



Creating a Data Collection and Processing Workflow for Three-Dimensional Slice-By-Slice Reconstruction of Nuclear Structural Materials Using Focused Ion-Beam Microscopy

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Advancement of the nuclear energy infrastructure through Generation IV reactor technologies

U.S. Energy Portfolio

“U.S. nuclear power plants are essential to achieving President Biden’s climate goals and DOE is committed to keeping 100% clean electricity flowing and preventing premature closures,”

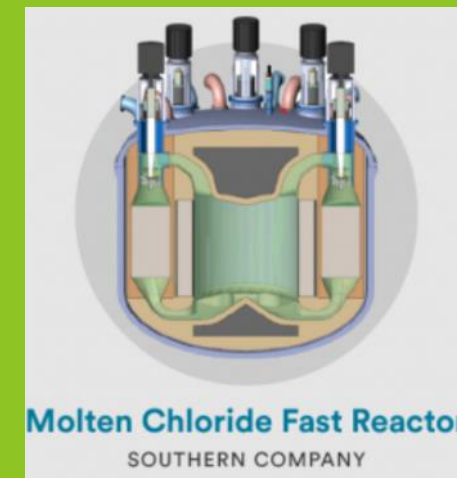
- Secretary of Energy Jennifer M. Granholm

DOE Office of Nuclear Energy



Advanced Reactor Development Program

Molten Salt Reactors

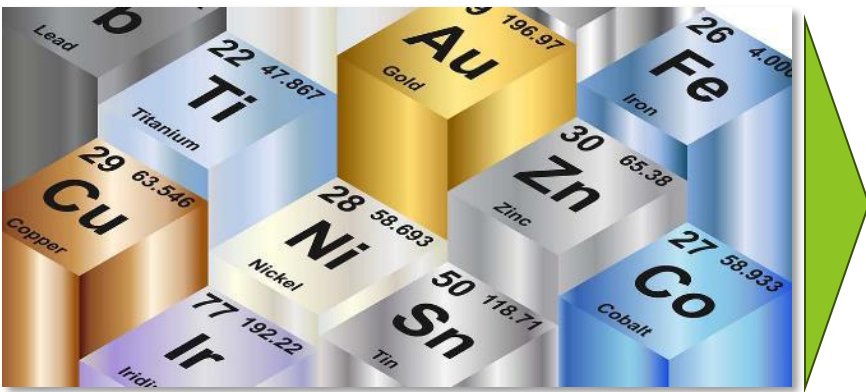


[Advanced Reactor Development | Department of Energy](#)

Current DOE mission objectives include commercialization of molten salt reactors for advancing U.S. nuclear energy infrastructure for increasing energy demand in the face of climate change.

The Materials Science Paradigm

Current Library of Nuclear Structural Materials



Structure-Property-Processing-Performance Relationships

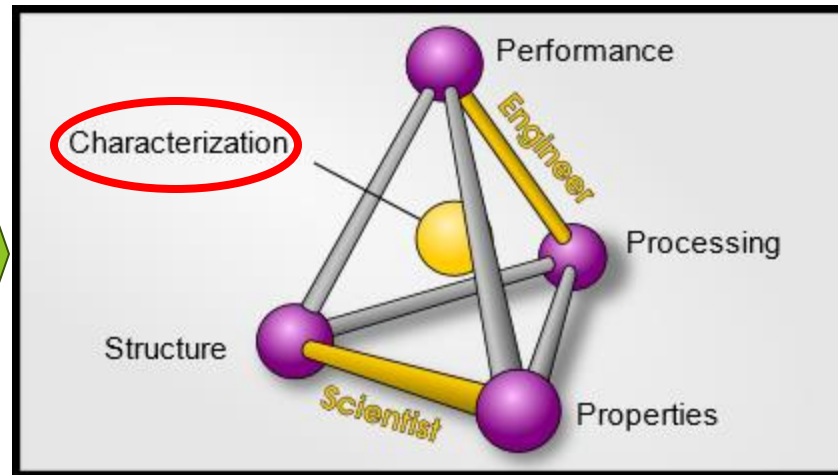


Image Courtesy : <https://mstudent.com/what-is-materials-science-tetrahedron-paradigm/>

