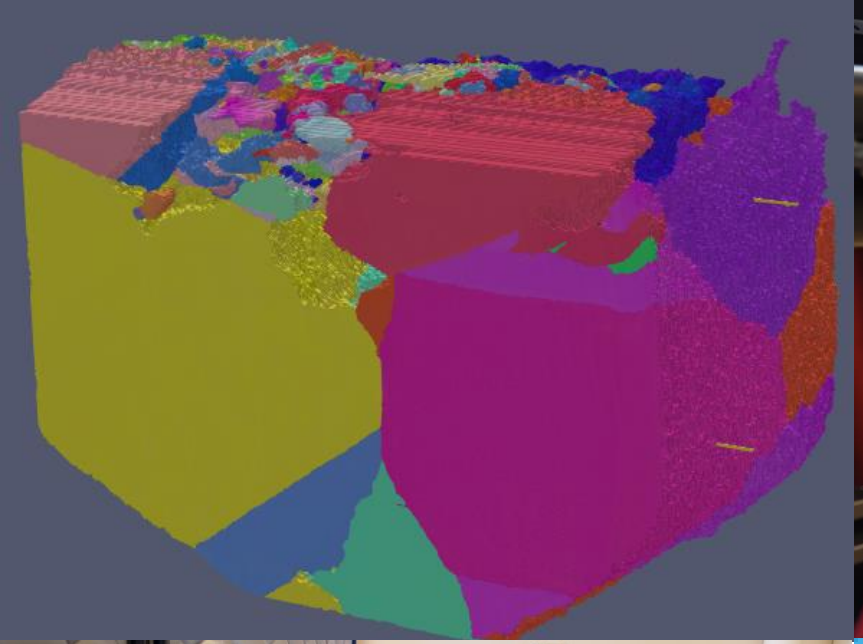
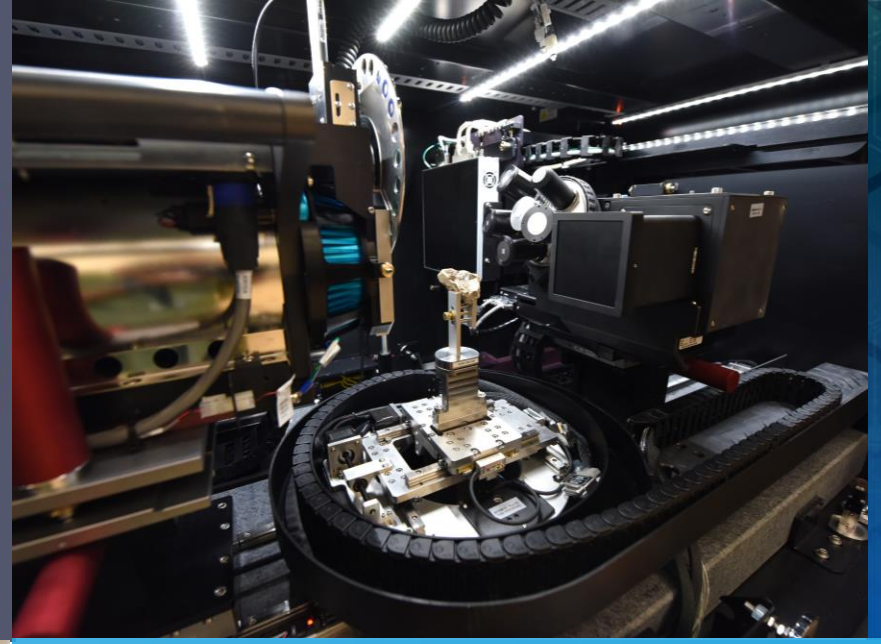




Trishelle Copeland-Johnson
Materials Research Scientist

Characterization &
Advanced Post-Irradiation
Examination

Multi-Modal Characterization of Nuclear Fuels and Materials at the Idaho National Laboratory Materials & Fuels Complex

Battelle Energy Alliance manages INL for the
U.S. Department of Energy's Office of Nuclear Energy

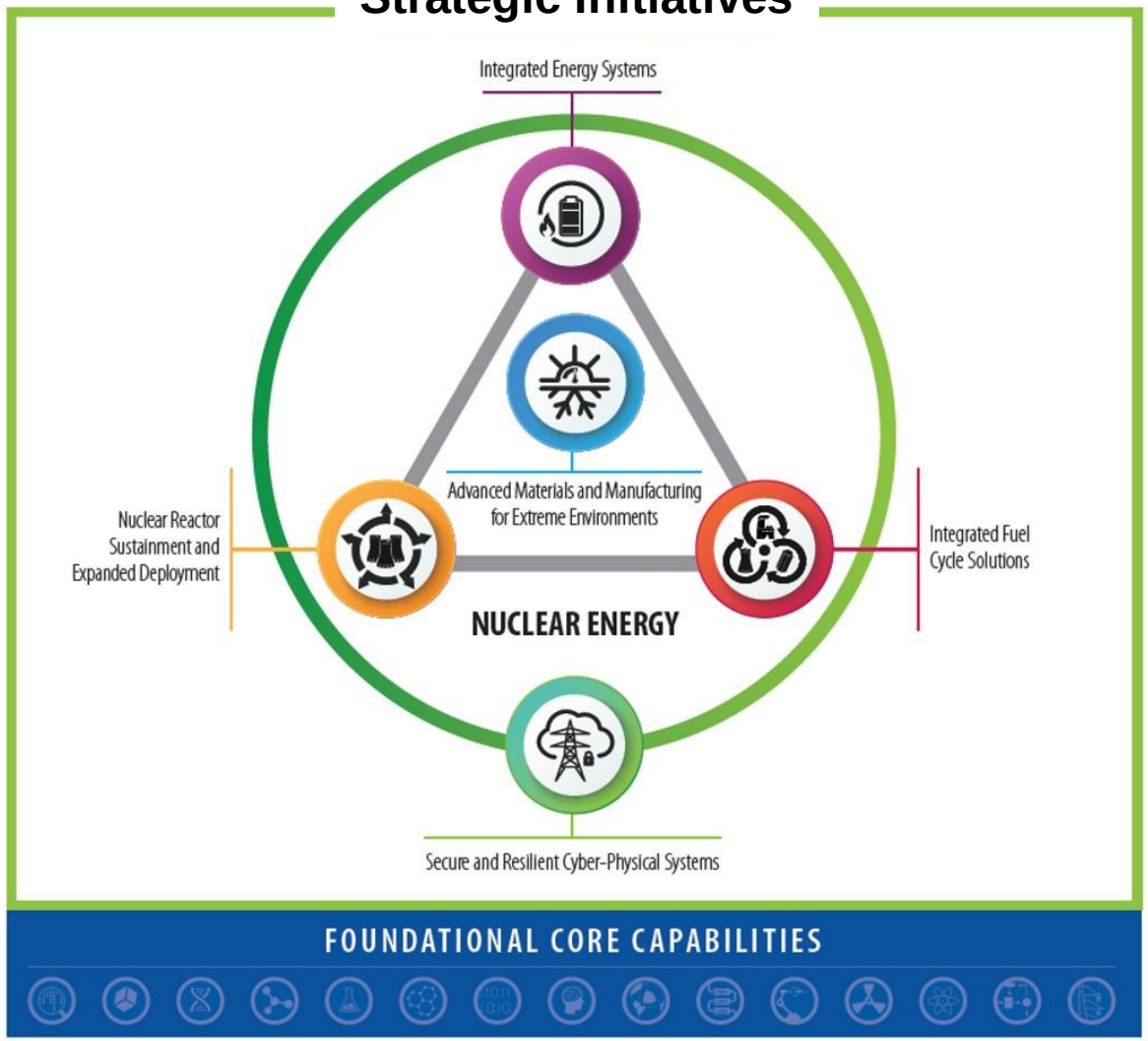
INL Idaho National Laboratory

INL Quick Facts

- One of the 17 Department of Energy (DOE) National Laboratories
- **Mission Outcomes**
  - **Develop, innovate, secure and demonstrate nuclear energy technologies**
  - **Provide national security solutions and protect critical infrastructure**
  - **Provide at-scale clean energy systems and complementary environmental solutions**
- **560,000** acres
- **6,000** employees
- **5** Strategic Initiatives
- **15** Core Capabilities

INL Quick Facts

Strategic Initiatives



Foundational Core Capabilities	
Advanced Computer Science, Visualization, and Data	Environmental Sub-surface Science
Applied Materials Science & Engineering	Advanced Instrumentation
Biological and Bioprocess Engineering	Mechanical Design and Engineering
Chemical and Molecular Science	Nuclear and Radio Chemistry
Chemical Engineering	Nuclear Engineering
Condensed Matter Physics and Materials Science	Power Systems and Electrical Engineering
Cyber and Information Sciences	Systems Engineering and Integration
Decision Science and Analysis	

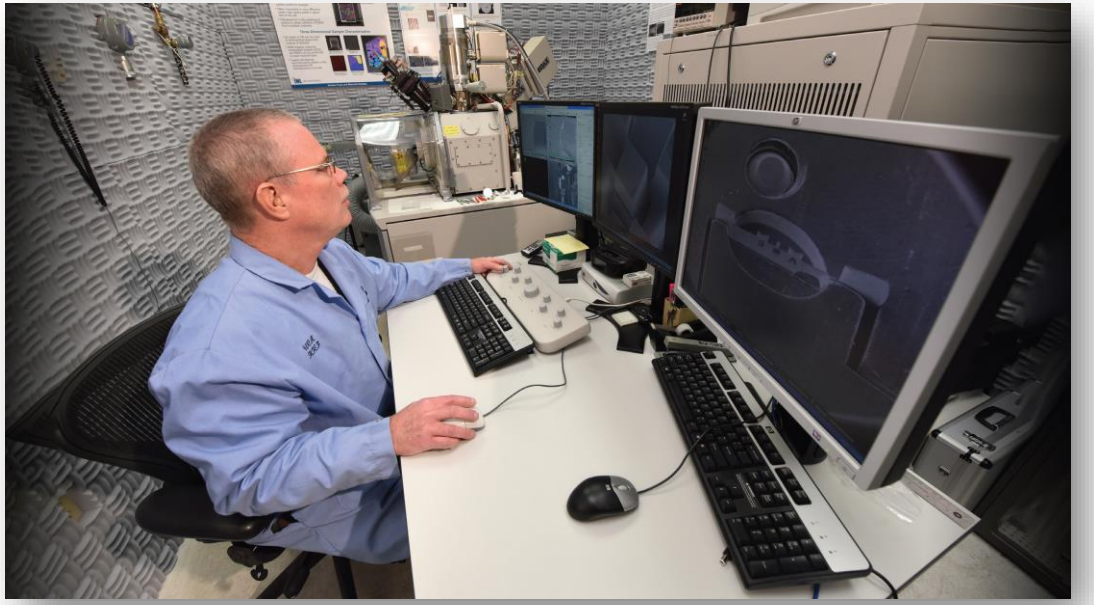
Welcome to the MFC!

IMCL

- 12,000 sq. ft. facility
- Characterization of nuclear fuels and materials
  - Structural
  - Chemical
  - Mechanical
  - Thermophysical
- Shielded instrument cells are reconfigurable to meet changing needs in nuclear research.
- Designed to enhance post irradiation examination throughput
- Capabilities available through the Nuclear Science User Facilities (NSUF)

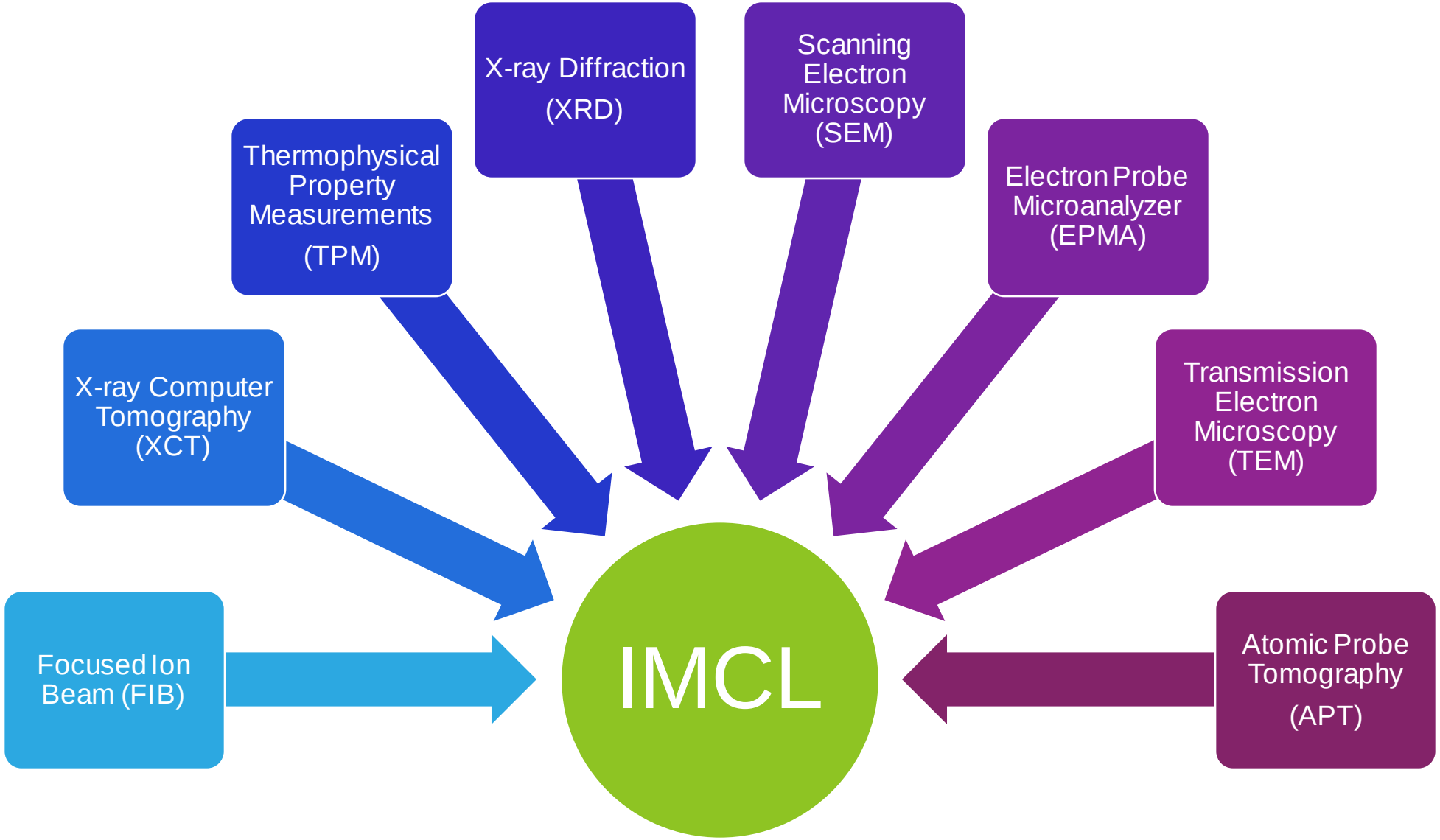
EML

- Materials Characterization with electron microscopy techniques
  - Optical microscopy
  - Scanning electron microscopy
  - Focused ion beam
  - Transmission electron microscopy
- Capable of handling both unclassified and classified radioactive materials

