INL/CON-17-42391-Revision-0

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

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September 2017

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Idaho National Laboratory
Idaho Falls, Idaho 83415

http://www.inl.gov

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Dr. B. D. Miller

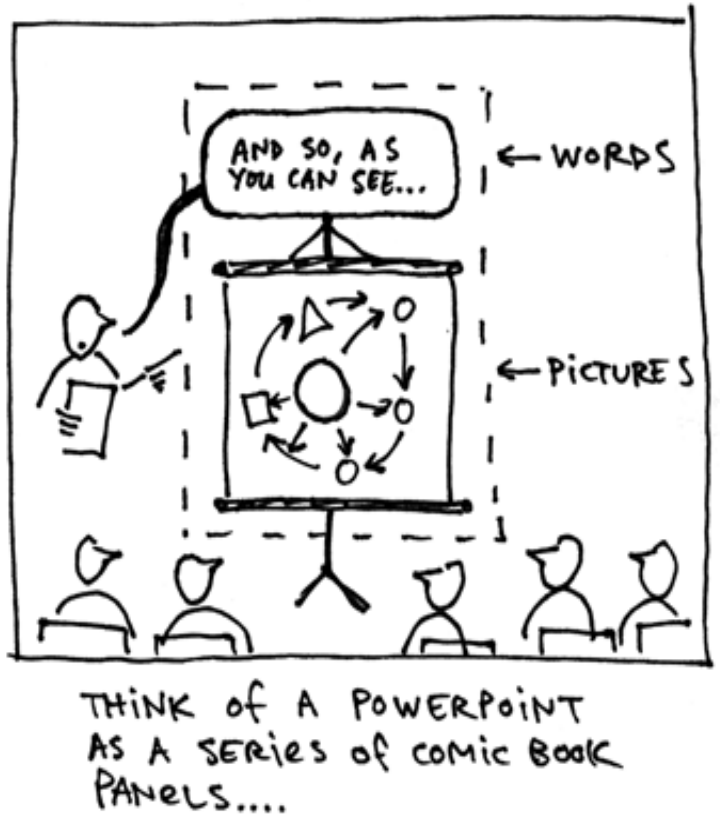
Idaho National Laboratory

Sept. 2017

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



http://open.lib.umn.edu/businesscommunication/chapter/11-4-visual-aids/

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)**

Outside of IMCL at INL

Researcher operating the EPMA

Design Basis of IMCL

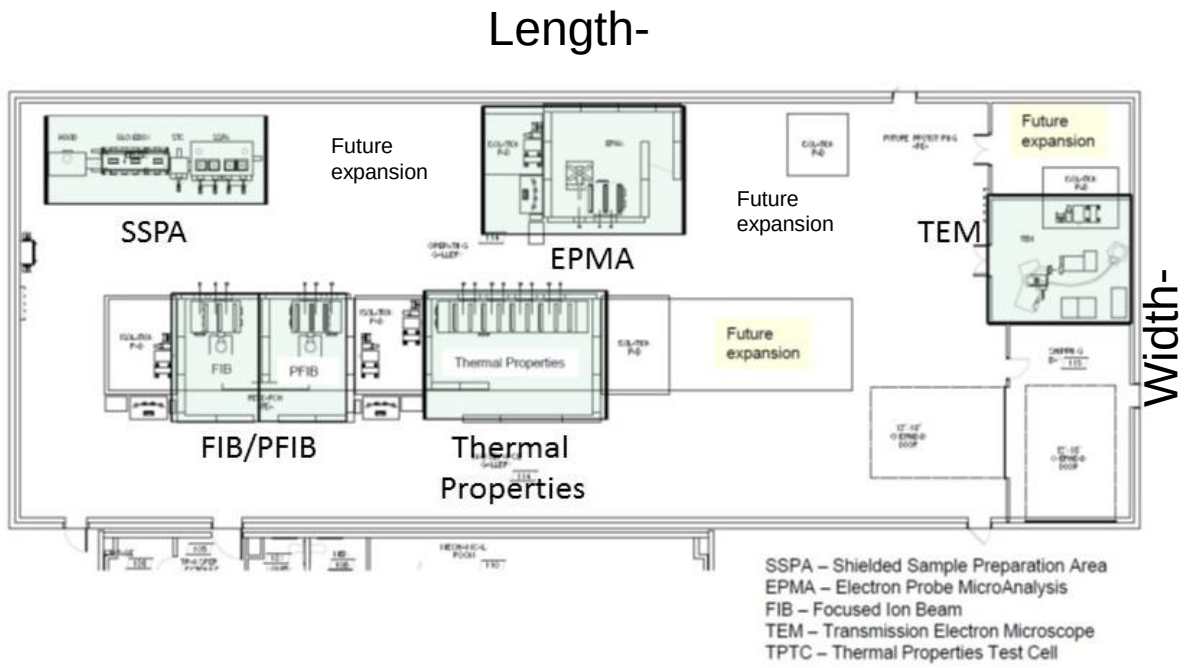
- **Low vibration**
  - Floor designed as a single concrete slab with isolation pads for vibrating equipment
- **Temperature control**
  - Less than 1°C per hour
- **Low electromagnetic interference**
  - As to not interfere with operation of high-end electron microscopes