Advancing nuclear structural materials

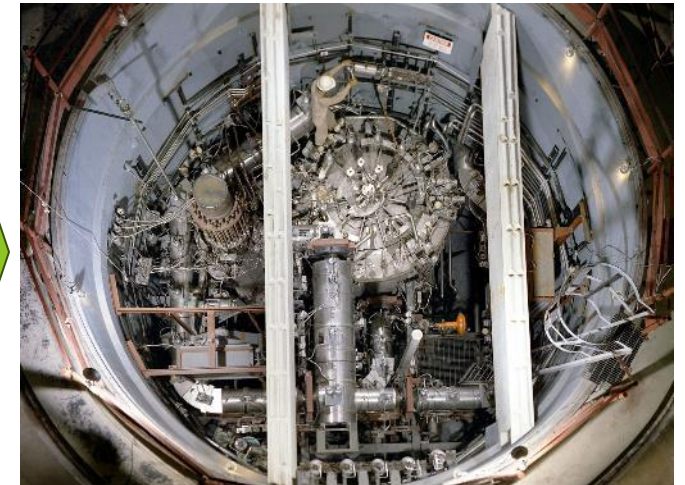
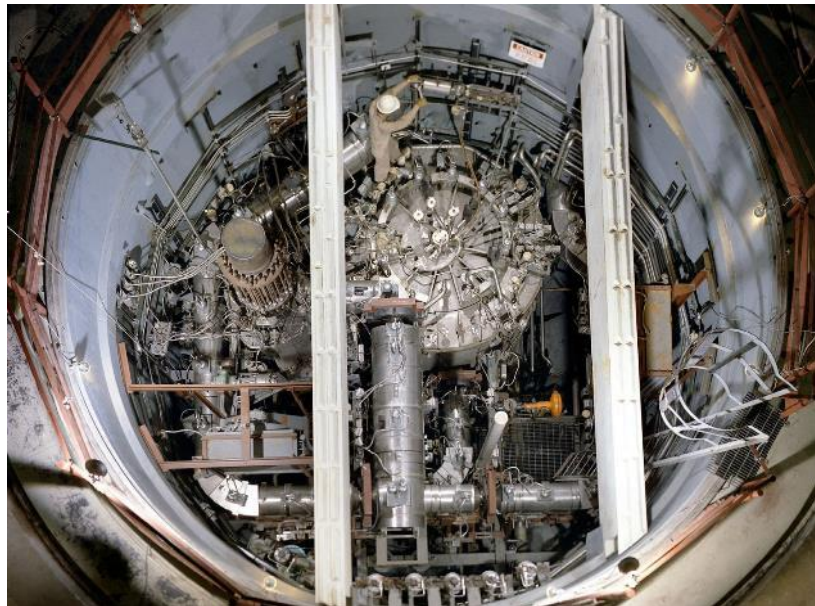


Image Courtesy : <https://www.iaea.org/newscenter/news/spotlight-on-innovation-molten-salt-reactors-for-a-sustainable-clean-energy-transition>

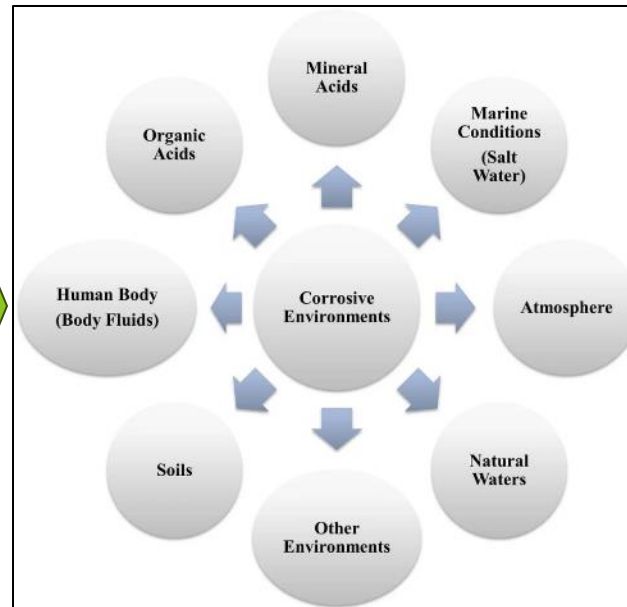
Characterization is the focal point of connecting structure-property-processing relationships in order to advance the current library of nuclear structural materials for advanced reactors.