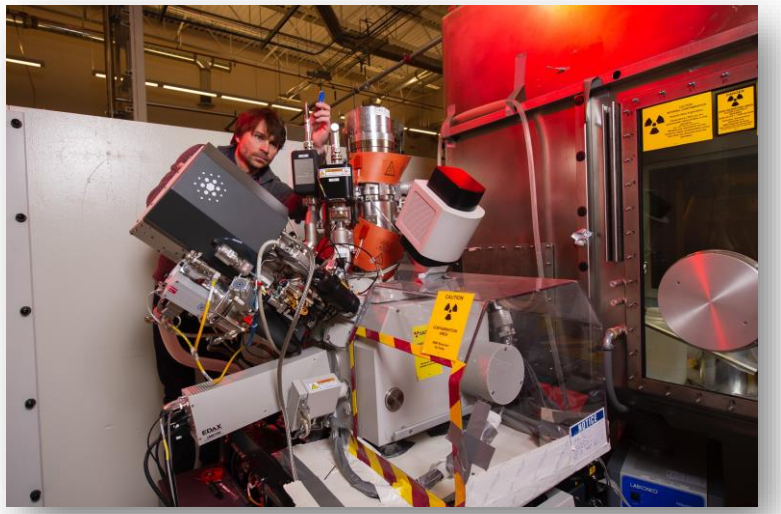


SPL

- Anticipated to be the most modern testing facility for analysis of nuclear structural materials
- Life-extension of existing and development of new reactor technologies
- Mechanical testing and failure analysis


X-ray Diffraction (XRD)
Scanning Electron Microscopy (SEM)
Thermophysical Property Measurements (TPM)
Electron Probe Microanalyzer (EPMA)
X-ray Computer Tomography (XCT)
Transmission Electron Microscopy (TEM)
Focused Ion Beam (FIB)
IMCL
Atomic Probe Tomography (APT)

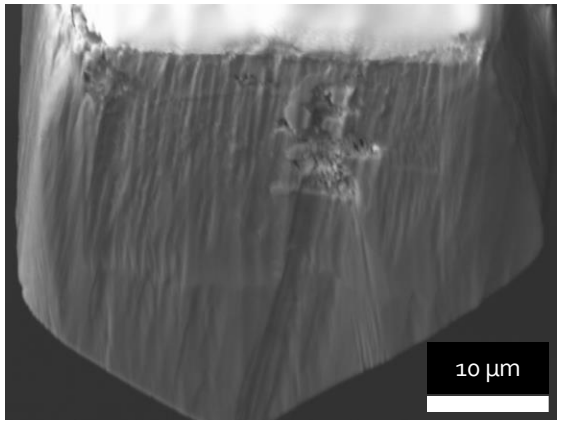
ThermoFisher G3 Plasma FIB



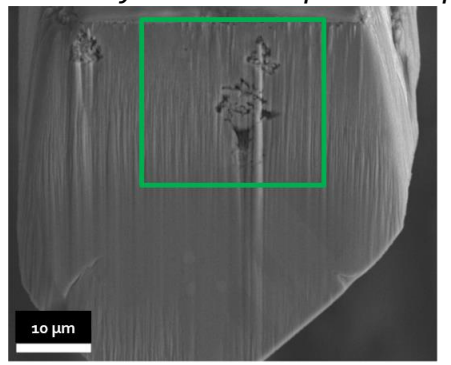
ThermoFisher G4 Helios Hydra Plasma FIB

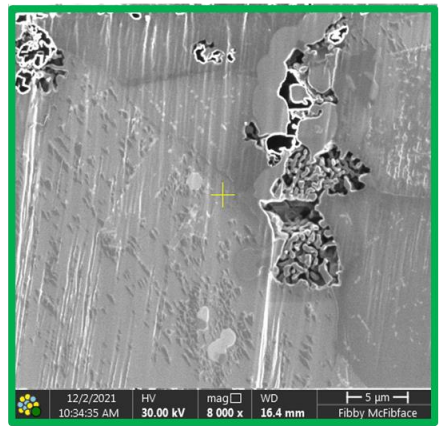
Characterization of Inconel 617 corroded in chloride molten salt

SEM/FIB Slice-by-slice reconstruction

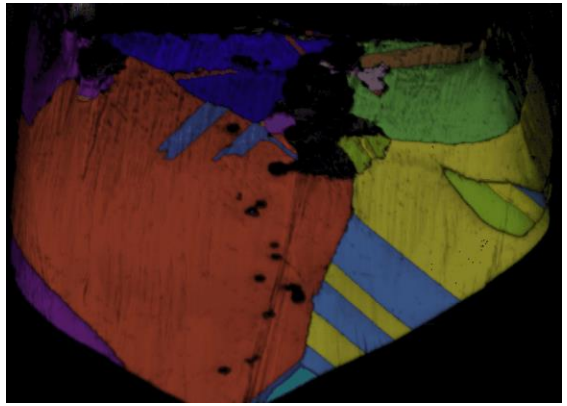


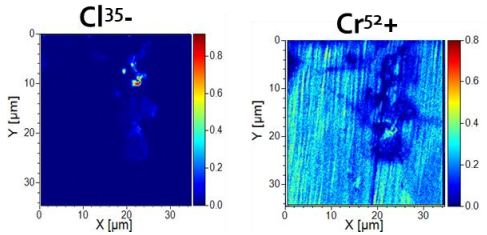
Secondary Ion Mass Spectroscopy





Electron backscatter diffraction



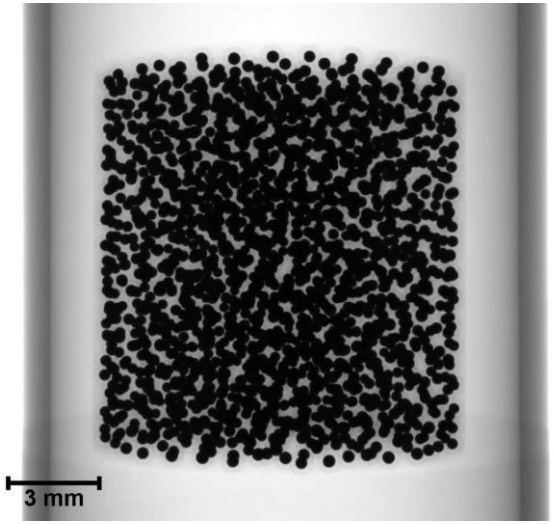


Data Courtesy of T.M Copeland-Johnson, D. Murray, G. Cao, 2022, Frontiers in Nuclear Engineering (Under Review)

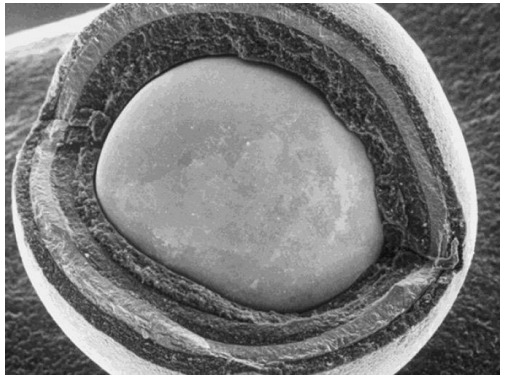
IMCL

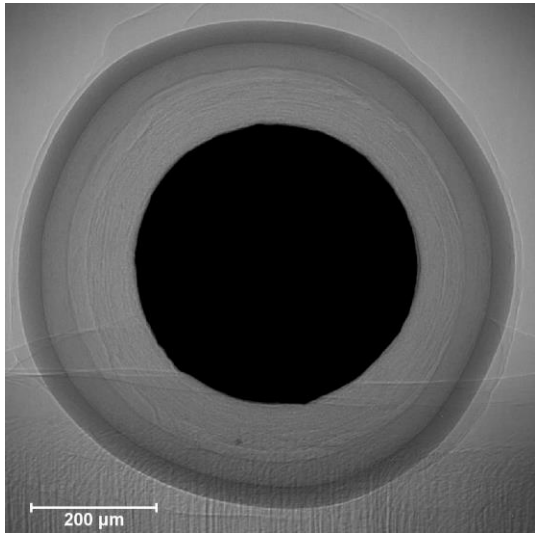
ZEISS Xradia 520 Versa XCT

TRISO Fuel Compact



TRISO Fuel Particle

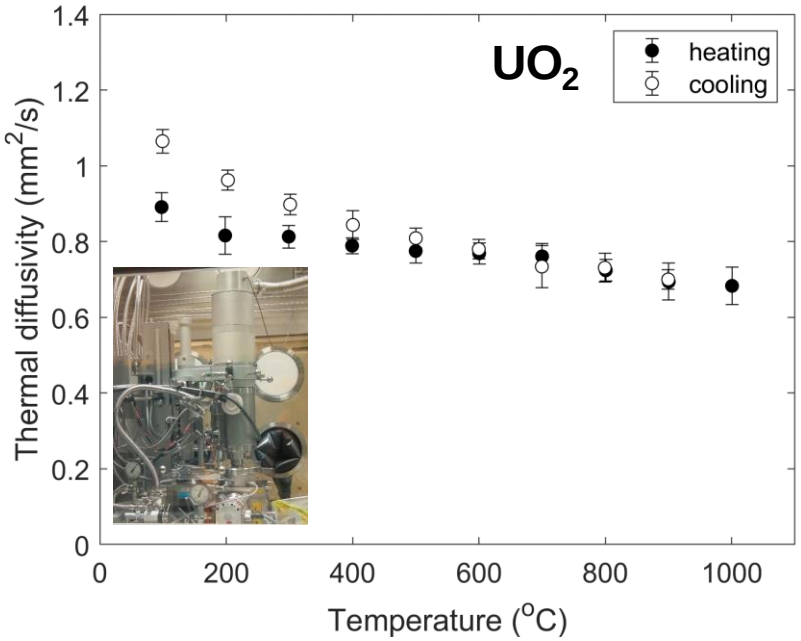




IMCL

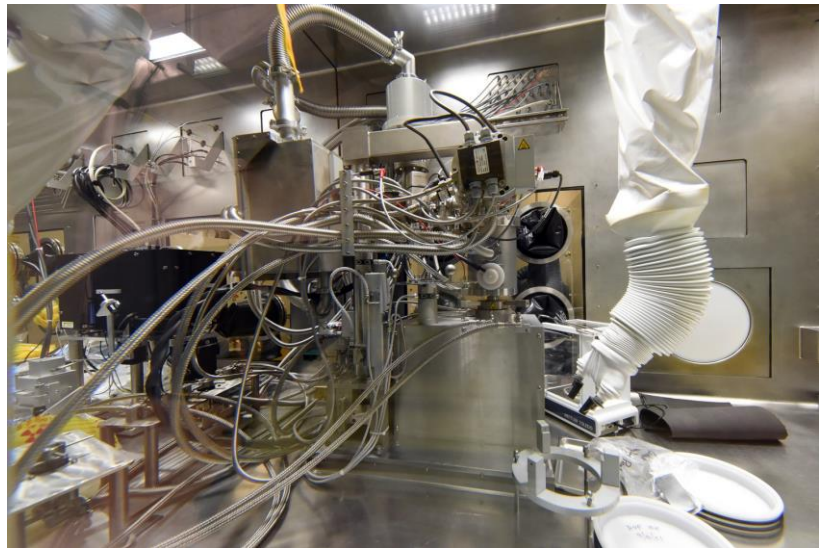
Laser Flash Analysis (<2000°C)

Thermal diffusivity, thermal conductivity



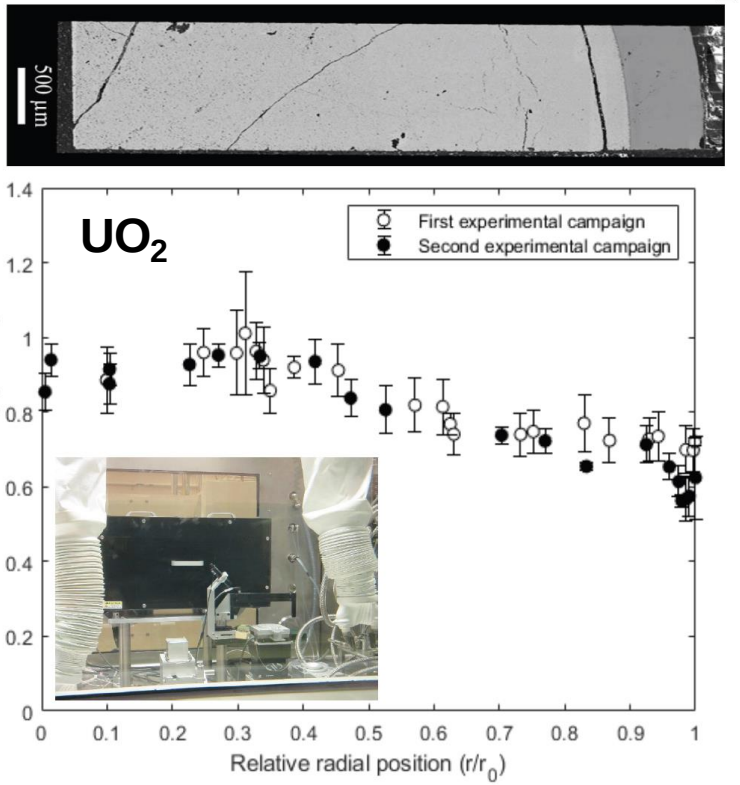
Simultaneous Thermal Analyzer/ Mass Spectrometer (<2000°C)

Post-irradiated materials for phase temperatures and enthalpy; specific heat; vapor pressure



Thermal conductivity microscope

Thermal diffusivity, thermal conductivity at the meso-scale (resolution ≈ 50 µm)



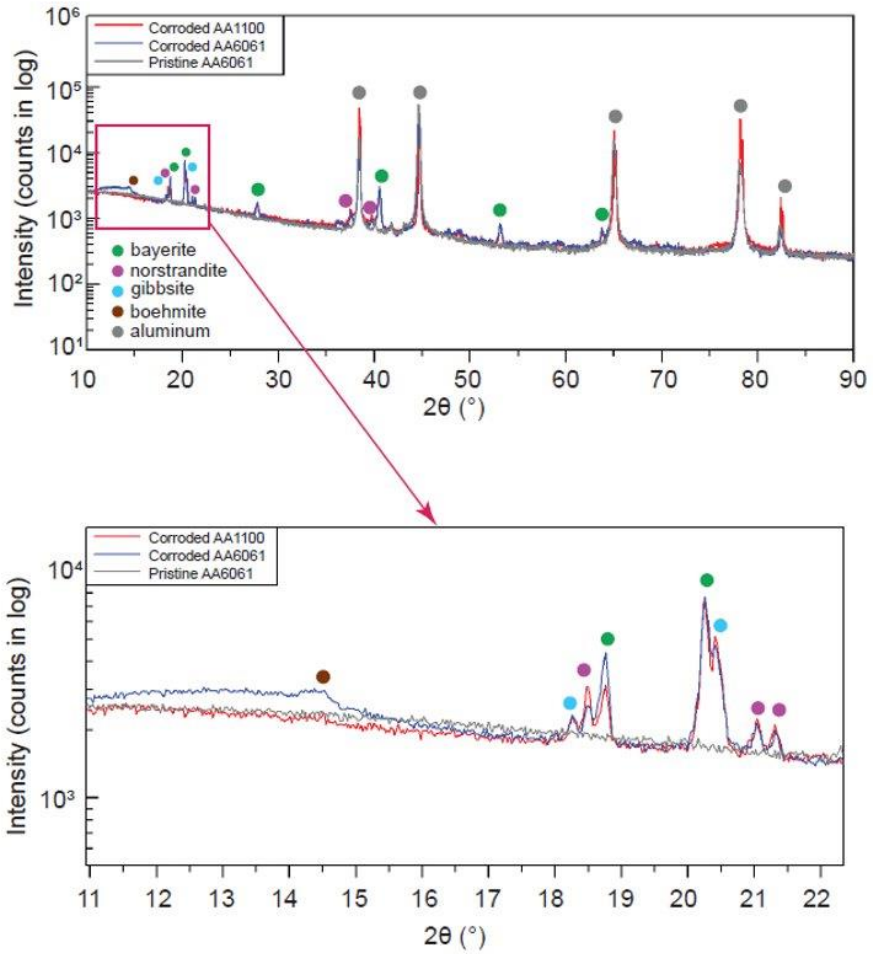
IMCL

Data courtesy of the United States Department of Energy Advanced Fuels Campaign

PANalytical powder X-ray diffractometer (right)