"Dang...the thermostat is broken. It looks like it's going to be a cold day in Hell."

https://www.google.com/url?sa=i&rct=j&q=&esrc=s&source=images&cd=&cad=rja&uact=8&ved=0ahUKEwjXstKI76XWAhUC1mMKHfAEARIQjRwIBw&url=https%3A%2F%2Fwww.cartoonstock.com%2Fdirectory%2Ft%2Ftemperature_gage.asp&psig=AFQjCNETPTu3PTe5ugNKryHbTg3WHTaNIA&ust=1505519198452054

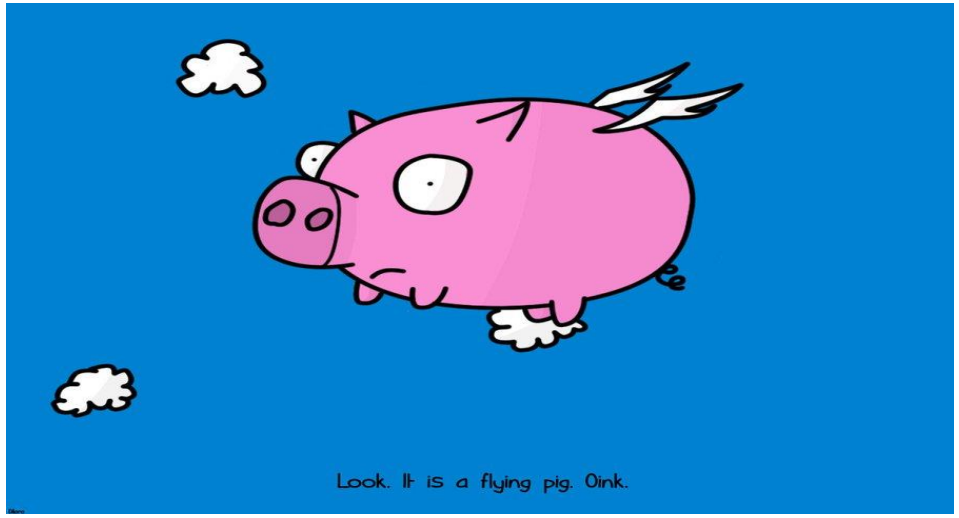
Current Layout of IMCL

- **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**



Flying Pig

IMCL shield cask (ISC)

ISC awaiting docking with the Shielded Sample Preparation Area

Shielded Sample Preparation Area-SSPA

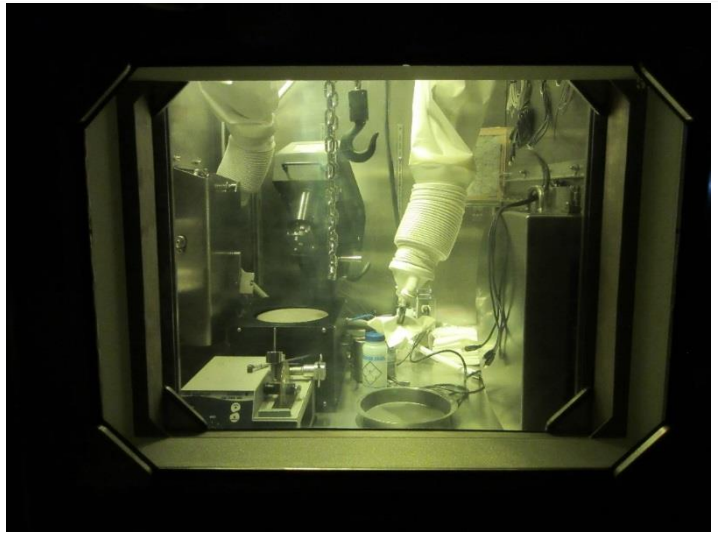
- **The SSPA's primary focus is sample preparation of highly radioactive materials/fuels**
- **All portions of the system are connected allowing easy transfer of samples**
- **Three shielded bays**
  - CRL Manipulators
    - Type L-HD
  - Lead equivalent of 21 cm steel
- **Radiological glovebox**
  - N_2 inert
  - Sample preparation of low dose samples
- **Radiological fume hood**
  - Decontamination activities
  - Sample preparation