Sources and Types of Corrosion

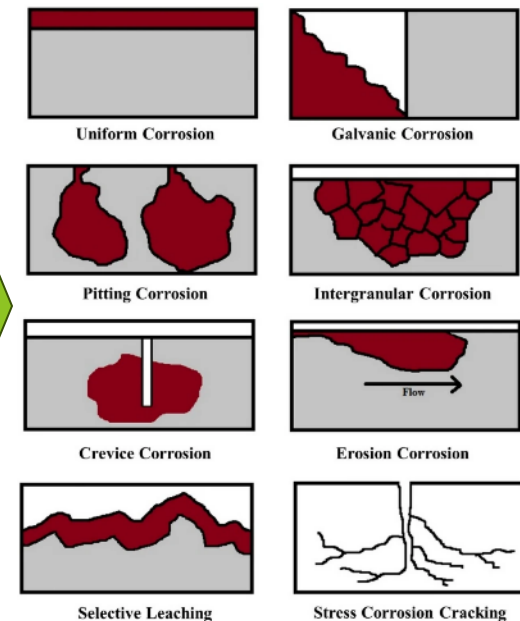
Novel Extreme Environments



Sources of Corrosion



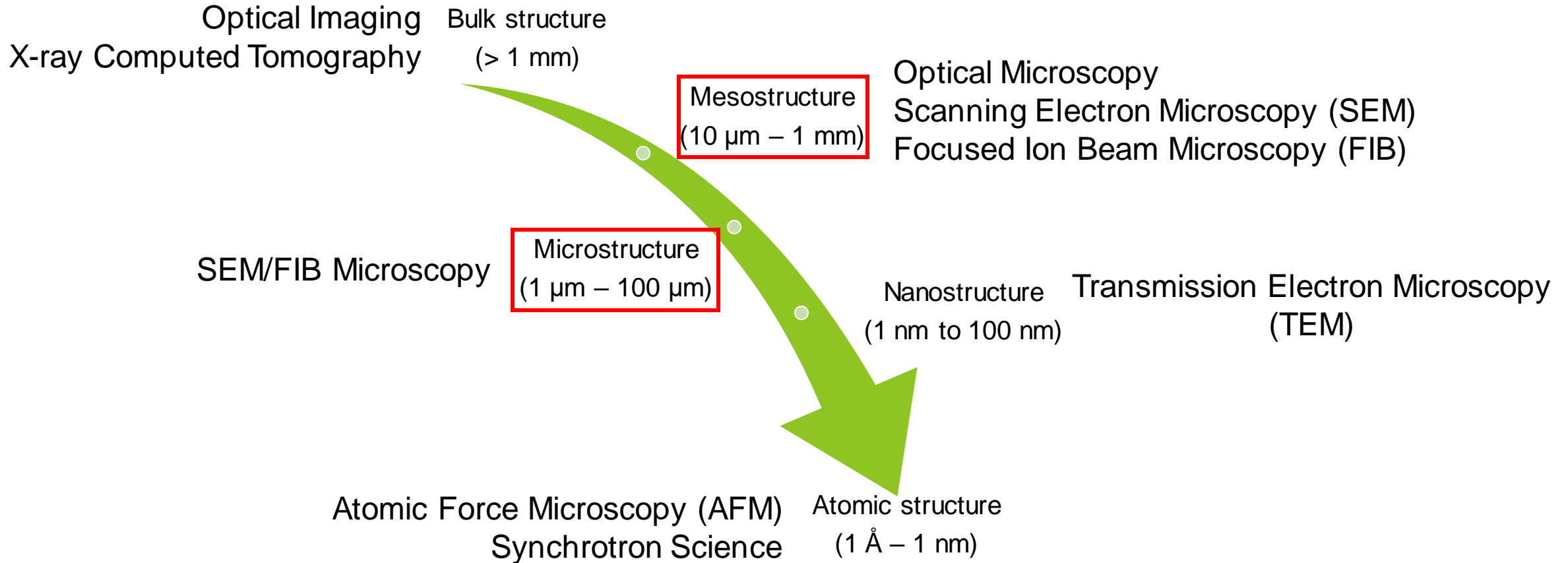
Corrosion mechanisms



Advanced reactor technologies introduce extreme environments that our existing library of structural materials need to be adapted to withstand. For instance, MSRs house highly corrosive media that challenge material performance.

Image Courtesy: <https://doi.org/10.1007/s40033-022-00367-5>; <https://www.metalltek.com/blog/how-to-evaluate-materials-properties-to-consider/>

A Library of Advanced Characterization Techniques



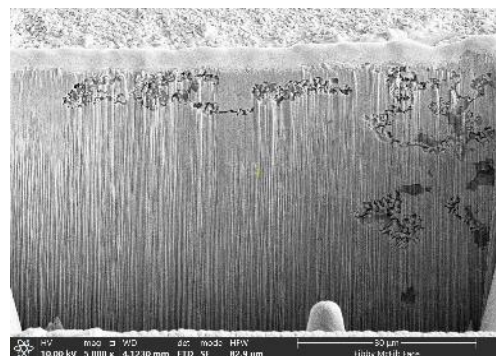
Different characterization techniques can be employed to study how the corrosion mechanism impacts material structure at different length scales.

About FIB microscopy and tomography

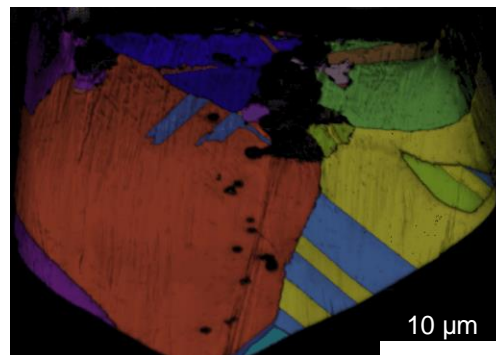
ThermoFisher G4 Helios Hydra Plasma FIB
(Irradiated Materials Characterization Laboratory, INL)



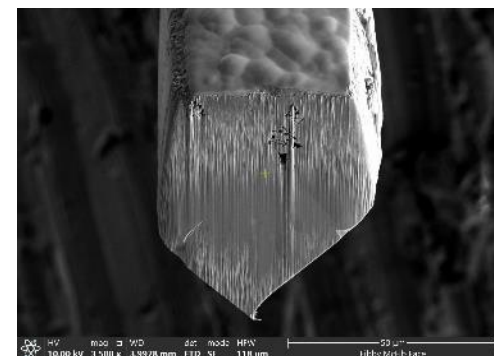
Trenched into Alloy 617 corroded in
 NaCl-MgCl_2 molten salt