Bruker D8 Discover X-ray diffractometer (Left)

H_2 overproduction from radiolysis of corroded aluminum spent nuclear fuel alloys

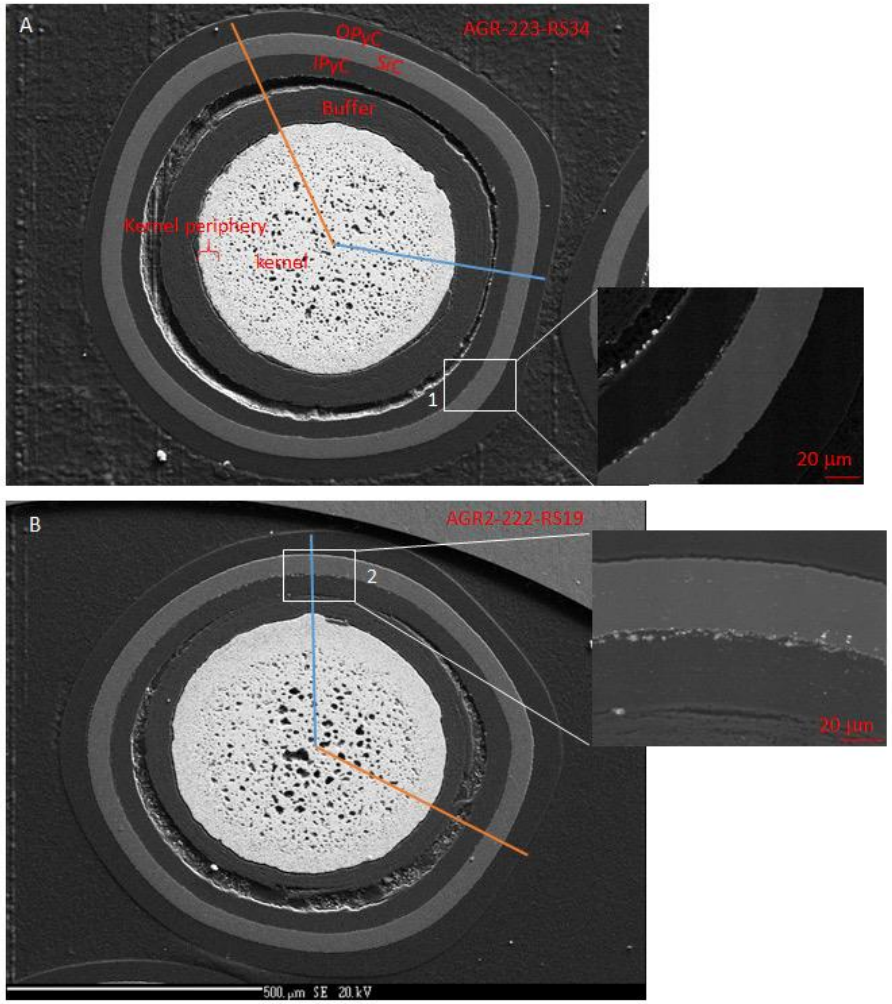


IMCL

Data courtesy of J. Conrad, X. Pu, A. Khanolkar, T. Copeland-Johnson, C. Pilgrim, J. Wilbanks, E. Parker-Quaife, G. Horne, 2022, MDPI Materials

JEOL 7600F High-Resolution SEM

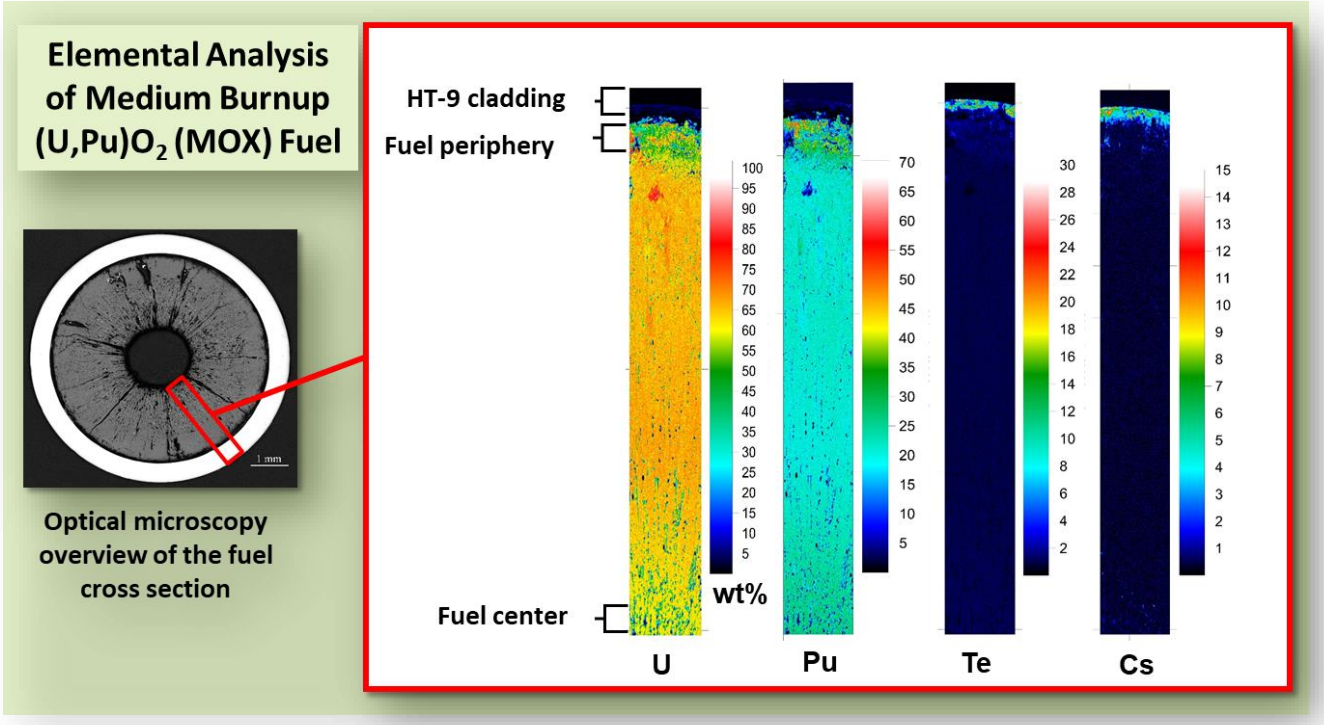
Imaging of a TRISO Particle



IMCL

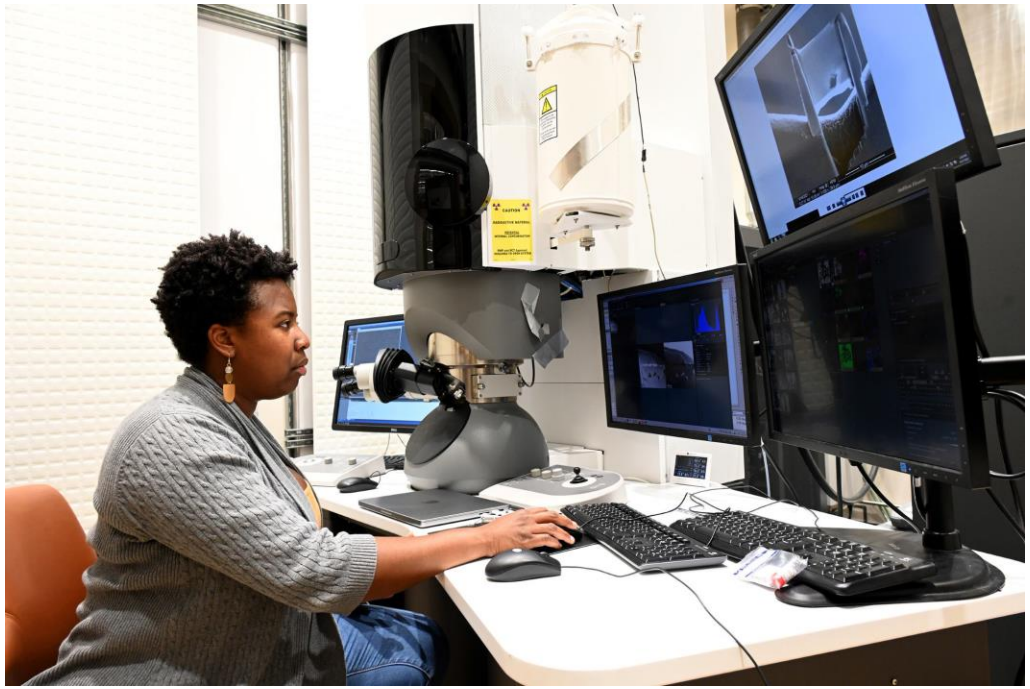
Data courtesy of the U.S. Department of Energy, Office of Nuclear Science under U.S. Department of Energy Idaho Operations Office Contract DE-AC07-05ID14517

Shielded Cameca SX100R EPMA



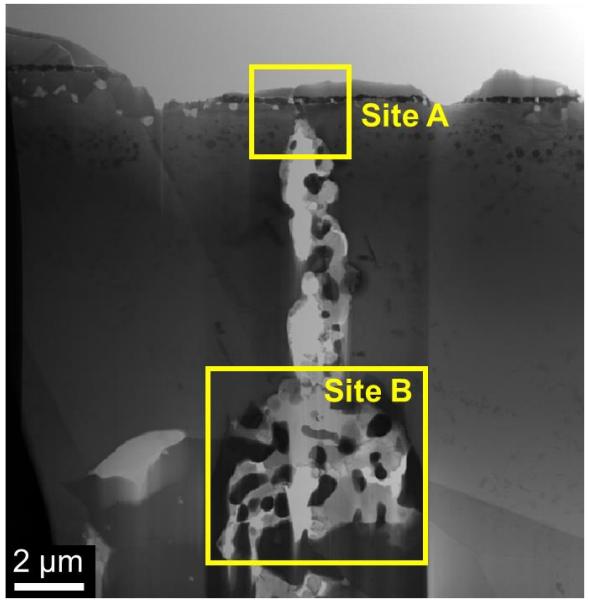
Data courtesy of the NSUF Consolidated Innovative Nuclear Research Program (Proposal #17-12976)

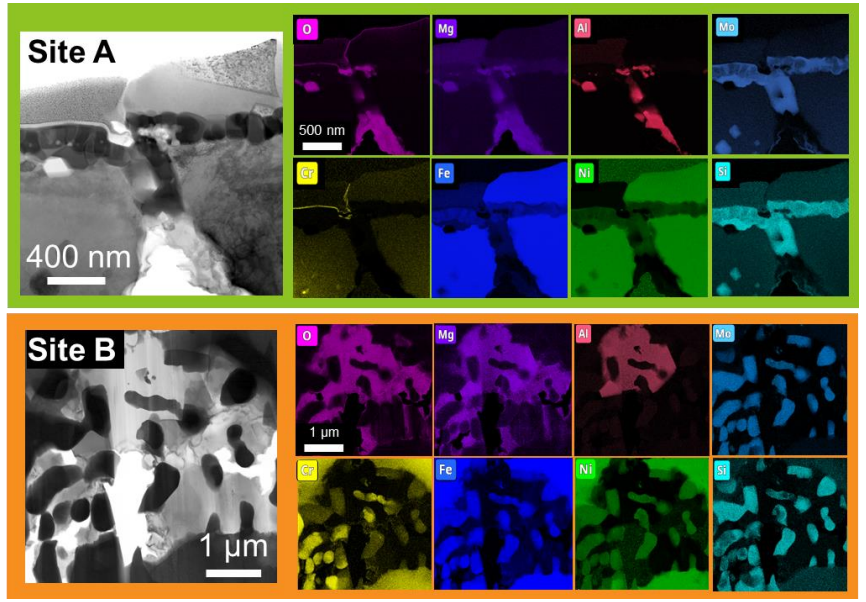
ThermoFisher - FEI Titan Themis 200 TEM



Characterization of Inconel 617 corroded in chloride molten salt

Energy Dispersive X-ray Spectroscopy





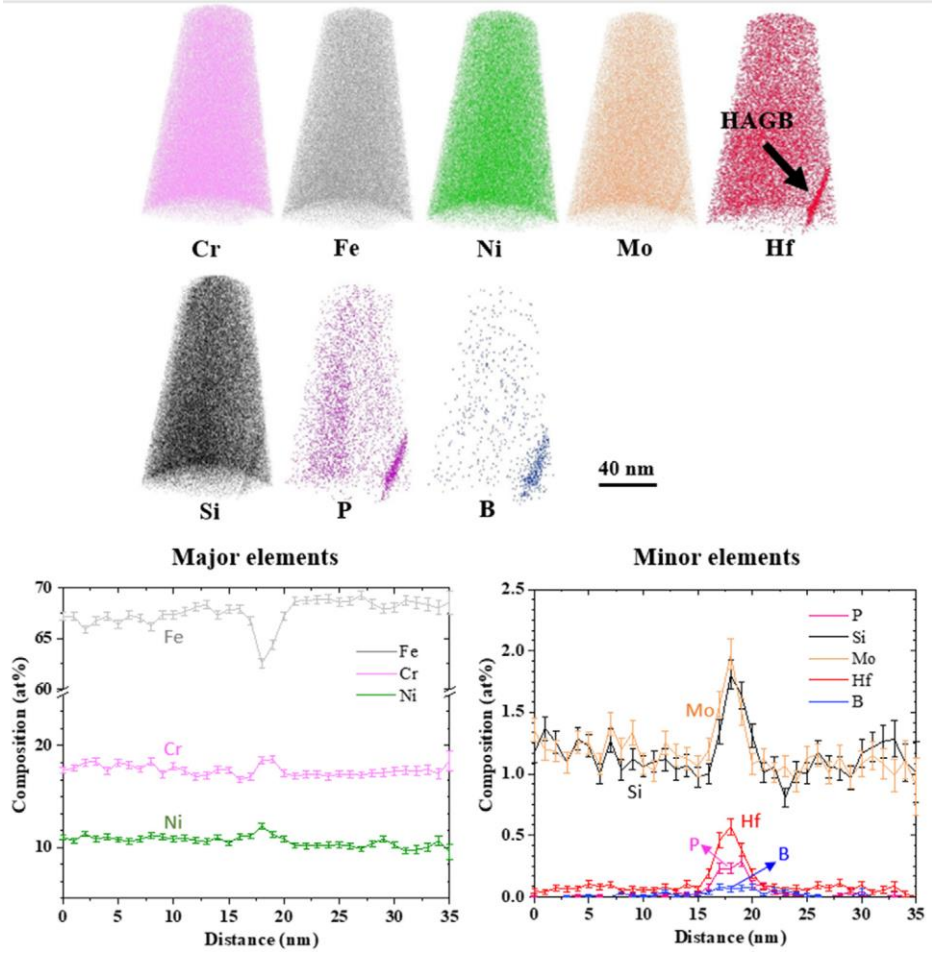
Data courtesy of T.M Copeland-Johnson, D. Murray, G. Cao, 2022, Frontiers in Nuclear Engineering (Under Review)