Relaxing after a long day at the SSPA

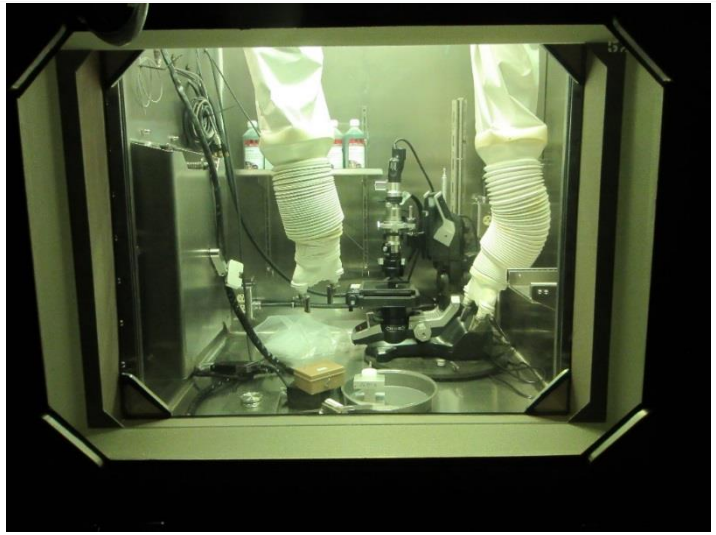
Various images of the SSPA line

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 preparation bay



Optical microscopy bay

Shielded transfer cell

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^{60} source**

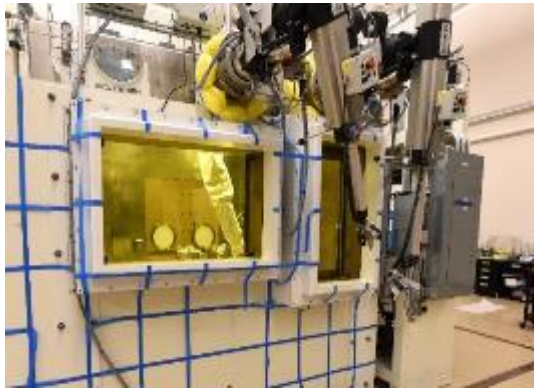
SAS layout for the Focused Ion Beam Microscopes