2D slice-by-slice mapping of
microstructure



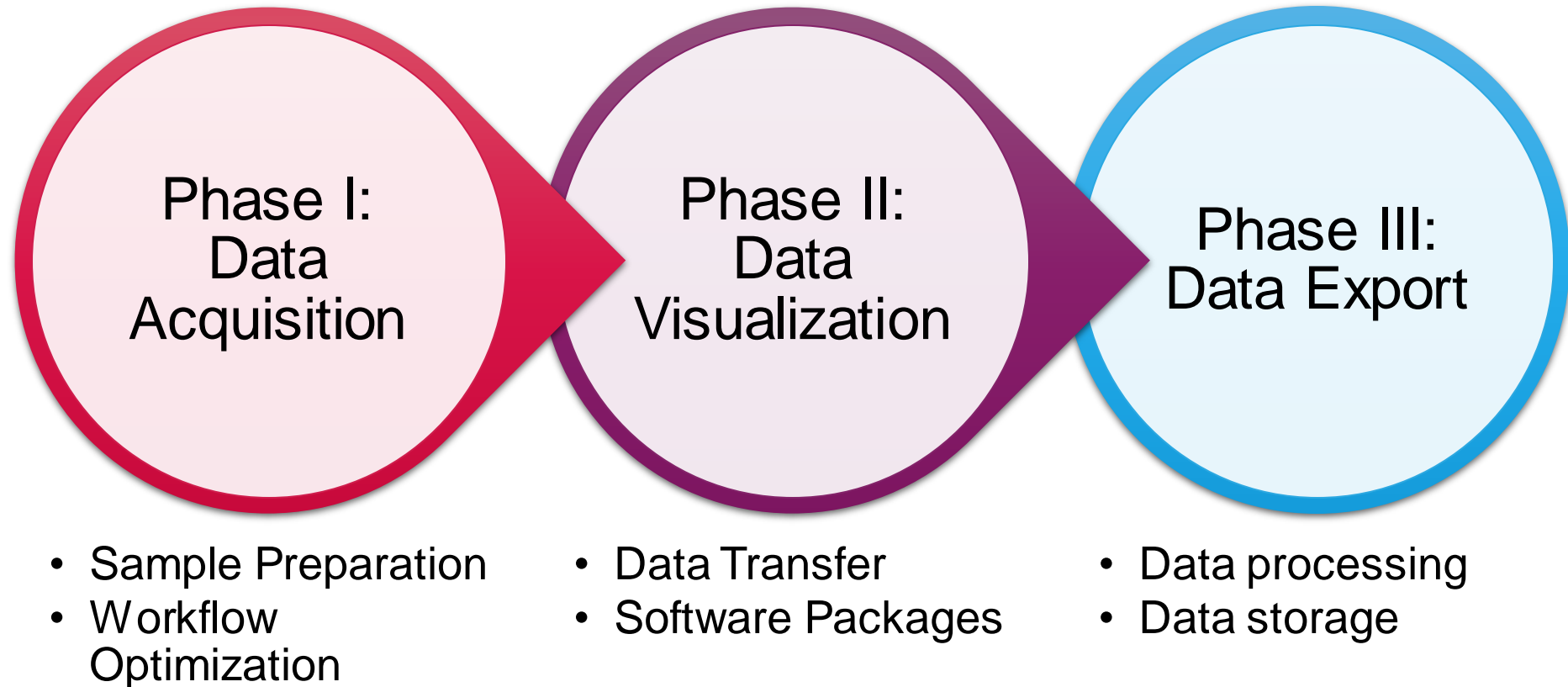
Lifted out ~50 by 50 μm cube
from Alloy 617



3D reconstruction of Alloy 617
microstructure

FIB tomography can enhance 2D meso/microstructure characterization to a comprehensive 3D characterization that can aid in validating computational modelling efforts to predict material performance.

FIB Tomography Workflow



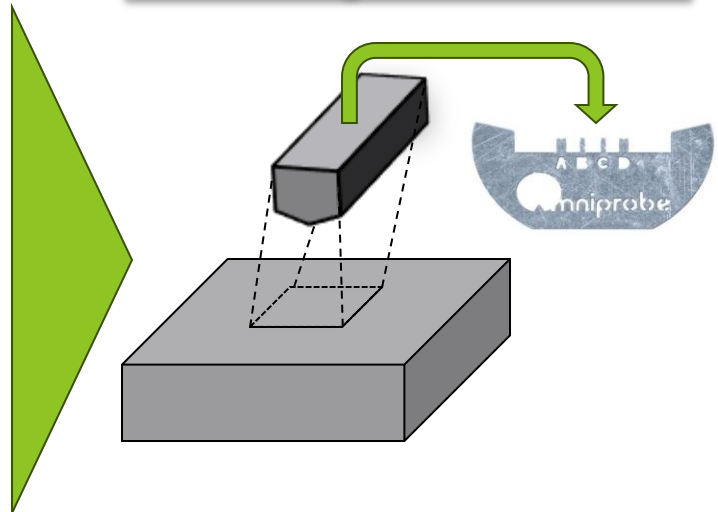
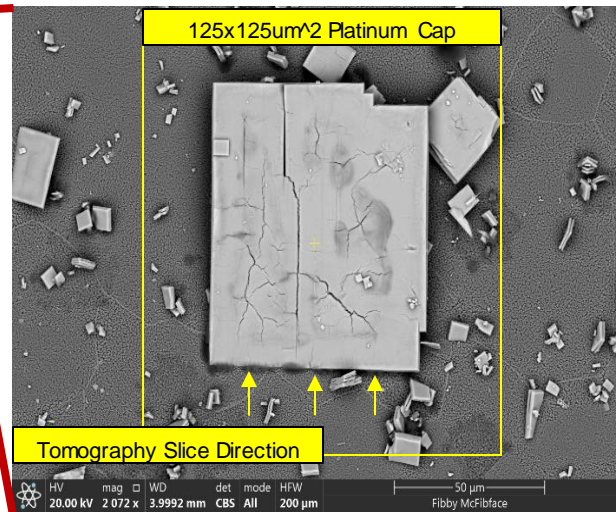
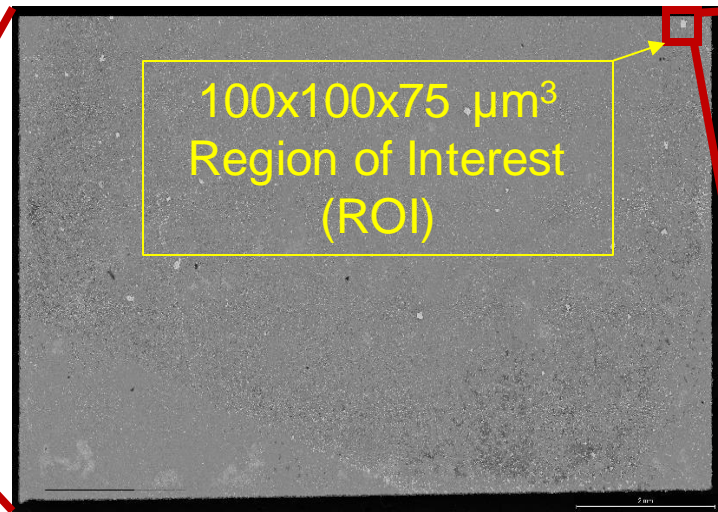
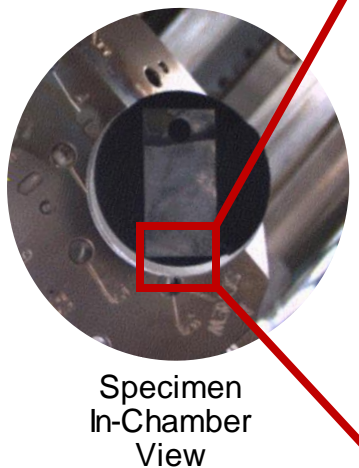
Preparing samples for FIB Tomography