IMCL

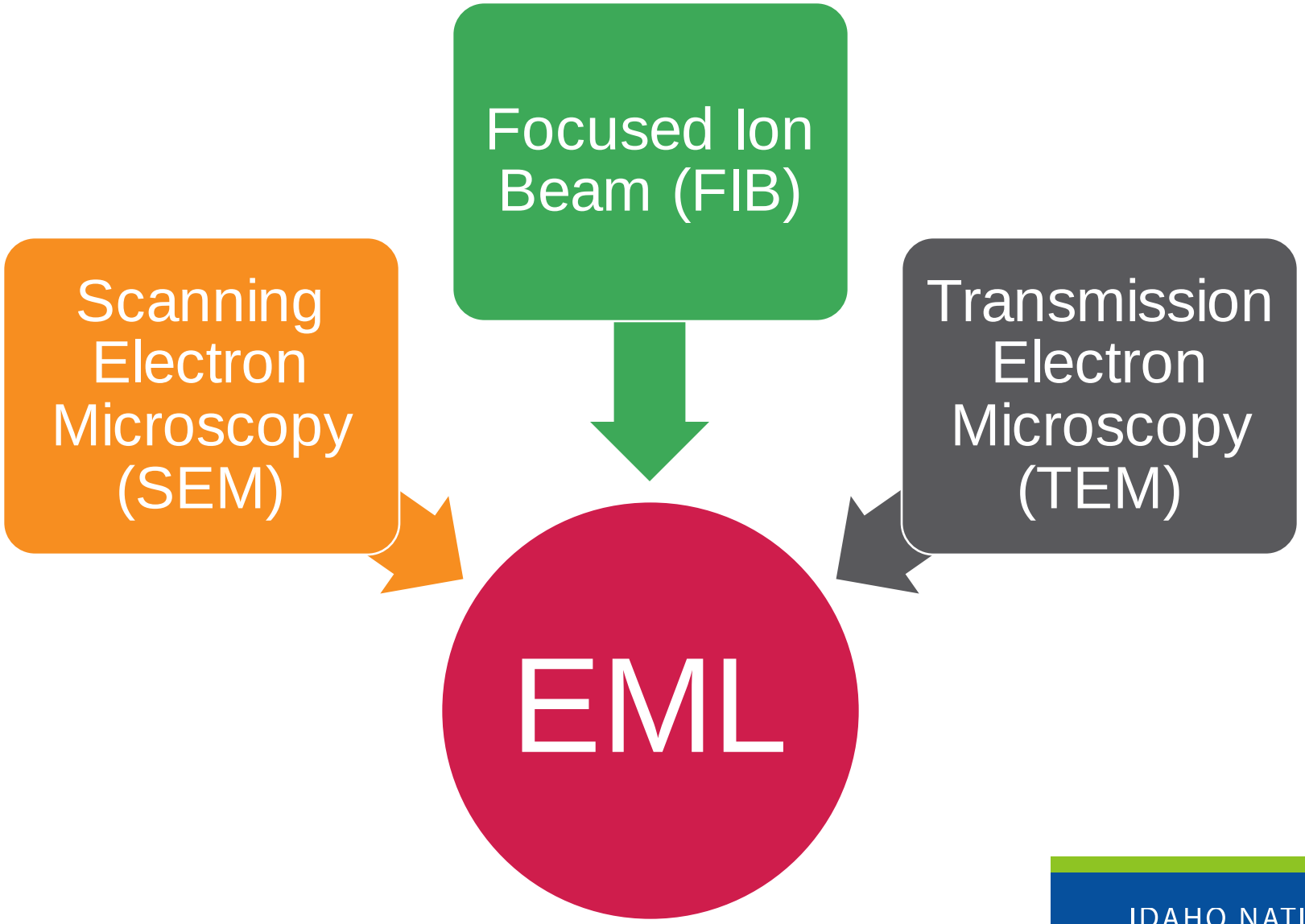
CAMECA LEAP 5000 APT

Irradiation-Assisted Stress Corrosion Cracking in Hafnium-doped Stainless Steel 316

Measuring Elemental Composition Across a High Angle Grain Boundary (HAGB)

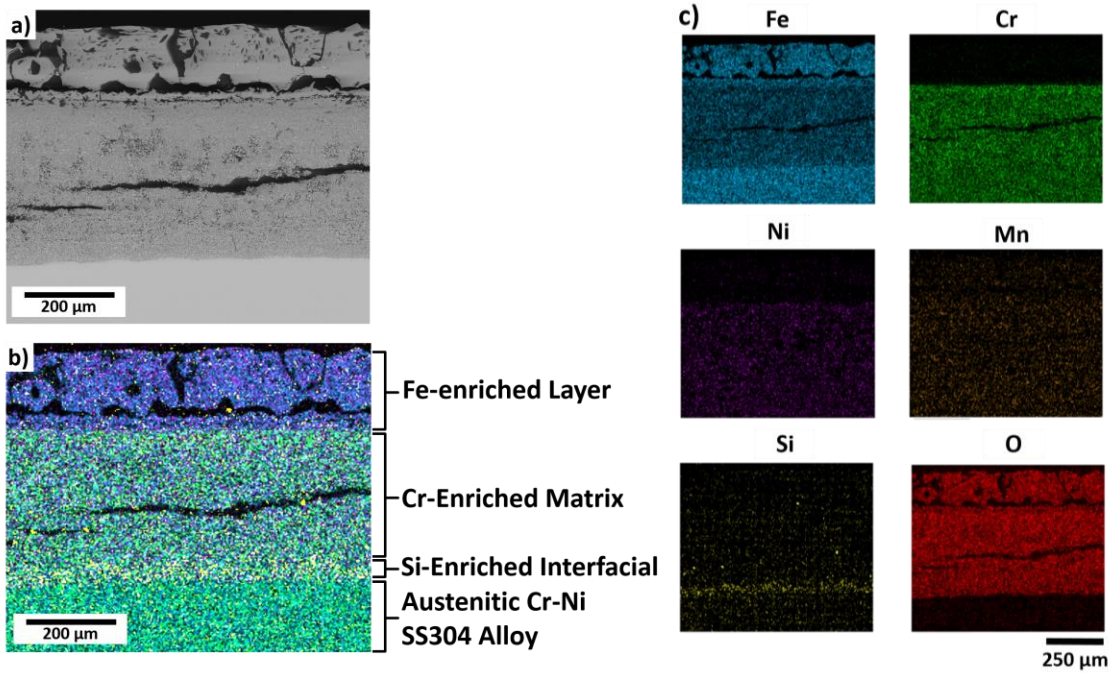


Data courtesy of J. Nuc. Mat. https://doi.org/10.1016/j.jnucmat.2021.153493


Focused Ion Beam (FIB)
Scanning Electron Microscopy (SEM)
Transmission Electron Microscopy (TEM)
EML

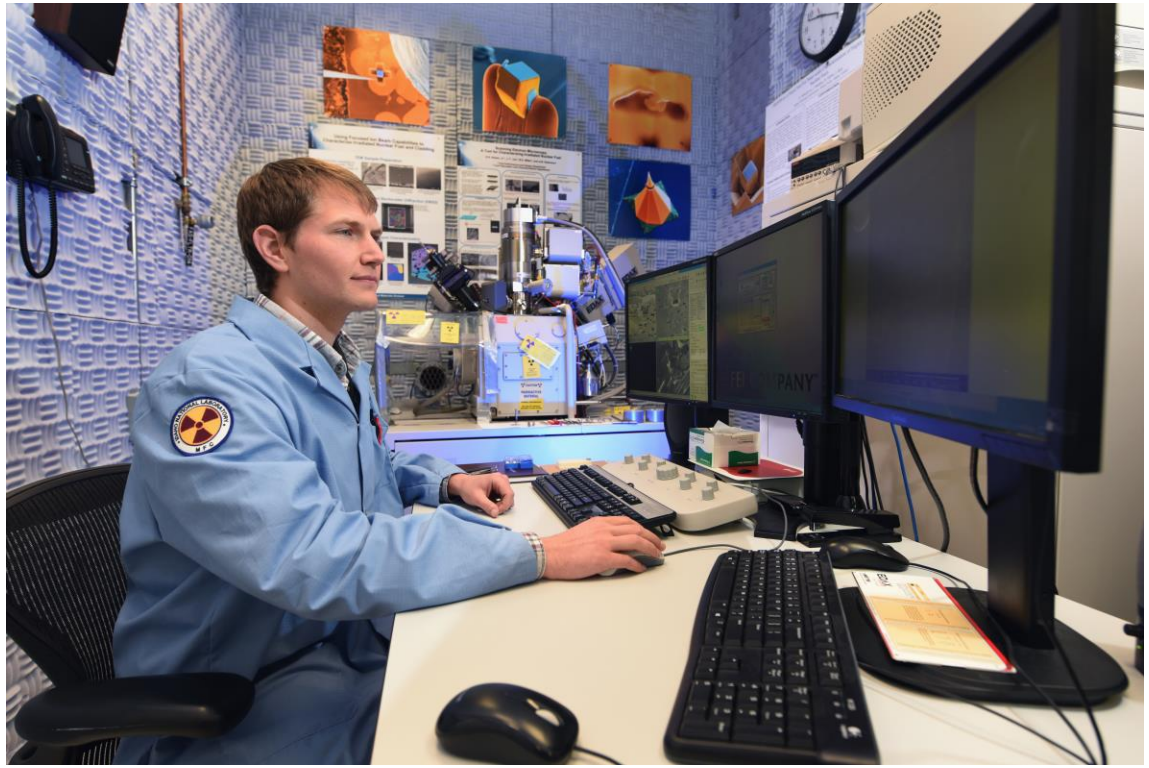
JEOL JSM-7000f SEM

Assessment of Stainless Steel 304 at the onset of a Beyond Design Basis Accident Temperatures in Steam

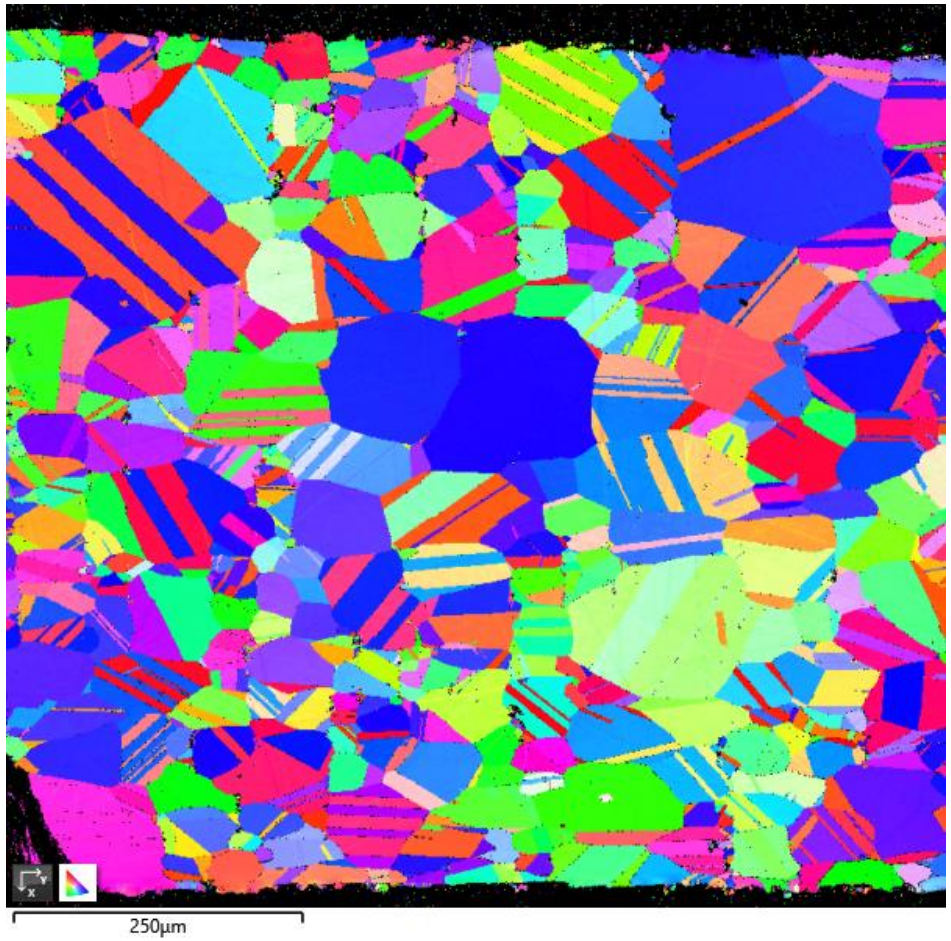


Data courtesy of T. Copeland-Johnson, C. Nyamekye, L. Ecker, N. Bowler, E. Smith, R. Rebak, and S. Gill, 2022, Corrosion Science (Submitted)