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**

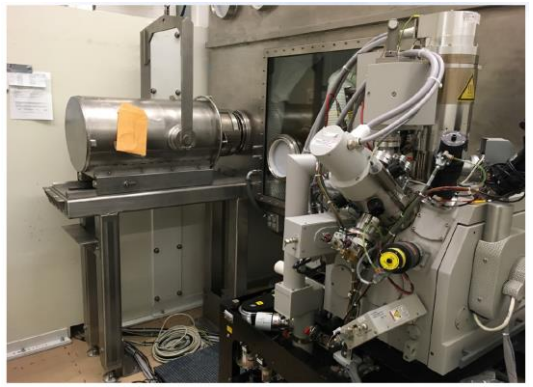




Images of the shielding in the FIB SAS's

View inside a SAS glovebox

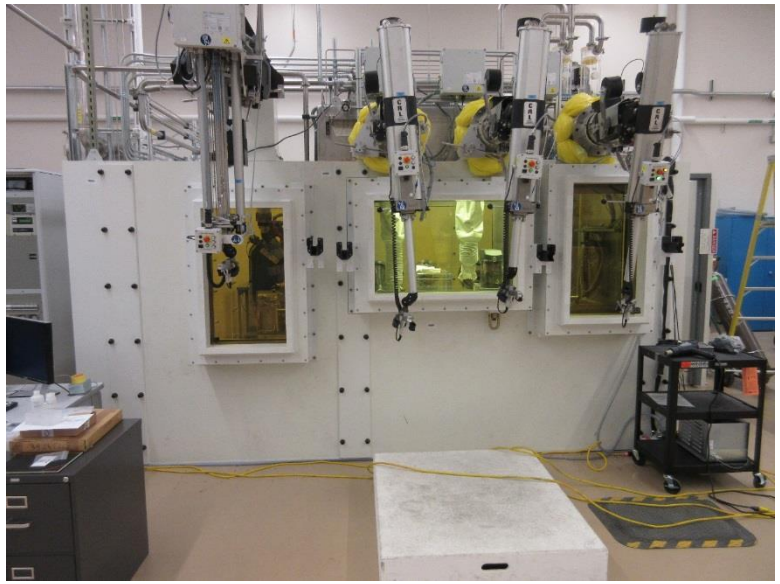
FEI Helios Plasma FIB



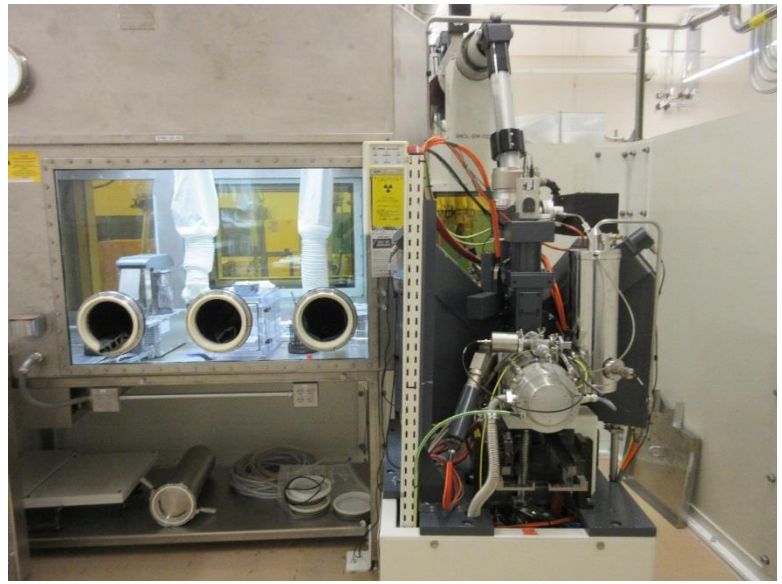
FEI Quanta 3D FIB

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**



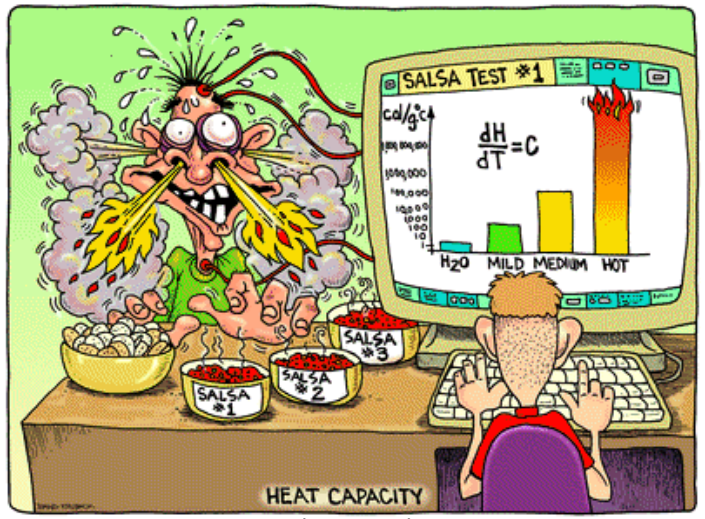
Outside of the EPMA SAS



Views from inside the EPMA SAS

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

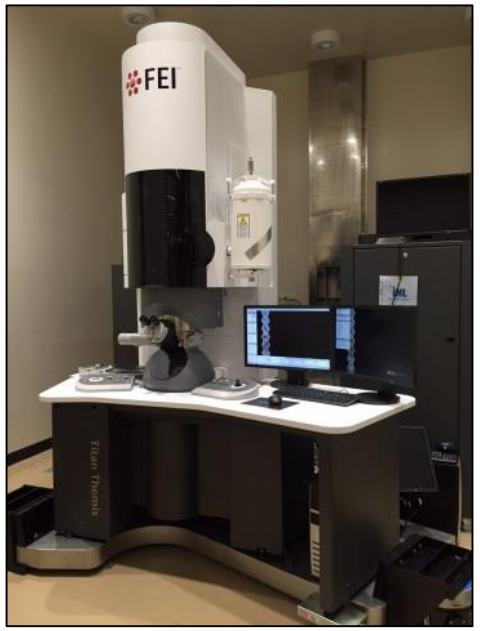


www.leapsecond.com

Thermal property SAS at the fabrication facility

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**

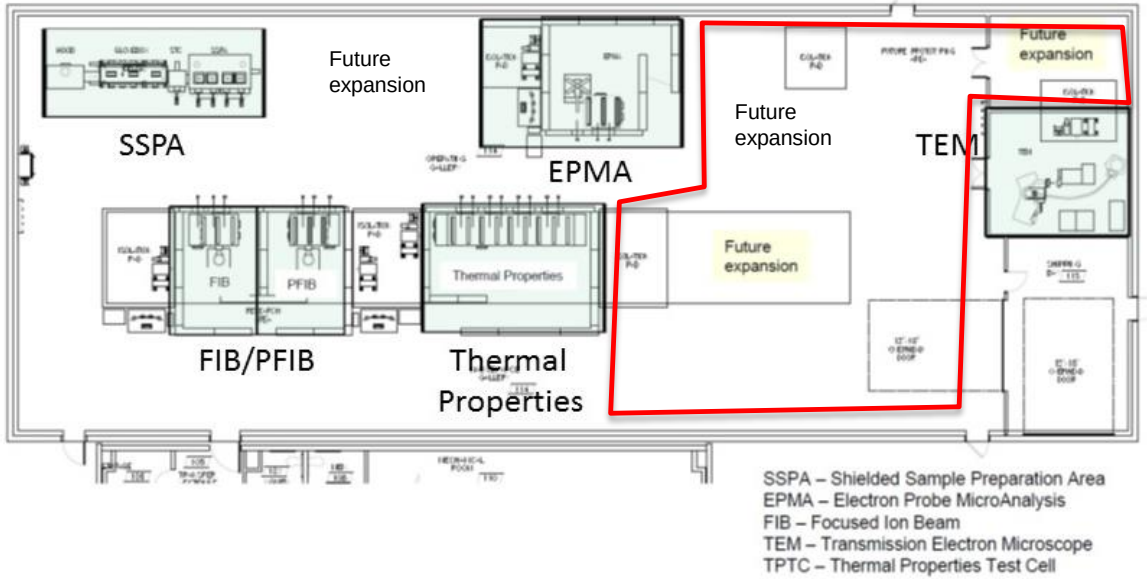


Titan TEM

TEM Room

Future Expansion?