Ni-Cr-Mo Alloy
Corroded in NaCl-
UCl₃ for 1000 h at
700 °C

Image Montage of Specimen

Selecting ROI for Lift-out

Lifting out Cube and
Mounting on TEM Grid

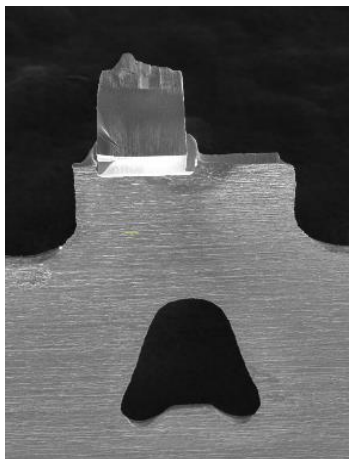


50,000 x 50,000 Pixel Wide Area

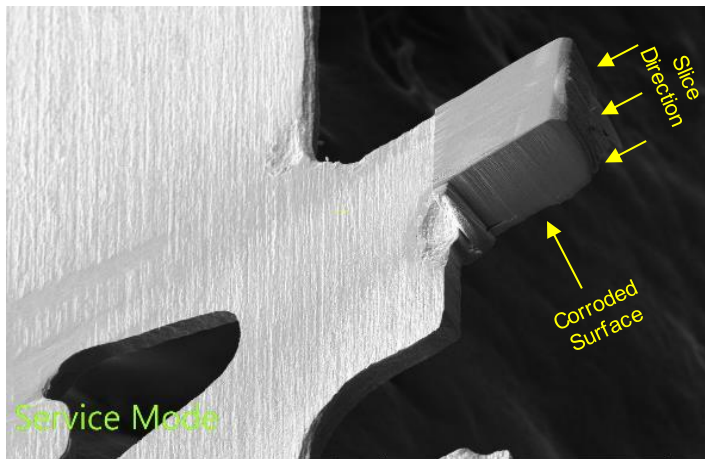
Sample preparation for FIB tomography requires correctly configuring the specimen for proper slice-by-slice milling.