TESCAN Lyra 3 FIB

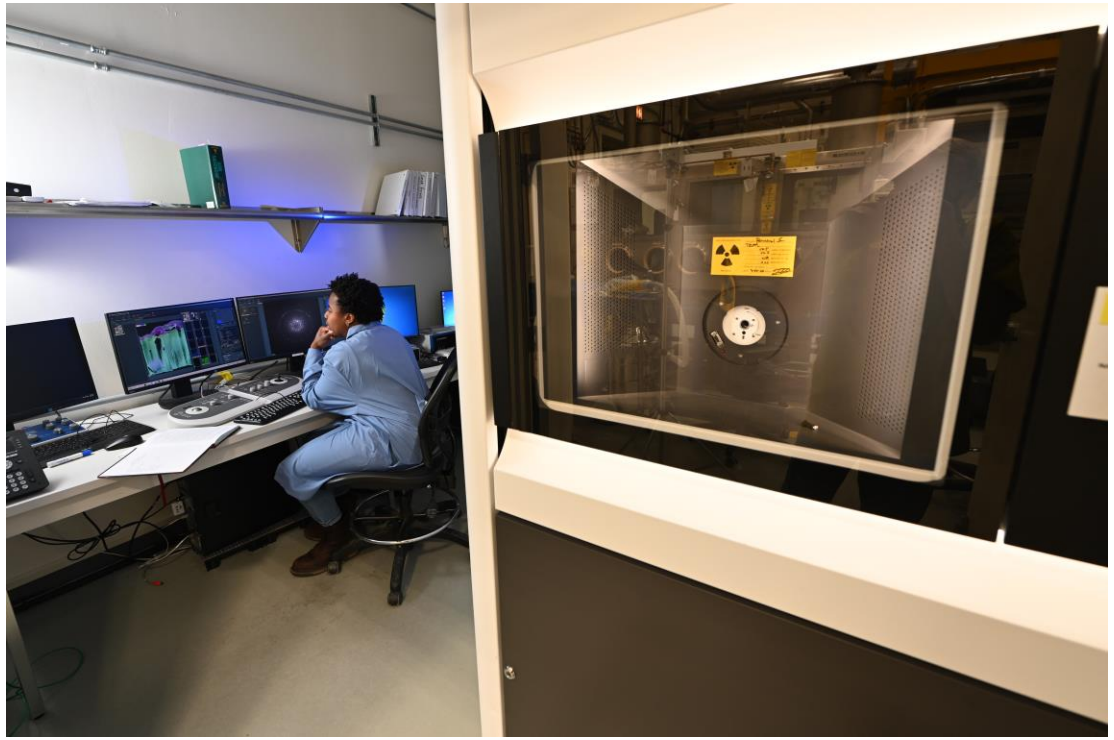


Microstructural Assessment of As-Received Inconel 617 before corrosion

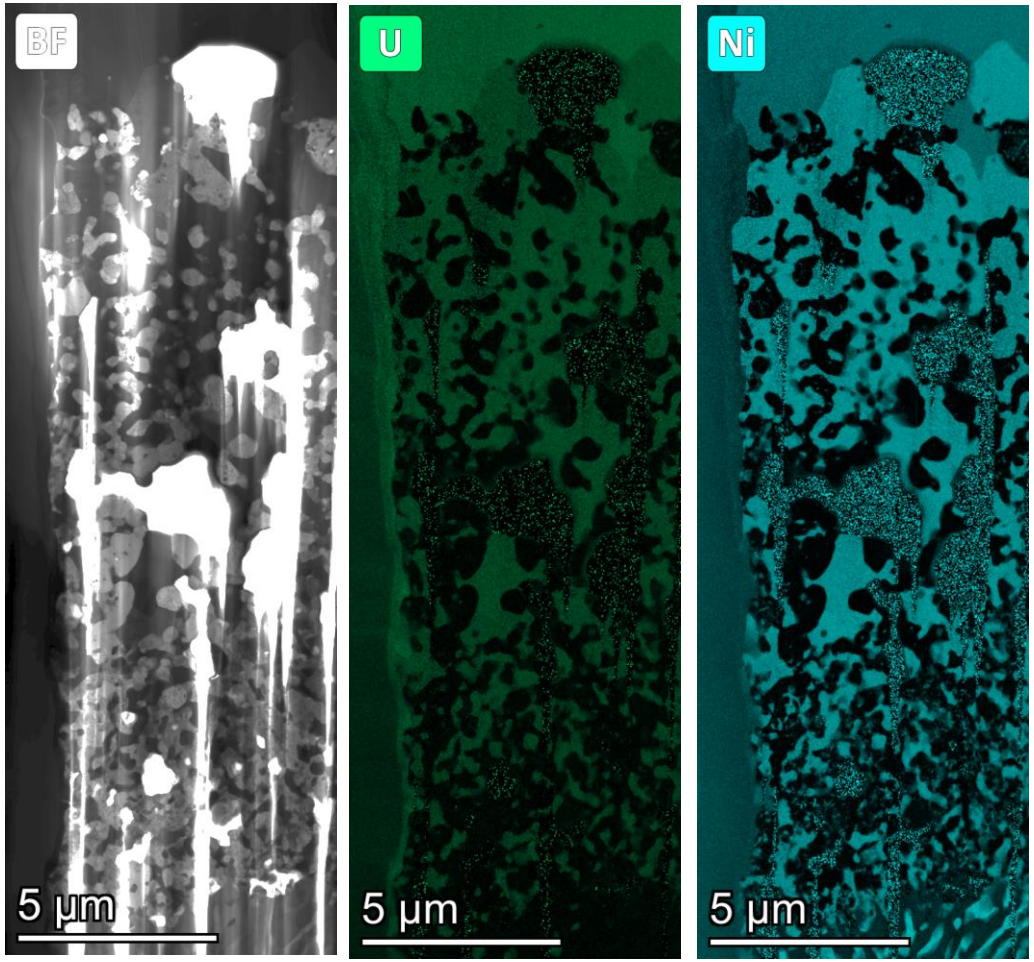


Data Courtesy of T.M Copeland-Johnson, D. Murray, G. Cao, 2022, Frontiers in Nuclear Engineering (Under Review)

ThermoFisher Talos TEM



Elemental Analysis of Inconel 617 after corrosion in UCl_3



Questions?

Correlated Characterization of Ni-based Superalloys Corroded in Uranium-containing Molten Salt Systems

Trishelle Copeland-Johnson, Daniel J. Murray, Guoping Cao, Lingfeng He

Commercialization of molten salt reactors to advance nuclear energy infrastructure

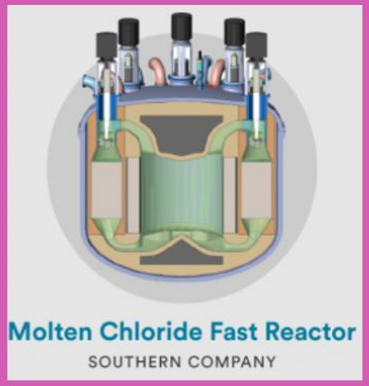
U.S. Energy Portfolio

"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

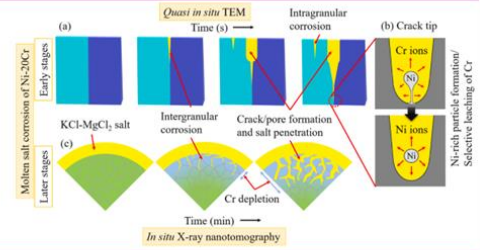
Advanced Reactor Development Program

Molten Salt Reactors



Advanced Reactor Development | Department of Energy

Knowledgebase for Qualification

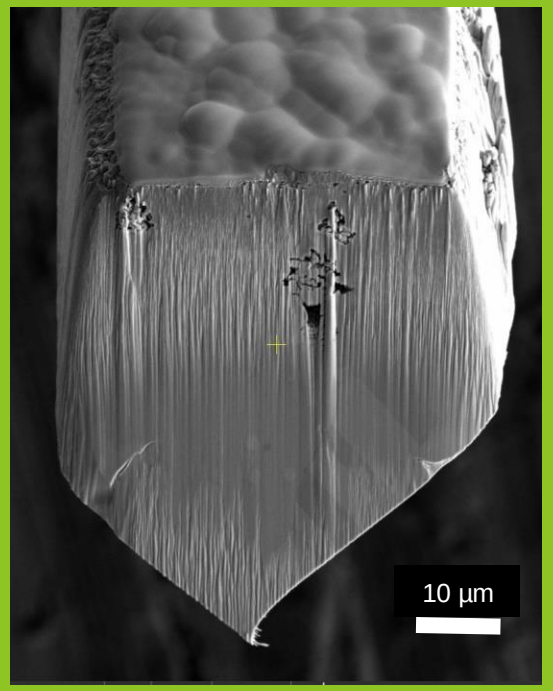


Bawane K, et al. (2021) *Corrosion Science*. https://doi.org/10.1016/j.corsci.2021.109962

Comprehensive qualification of nuclear structural alloys for construction of MSRs requires expansion of our knowledgebase on their corrosion performance in chloride molten salts through multimodal characterization.

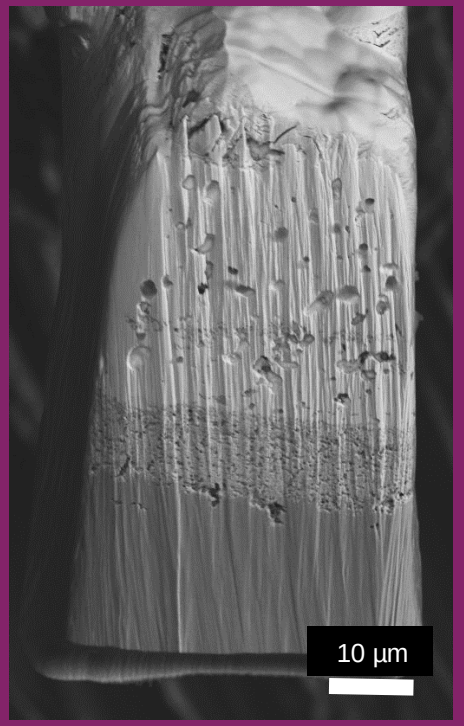
Assessment of Inconel 617 (A617) in chloride molten salt

Part I: No UCl_3



$NaCl\text{-}MgCl_2$ @ 700 °C (1000 h)

Part II: With UCl_3

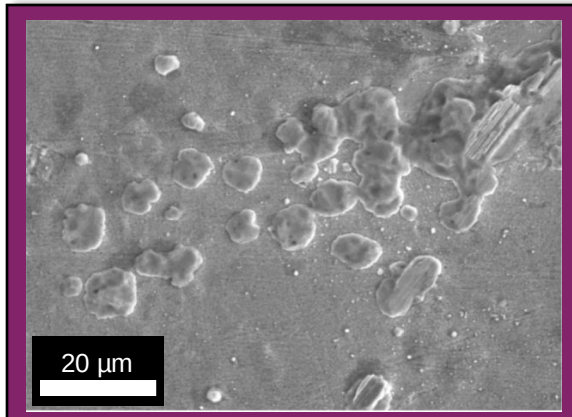


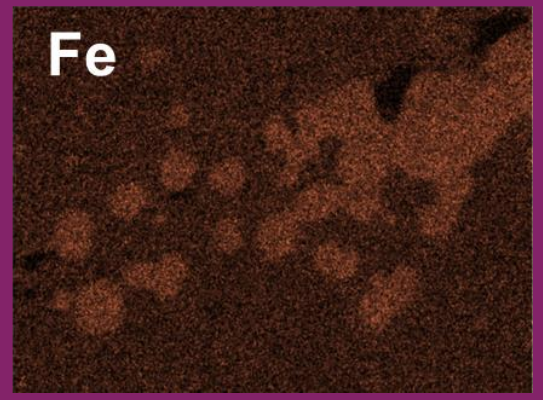
$LiCl\text{-}KCl\text{-}UCl_3$ @ 700 °C (1000 h)

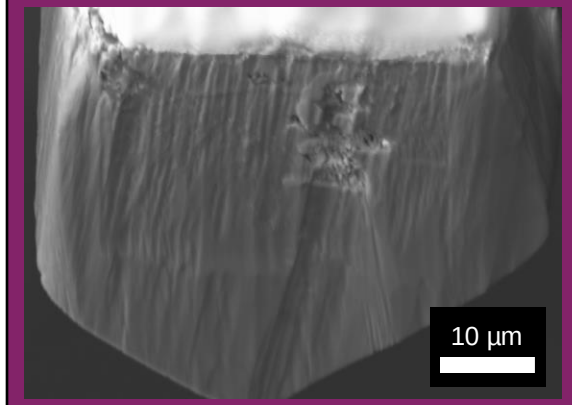
Correlated Characterization Workflow

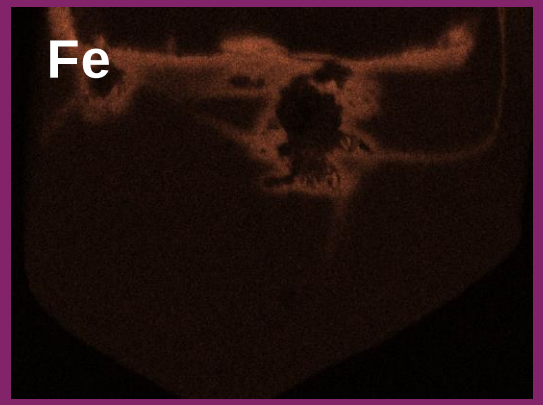
Global (Context) → **Local (Mechanism)**

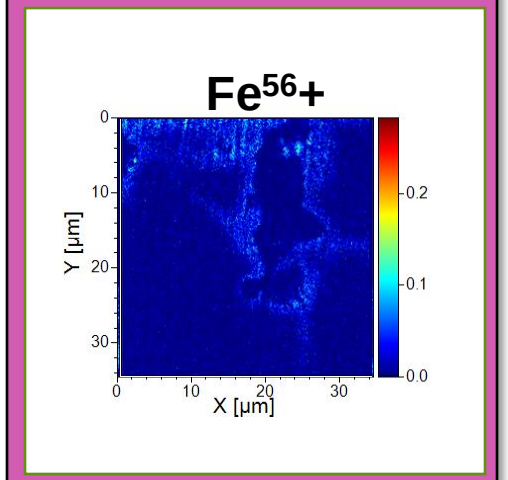
SEM/FIB - Imaging	SEM/FIB - Elemental	SEM/FIB - Chemical	TEM - Imaging	TEM - Elemental



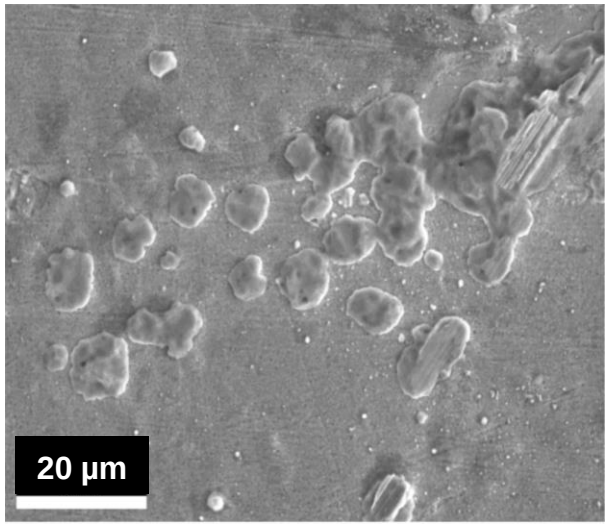


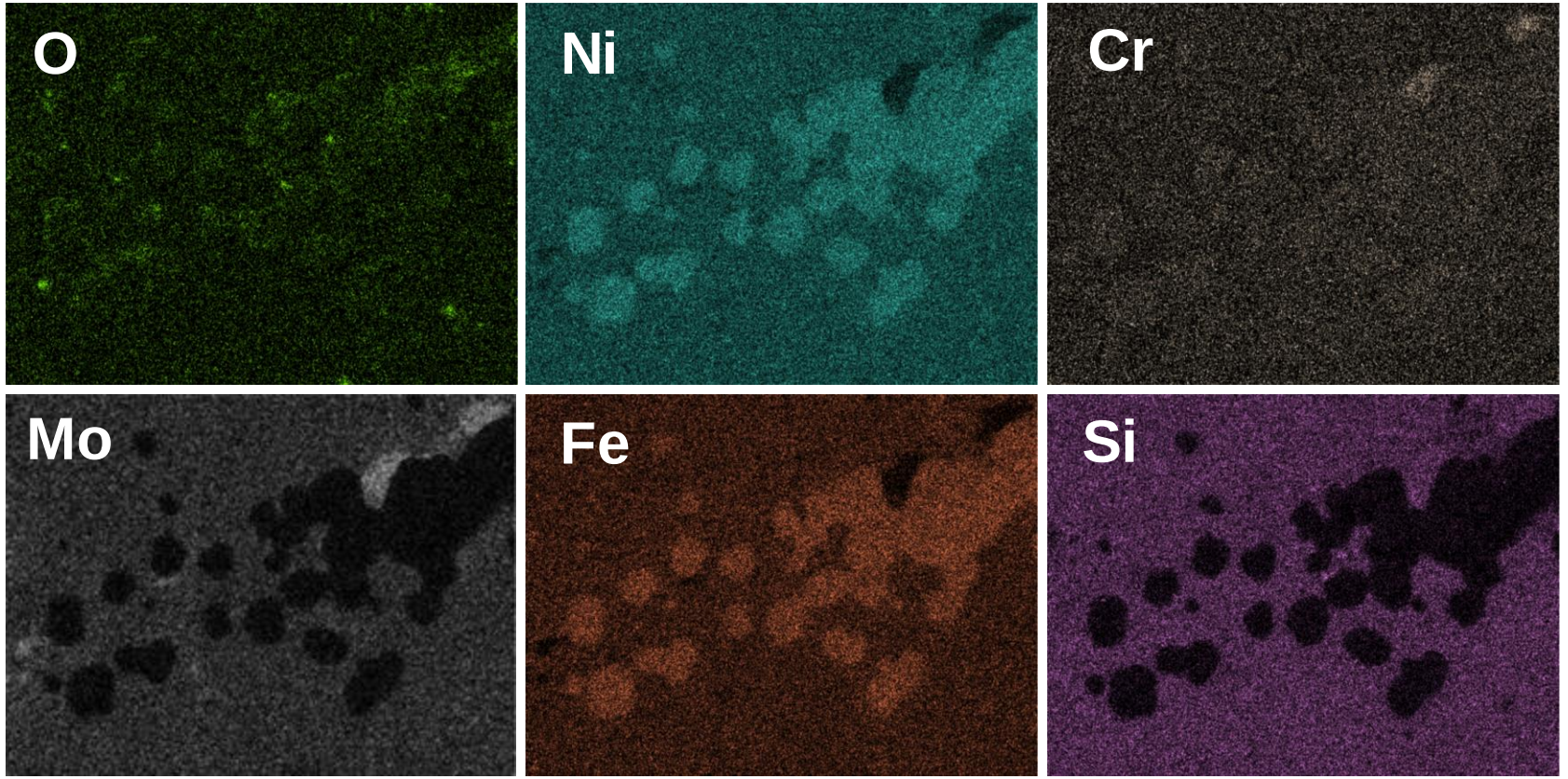






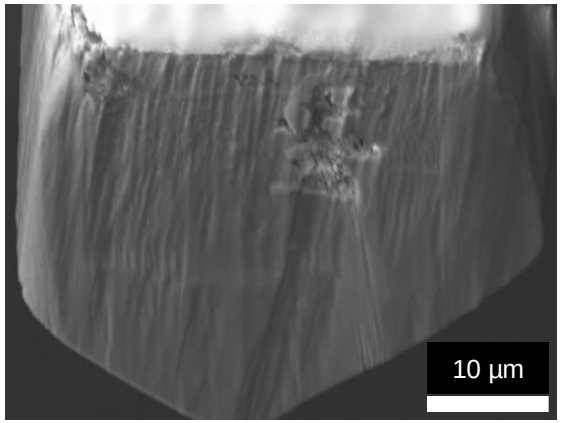
Elemental Analysis – Surface (Atomic %)