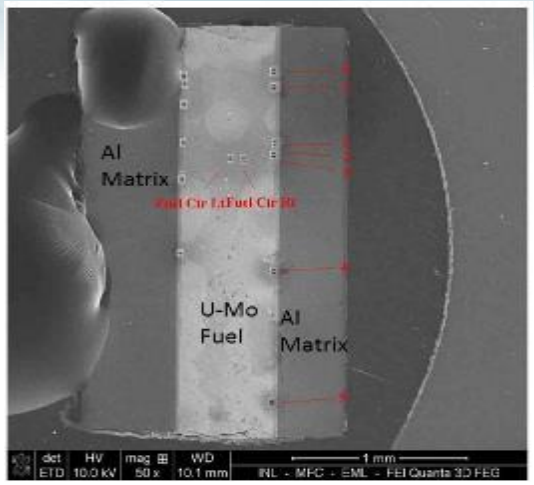
- **Options include:**
  - Atom Probe Tomography
  - Shielded Scanning Electron Microscope
  - Mechanical Properties Cell
  - Additional FIB's?



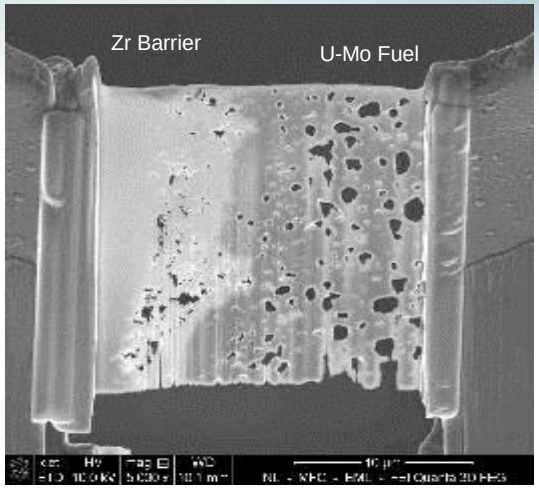
http://toonut.com/dog-house-expansion/

Post Irradiation Examinations-FIB

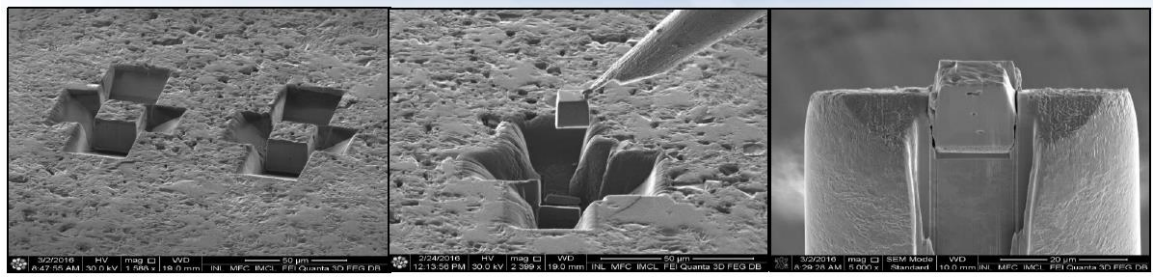
- **FIB/PFIB offer site specific characterization of materials on micron scale and below**
- **Capabilities of the FIB/PFIB include**
  - TEM lamella preparation
  - Cube preparation for serial sectioning and chemical profiling of specific regions
  - Cross-section milling for visualization of the microstructure under the polished surface
  - Electron backscatter diffraction surface preparation
  - and many others…….

