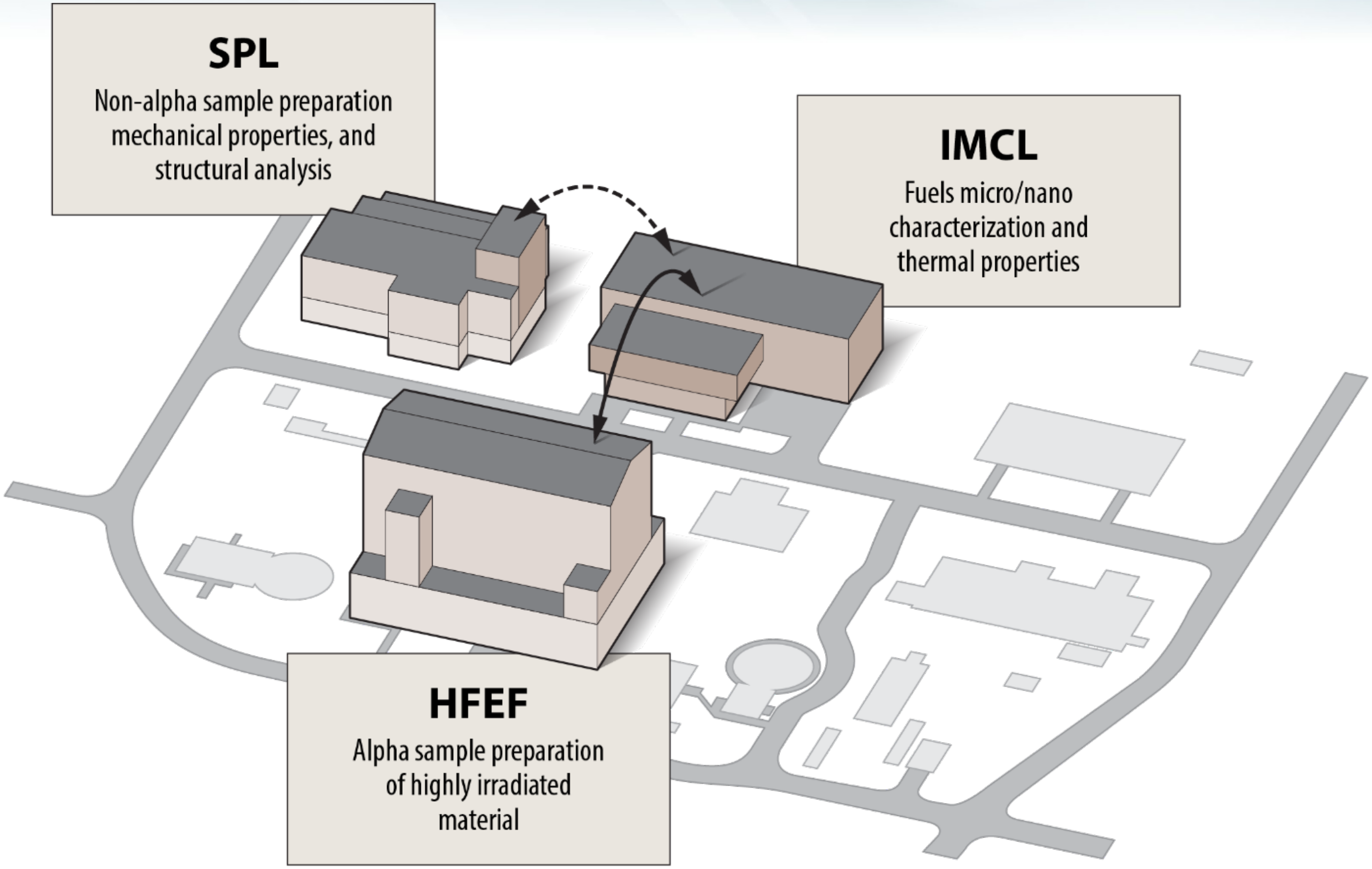
Quantum systems Dynacool-9 PPMS
- 2 - 400 K
- 9 Tesla field
- Electrical, magnetic, and thermal property measurements
- January 2019

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

- Bruker D-8 µ-XRD, 50 µm resolution
- Panalytical Empyrean powder XRD with heated stage
- Unshielded
- Currently installed

- 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

Looking Forward



- 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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