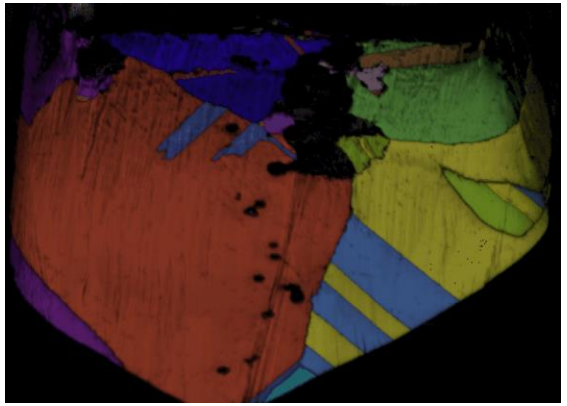


SEM surface analysis illustrates features globules enriched in primarily Ni and Fe based on elemental analysis.

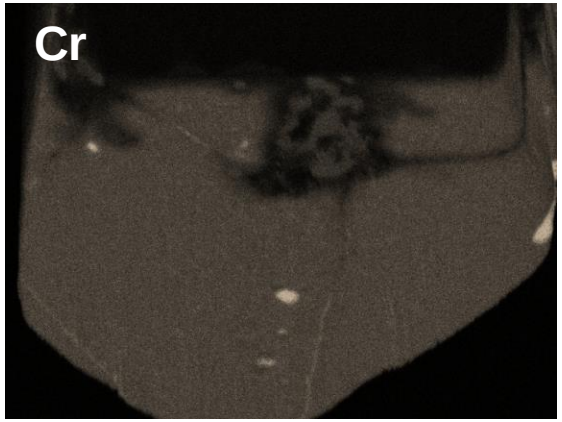
3D Reconstruction

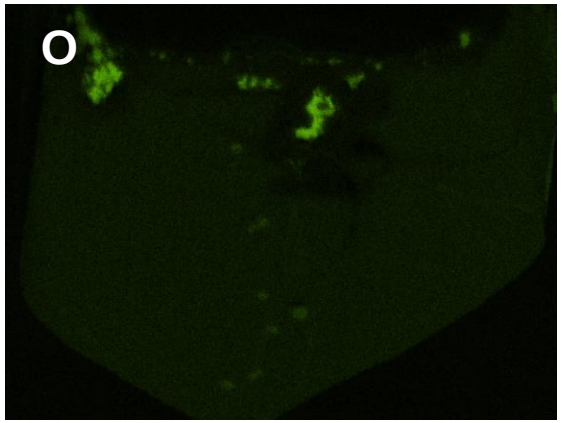


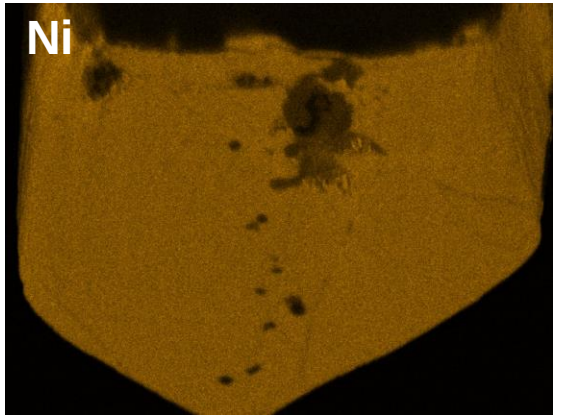
Microstructure

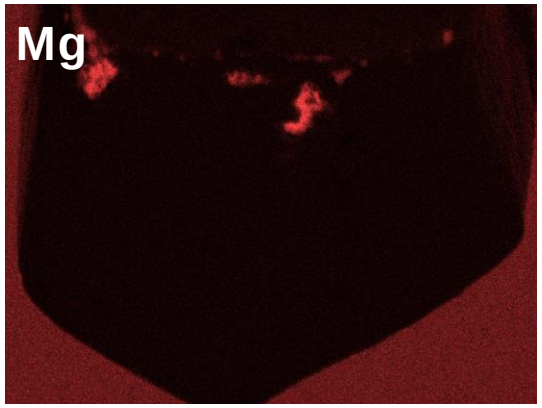


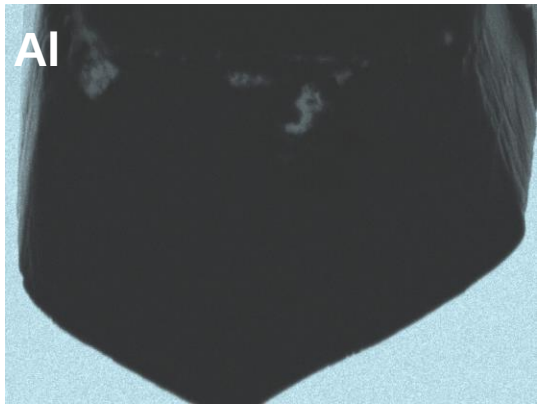
Elemental Analysis

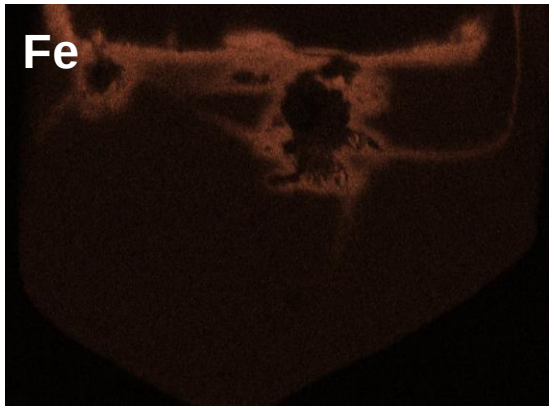






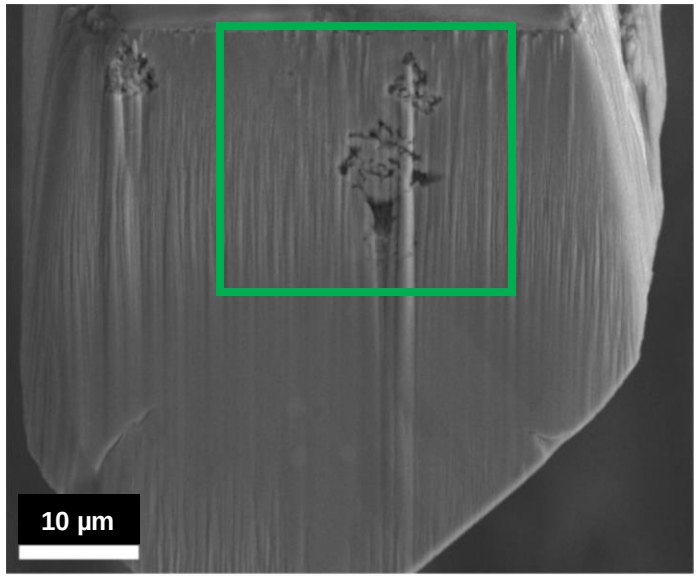


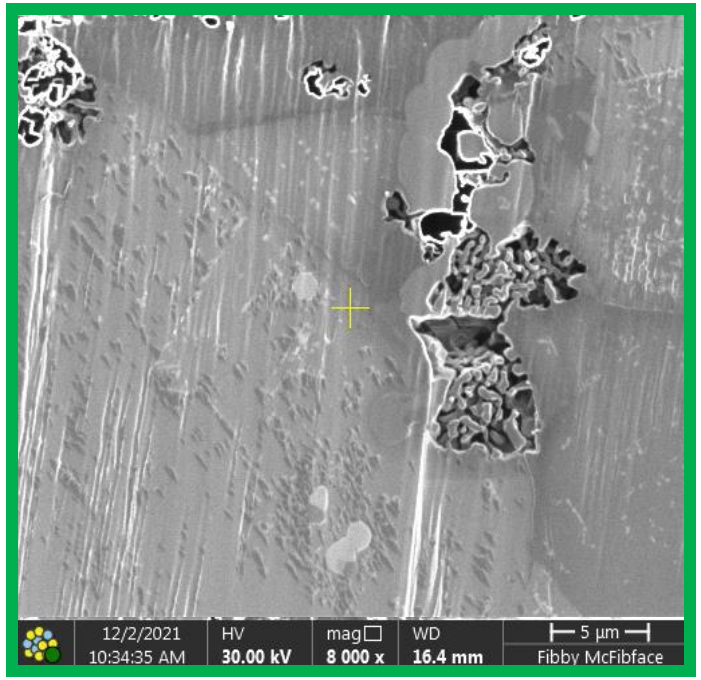


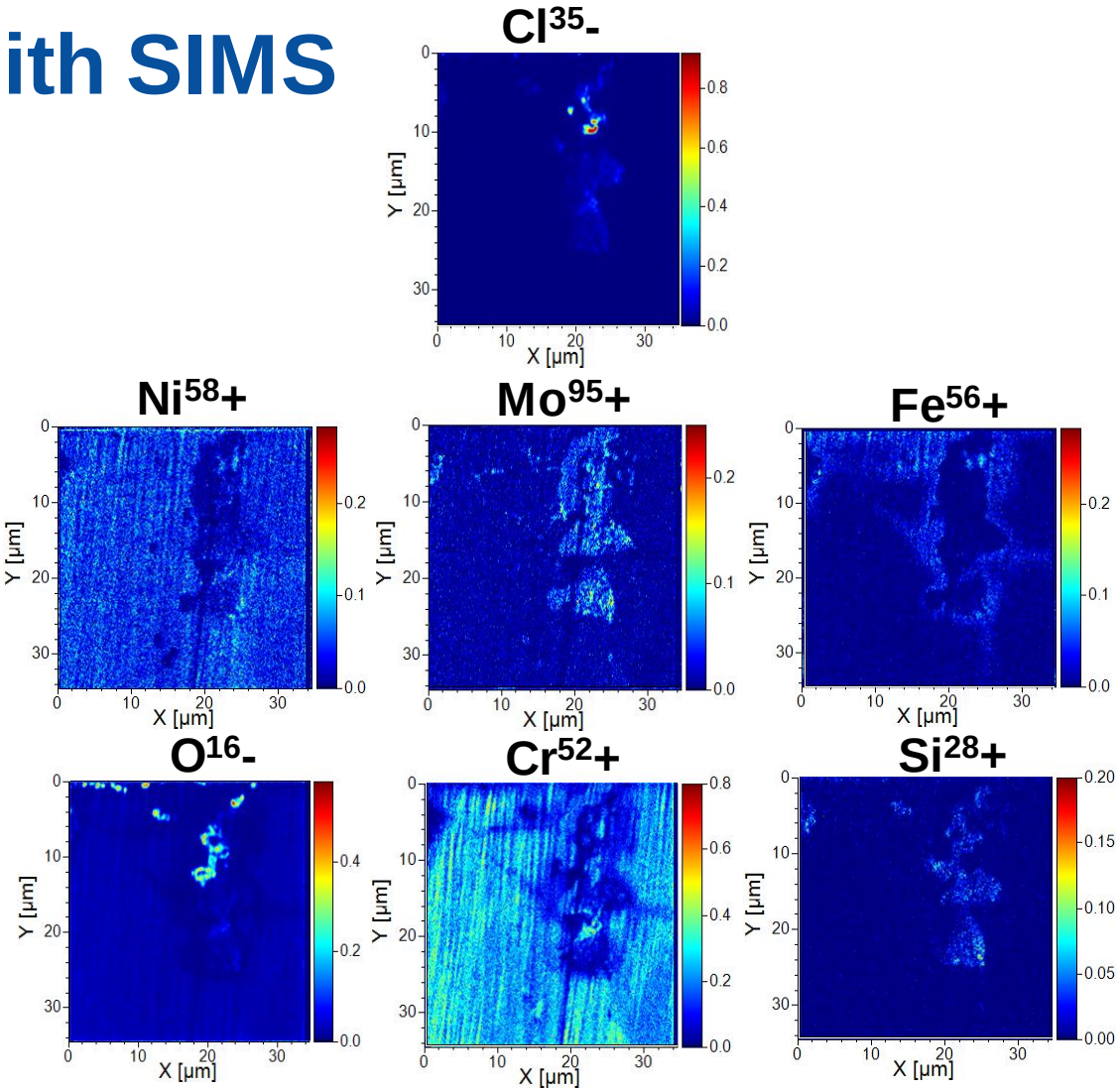


FIB can be utilized to construct a novel perspective of structural and elemental changes through 3D reconstruction, including dealloying of elements, Fe and Ni, intergranular Cr-O and Mg-Al-O based compounds