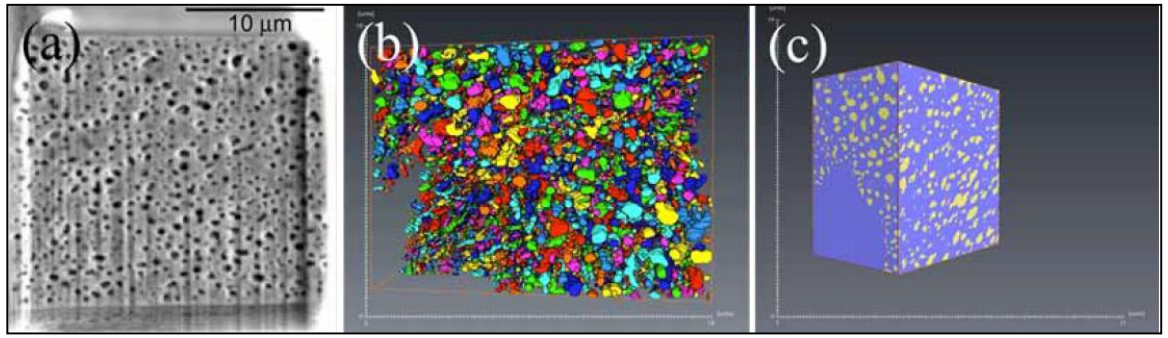


Site Specific Capability of the FIB



TEM lamella liftout of U-Mo fuel

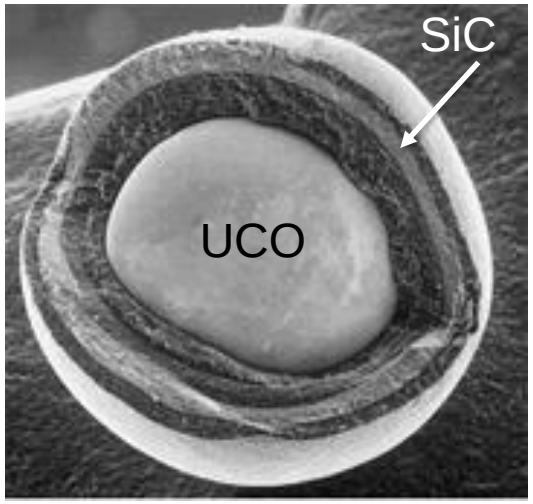

Cube liftout of irradiated UO_2 from BR3 reactor



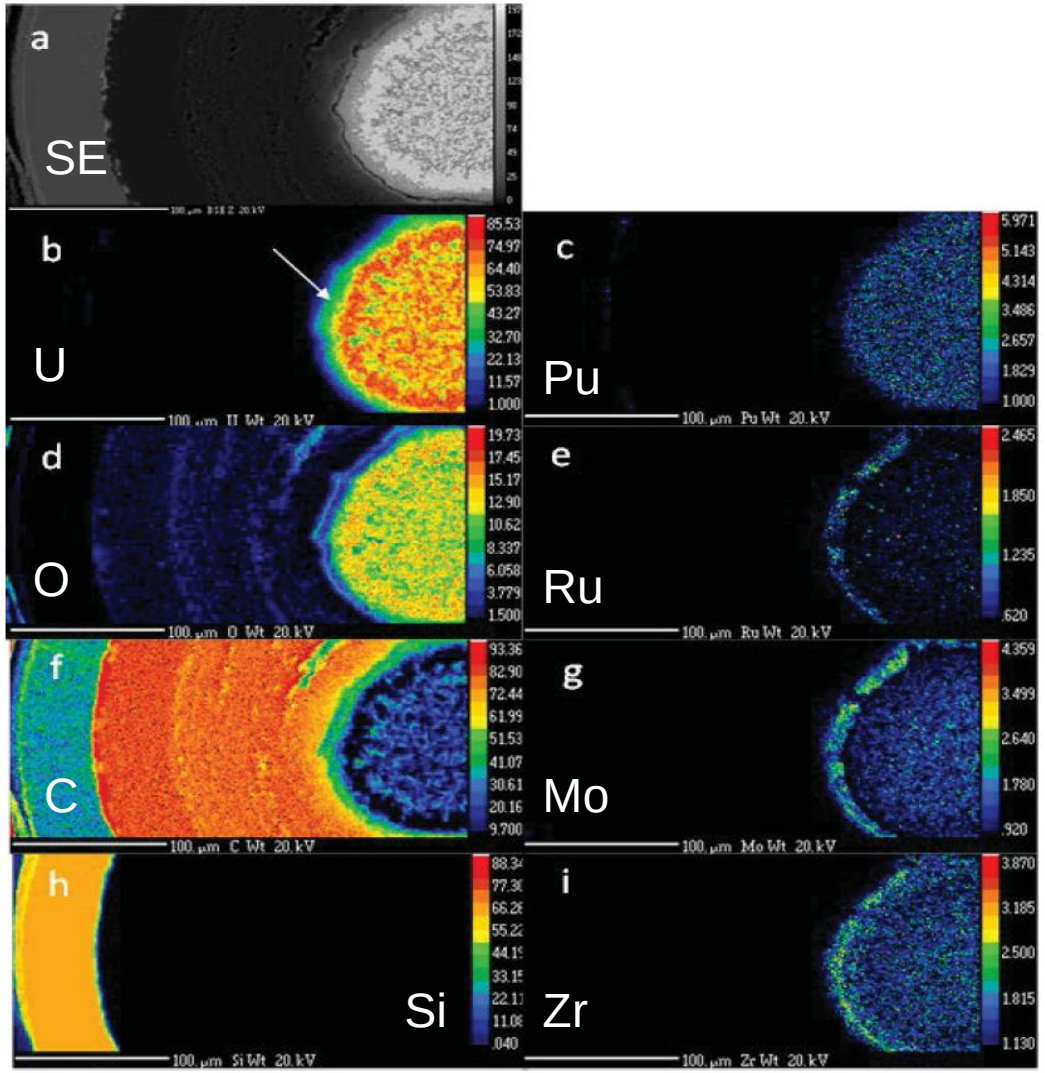
3D reconstruction of porosity in U-Mo fuel