Inverted Sample Preparation & Data Acquisition

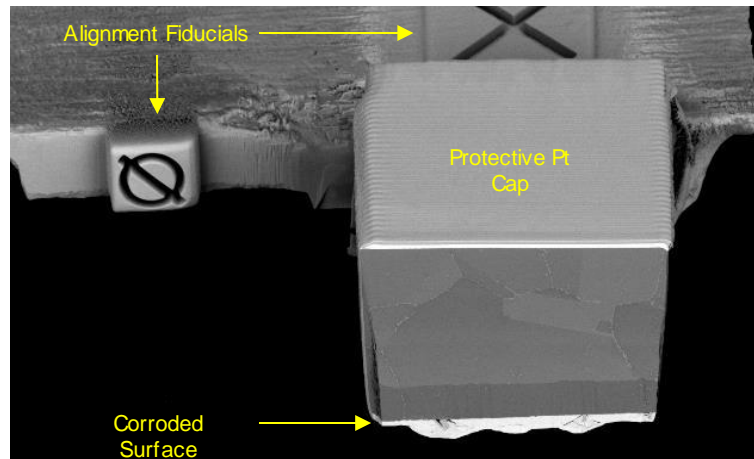
Mounted Specimen



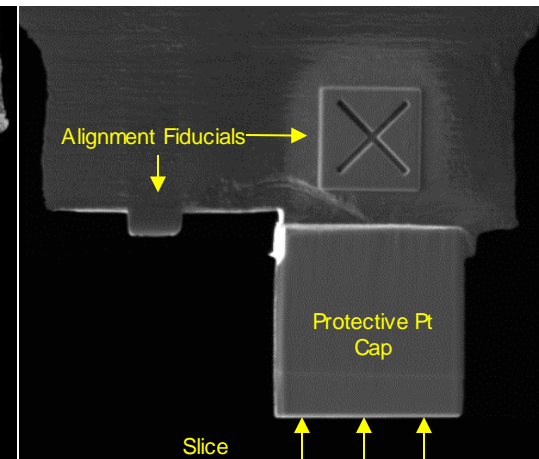
Sample Cleaning/Pt Cap Deposition



SEM (Acquisition) View



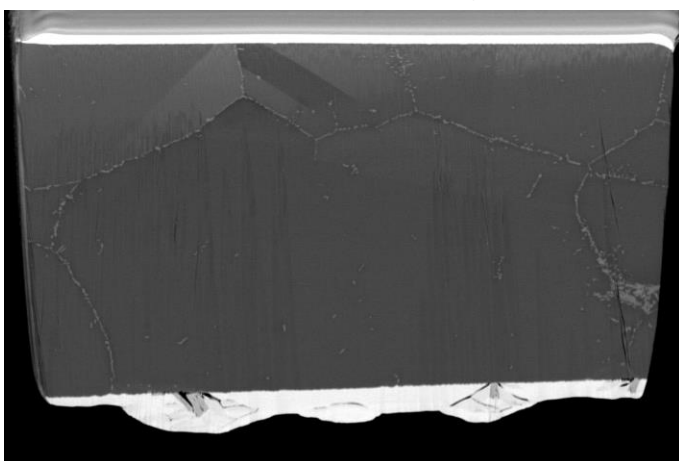
FIB (Milling) View



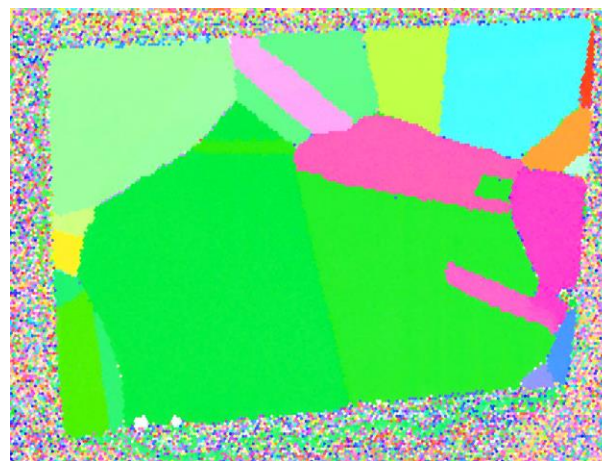
FIB Milling – SEM View



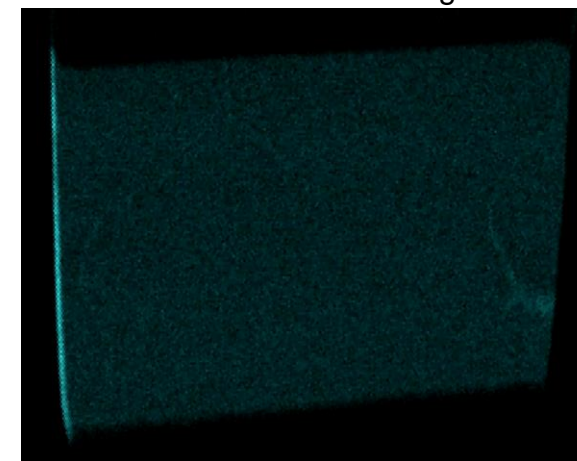