Verifying elemental analysis with SIMS

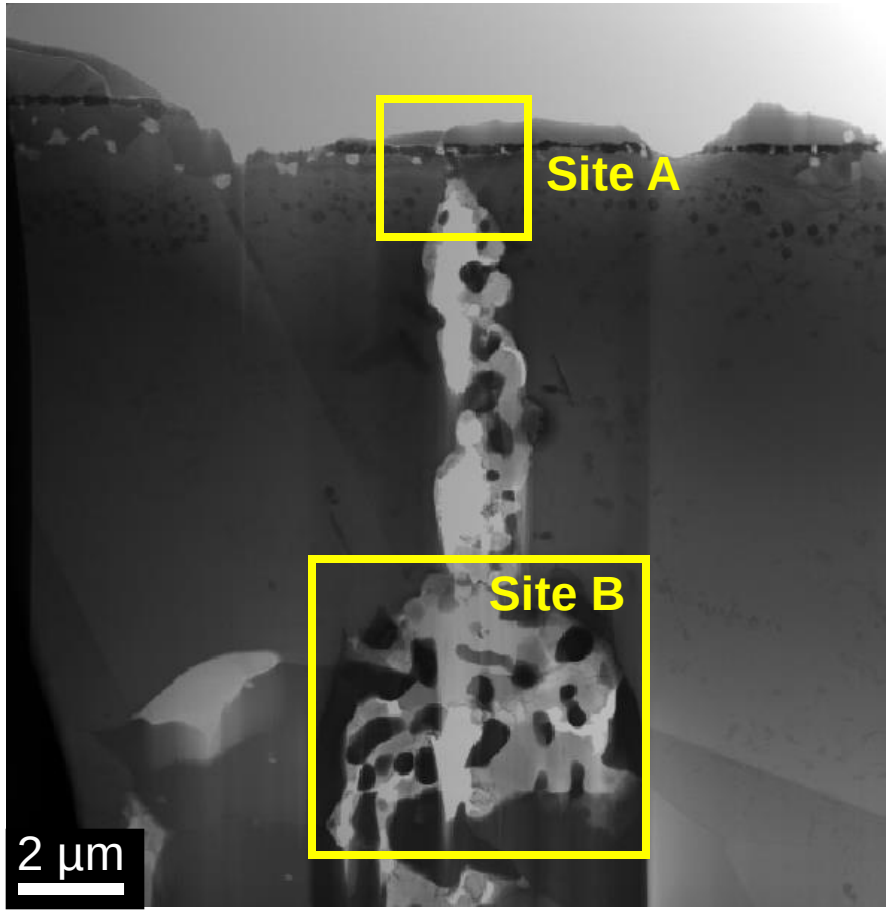


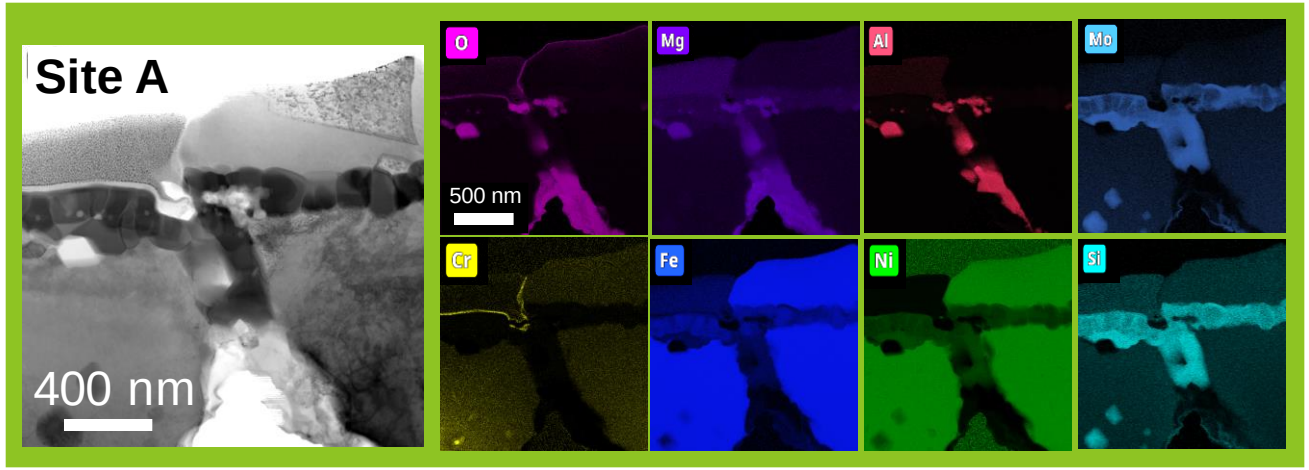


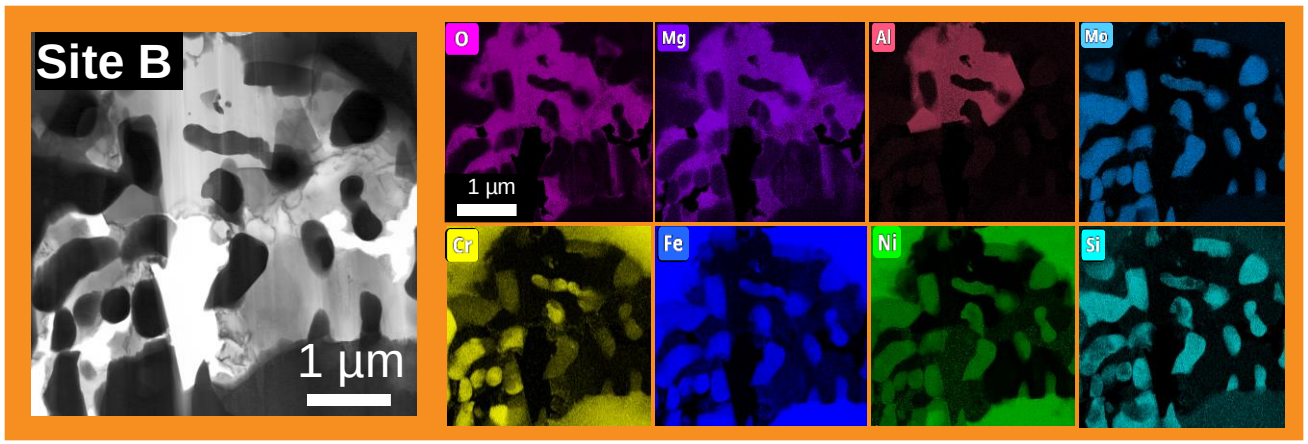


Secondary Ion Mass Spectroscopy (SIMS) verified EDS analysis, noting depletion of Fe, Ni, and Cr around the site that has been directly attacked by the salt (-Cl).

Site #1: Elemental Analysis (Atomic %)

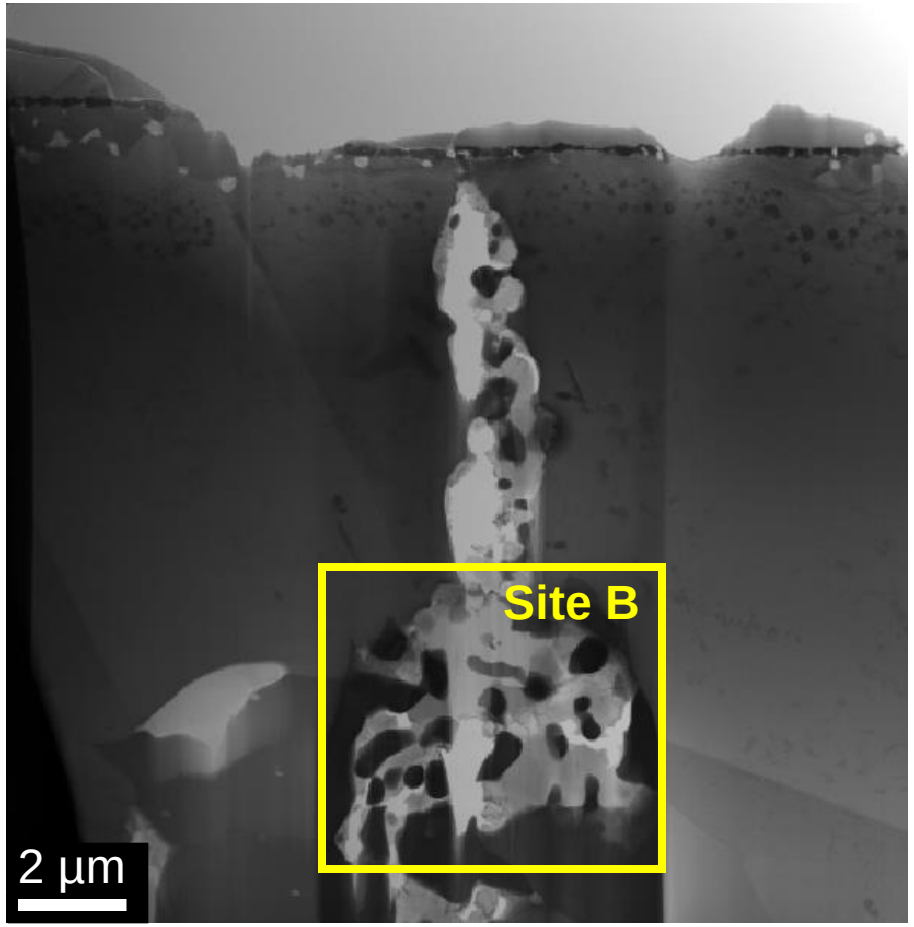


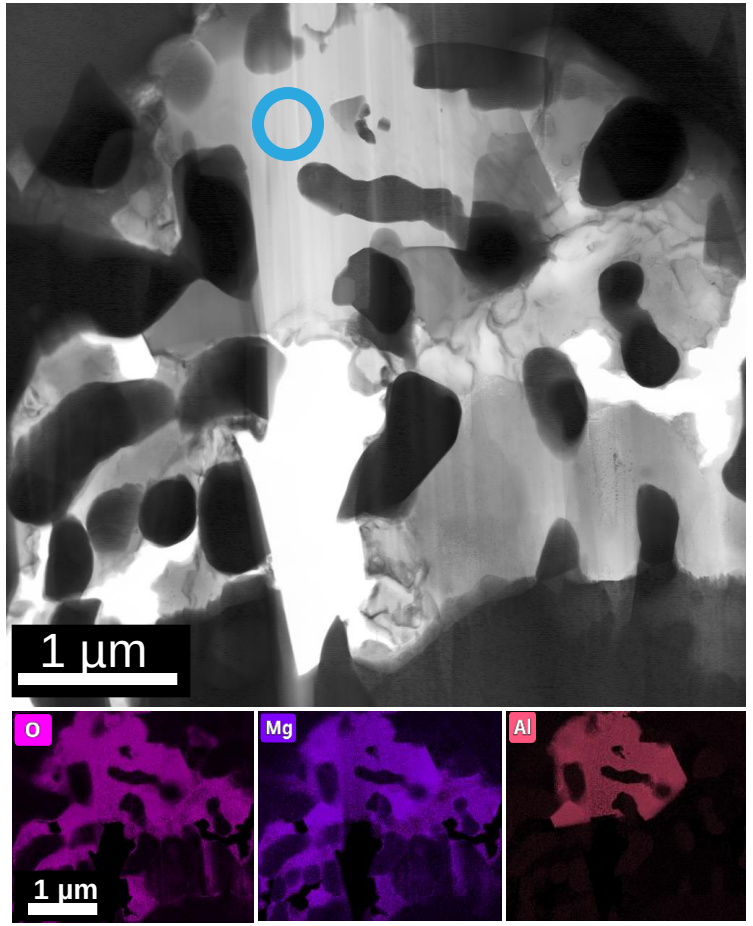


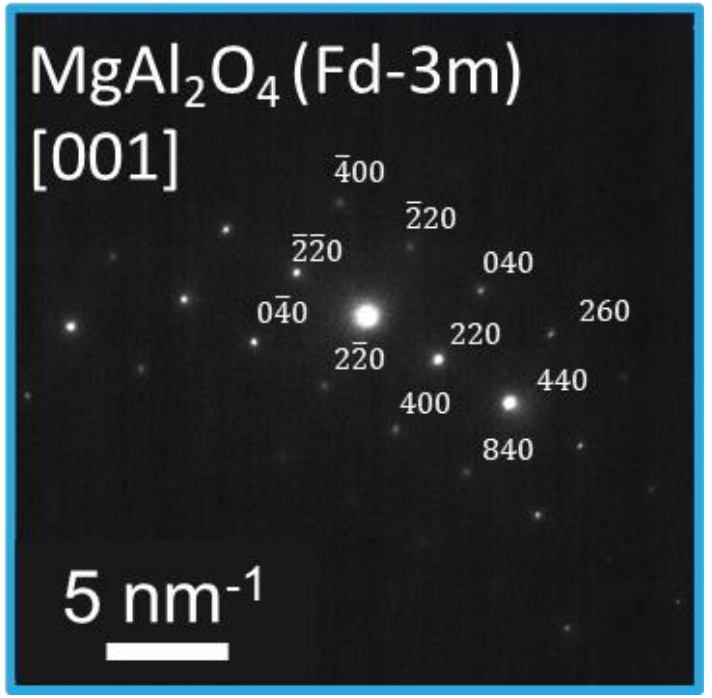


TEM elemental analysis corroborates with SEM, identifying Mo-Si and Mg-Al-O enriched sites where the salt was in direct contact with the alloy.