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 partilce**



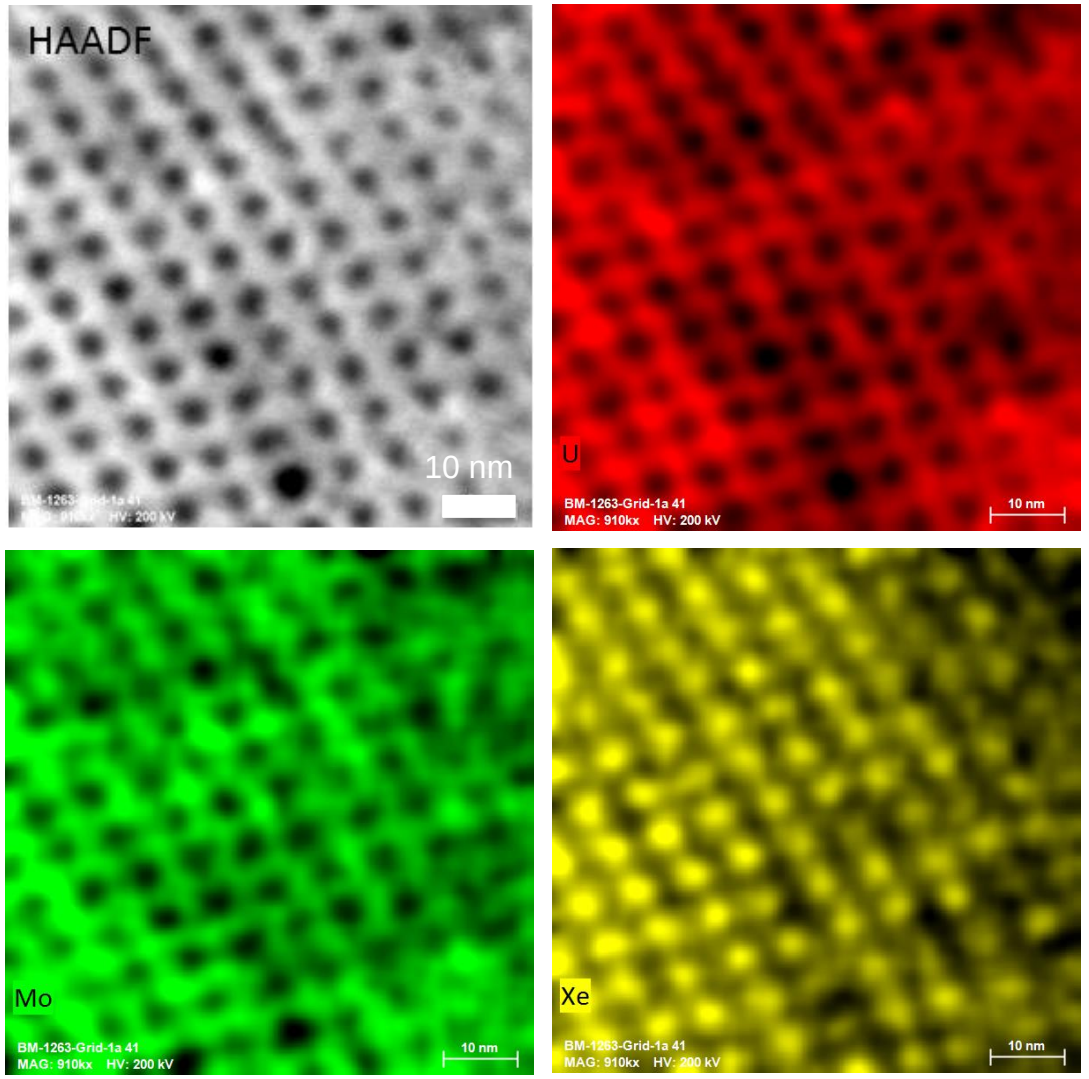
Cracked TRISO particle



WDS maps of a TRISO fuel particle

Post Irradiation Examination-TEM

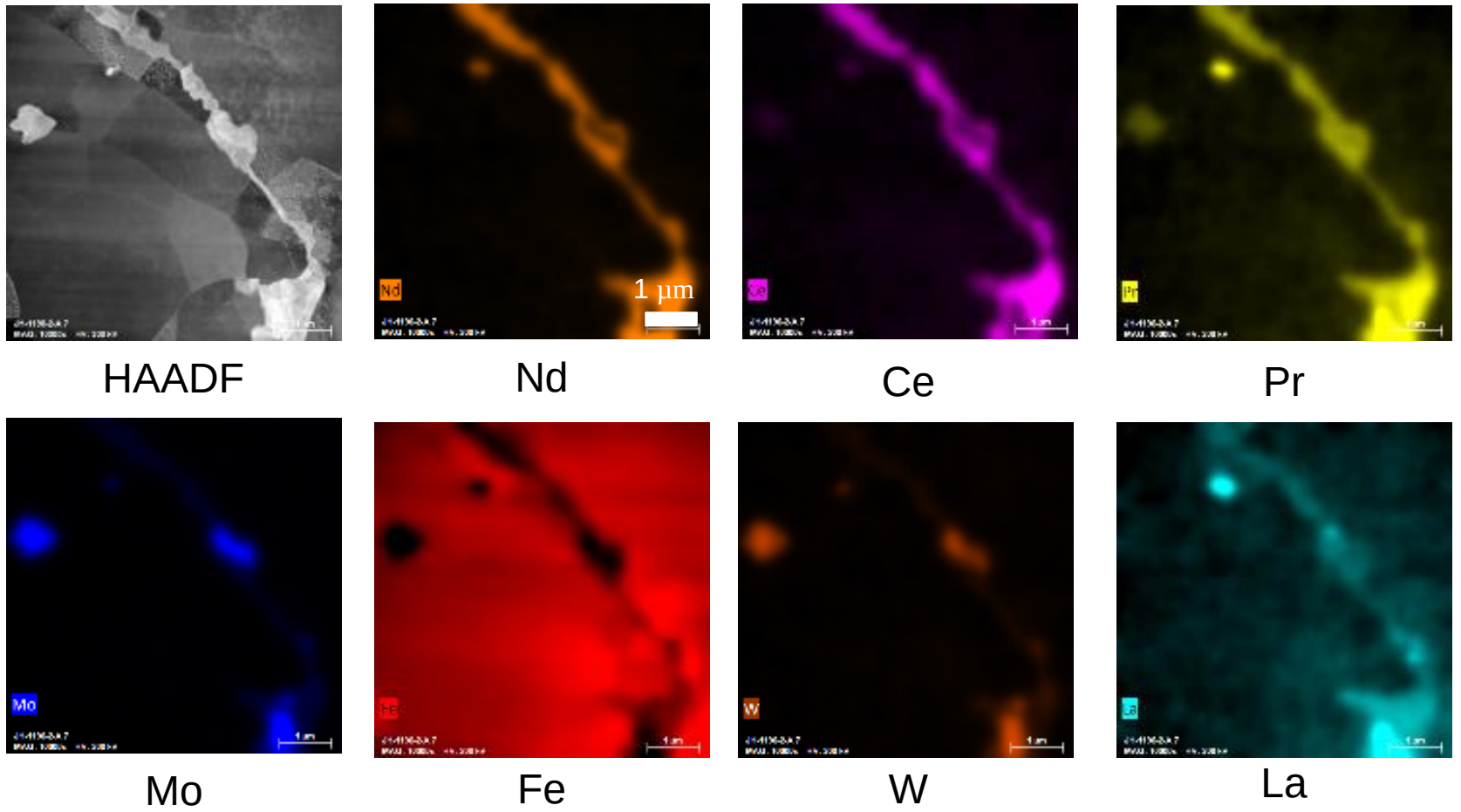
- U-Mo fuels being studied for use in research and test reactors throughout the world
- Forms ordered bubble superlattice at fission densities typically lower than $4.5x10^{21}$ fissions/cm^3
- It was assumed that the bubbles were stabilized by Xe fission gas
- Proof that Xe is indeed present in the 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



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!