5kV - Backscattered Signal



20kV - Microstructure Signal

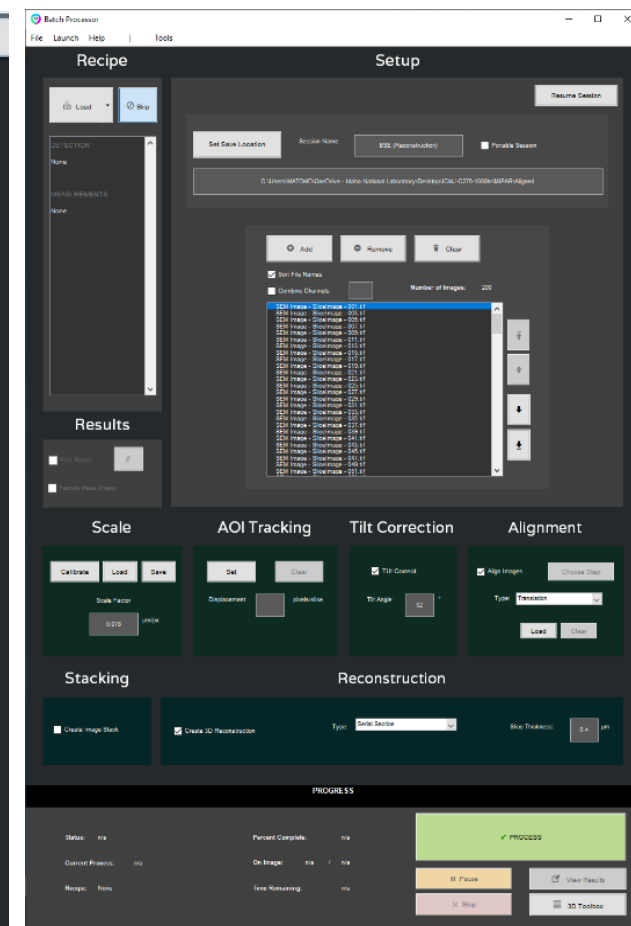
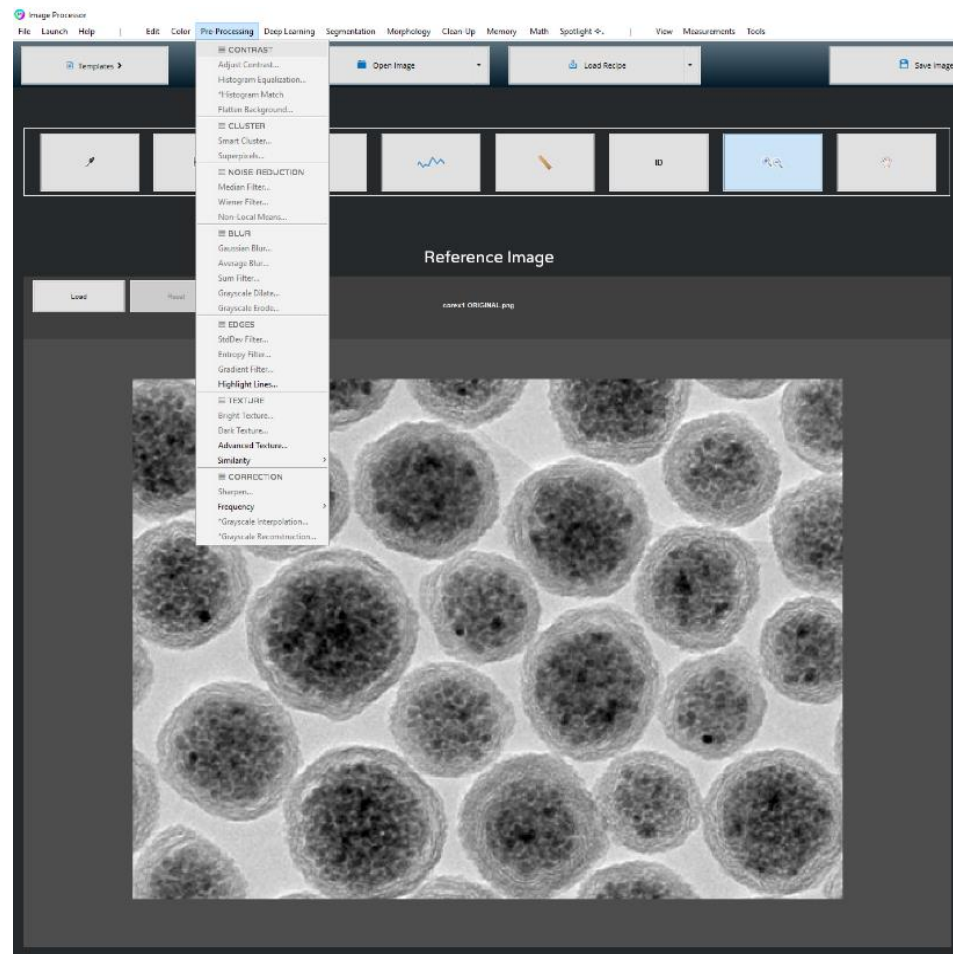


20kV - Mo Elemental Signal



Data Processing and Visualization: Pre-Processing

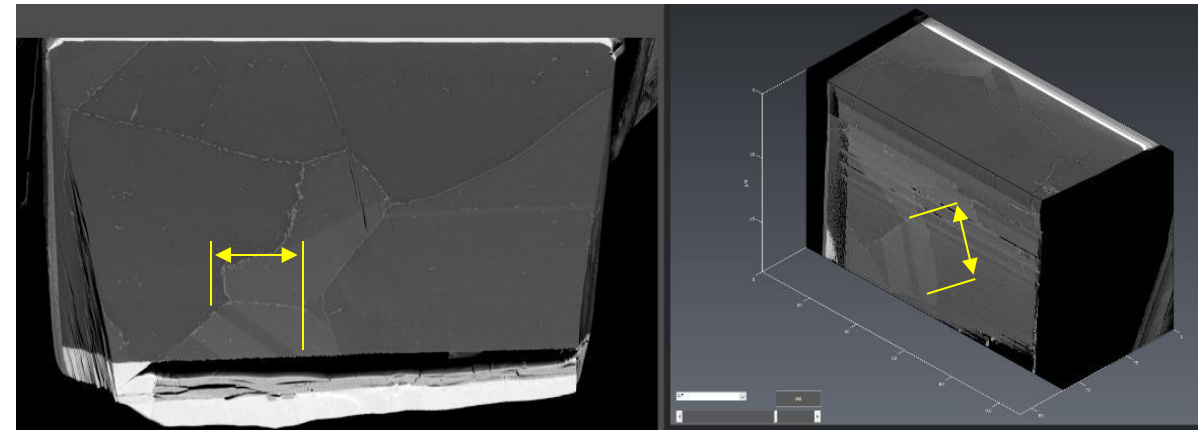
- Image Cropping
- Image Rescaling
- Tilt Correction
- Histogram Adjustment
- Noise Reduction
- Filtering
- Image Registration



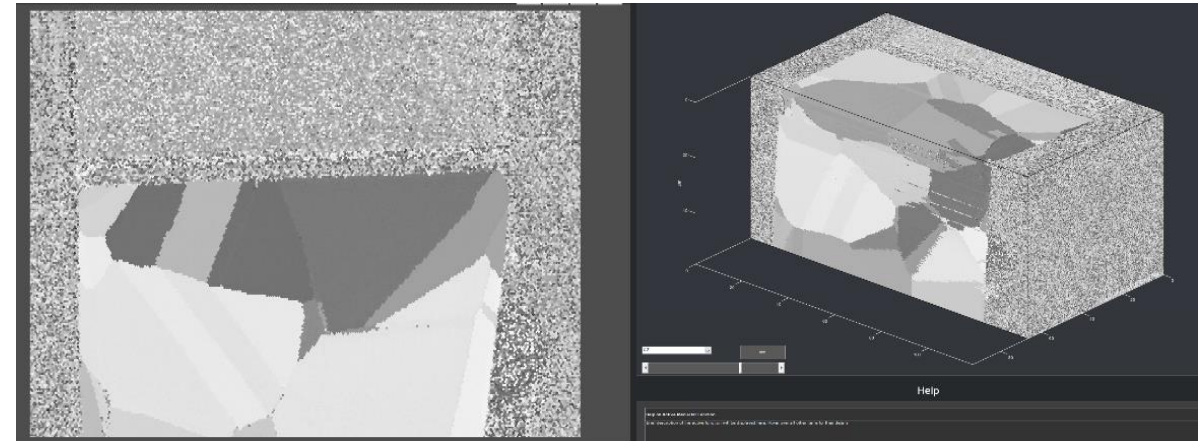
Data Processing and Visualization: 3D Reconstruction

- Segmentation Algorithms performed in 3D volumetric data instead of slice-by-slice which may yield more accurate results
- Regions of interest can be visualized in 3D, reducing ambiguities experienced with traditional 2D Cross-sectional imaging techniques

5kV - BSE Reconstruction



20kV - EBSD Reconstruction



Ensuring accessibility to FIB Tomography Data through open-source file formats

Raw Data

- Tiff Image Stacks**
 - Electron Images
 - EDS Maps
 - EBSD Maps
- EDAX – EDS/EBSD Project Files**
 - .HD5 Files
 - .Ang Files

Pre/Post Processing

- MIPAR
- DREAM3D
- Dragonfly
- Matlab
- Python
- AVIZO

Reconstruction Files

- MIPAR Formats**
 - .Rec Files (Image Data)
 - .STL Files (Surface Data)
- DREAM3D Formats**
 - .HD5 Files
 - .Ang Files

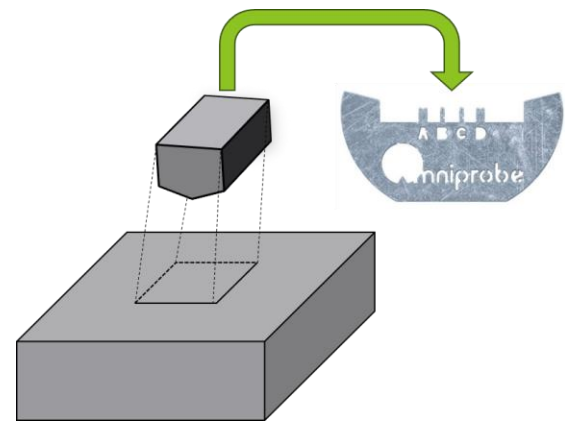
NRDS Nuclear Research Data System

Image Segmentation & Quantitative Analysis

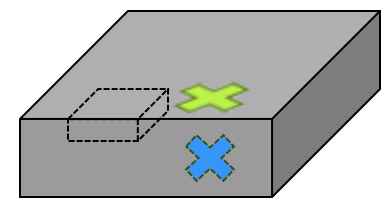


Optimizing FIB Tomography data collection and visualization

Specimen Configuration



VERSUS



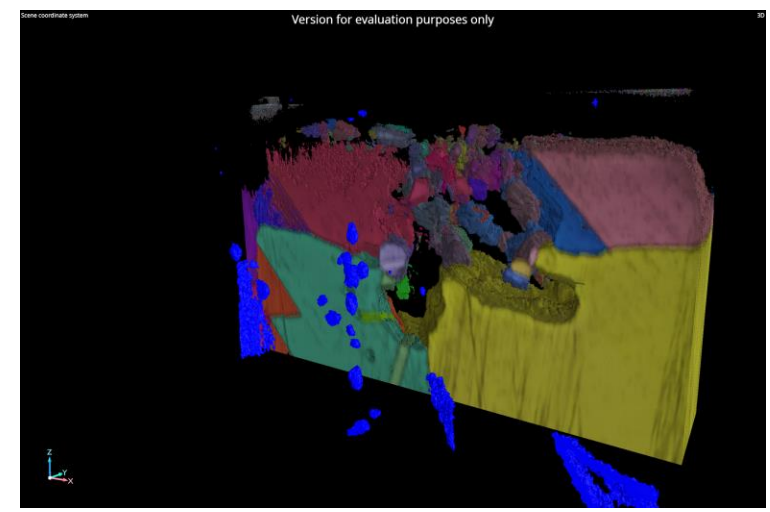
Data Processing and Visualization Software Packages



MIPAR
Image Analysis Software



Incorporating Elemental Analysis Data into Microstructural Data



Summary

- FIB tomography can enhance 2D meso/microstructure characterization to a comprehensive 3D characterization that can aid in validating computational modelling efforts to predict material performance
- Data collection can be performed on corroded/contaminated/irradiated structural material and nuclear fuels
- Data processing and visualization can be performed with different software packages that require training to use
- FIB tomography data has been uploaded to the NRDS
- Current efforts are on optimizing data acquisition, processing, and visualization

Acknowledgements

- **Office of Nuclear Energy**
 - **Nuclear Science User Facilities**
 - **Instrument Scientist Program (Thank you, Jeff!)**
- **Characterization and Post-Irradiation Examination (CAPIE) Division**
 - Advanced Ion Characterization and Micro-mechanics Group
 - Materials Informatics & Transmission Electron Microscopy
 - Nuclear Structural Materials Group



Questions?