Site #1: Structural Analysis

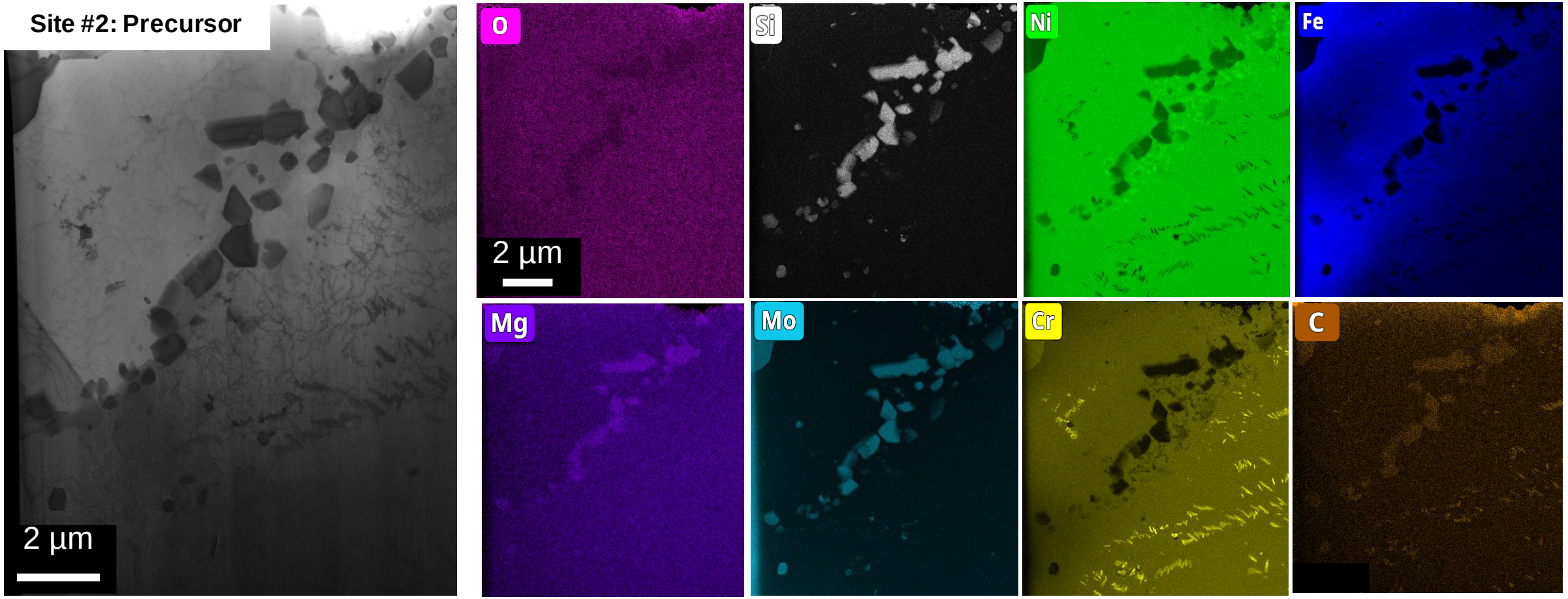






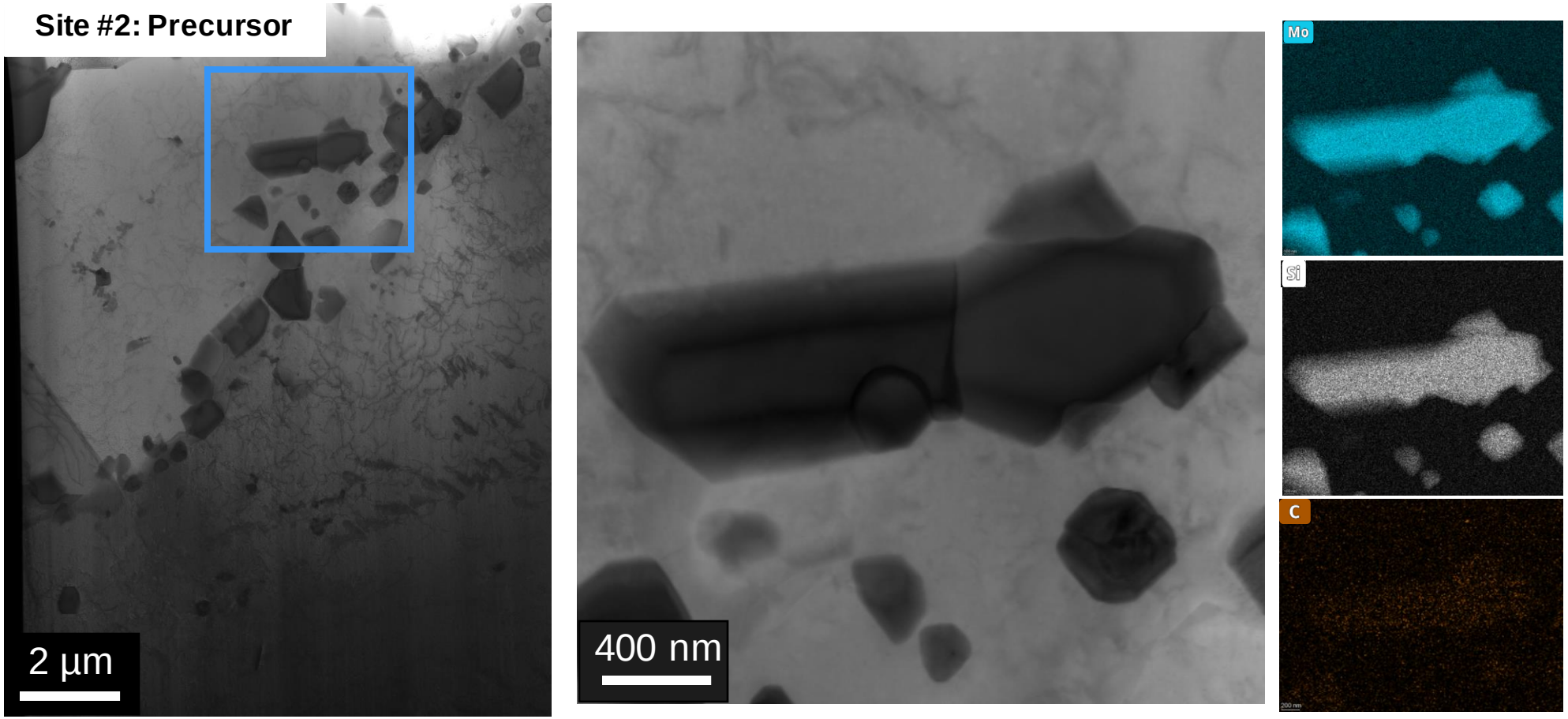
TEM structural analysis suggests that corrosion attack may have been intergranular, corresponding to the development of $MgAl_2O_4$ within the direct attack site

Site #2: Elemental Analysis



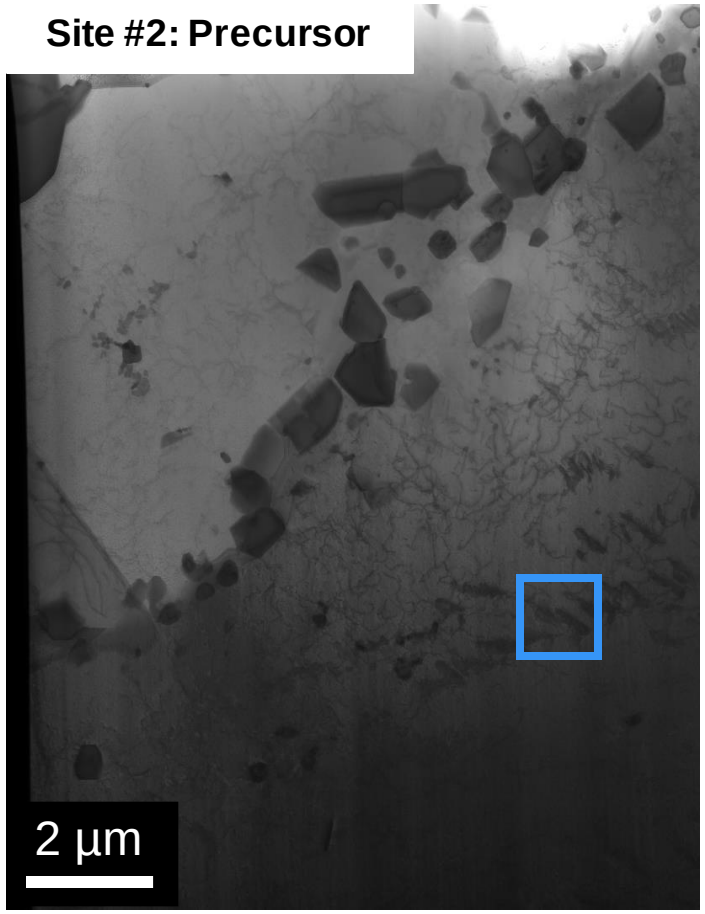
TEM elemental analysis of precursor attack site, notes development of Mo-Mg-Si-C and Cr-C enriched precipitates. Fe enrichment observed around the Mo-Mg-Si-C enriched precipitates.

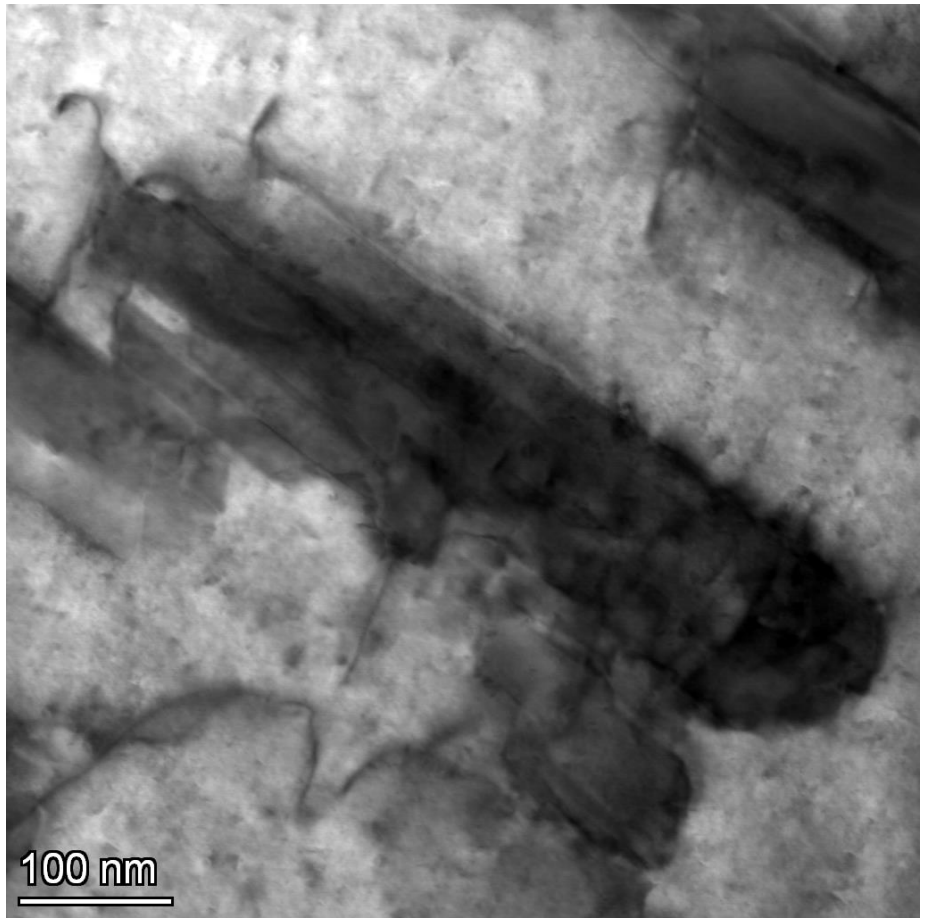
Site #2: Elemental Analysis

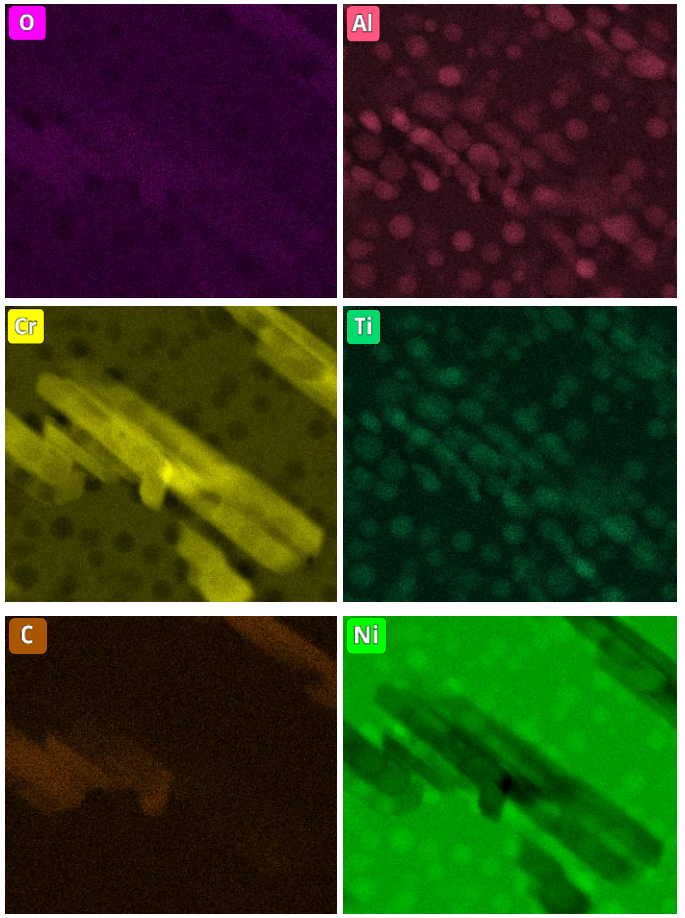


TEM elemental analysis of precursor attack site, notes development of Mo-Mg-Si-C and Cr-C enriched precipitates. Fe enrichment observed around the Mo-Mg-Si-C enriched precipitates.

Site #2: Elemental Analysis

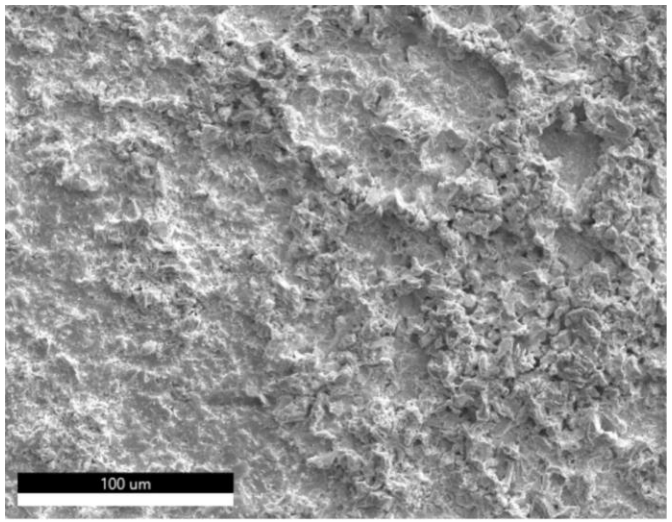


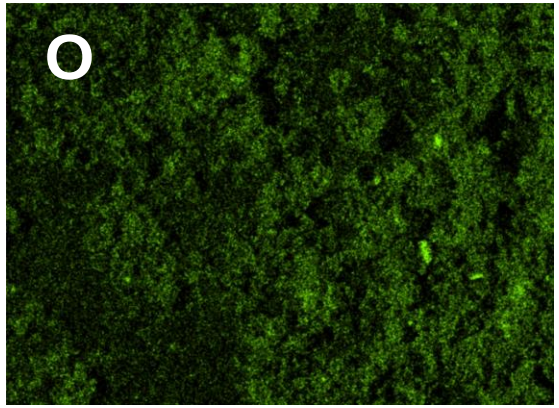


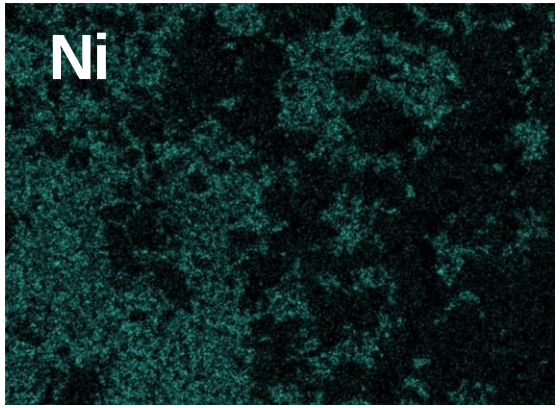


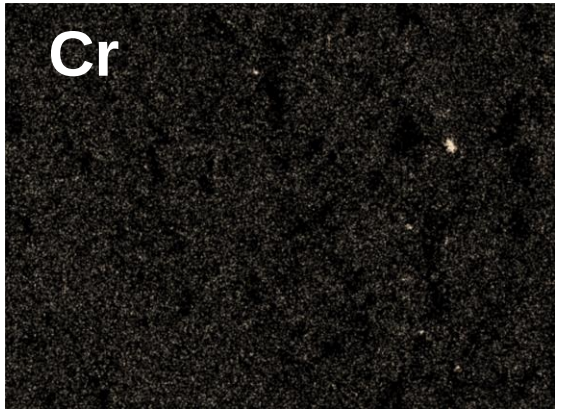
Cr-C enriched precipitates show a slight enrichment in O compared to the surrounding matrix. Ni-Al-Ti enriched sites are also observed in the surrounding matrix may correspond to γ' ($Ni_3(Al,Ti)$) precipitates.

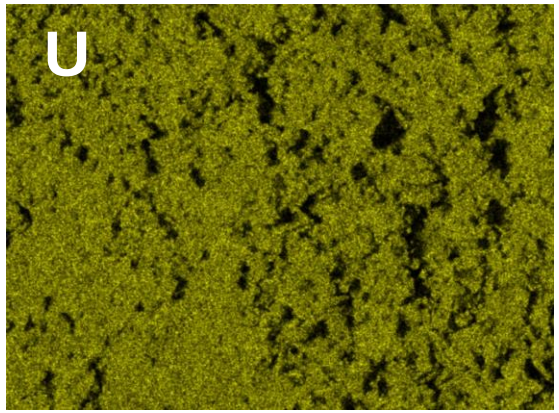
Elemental Analysis – Surface (Atomic %)

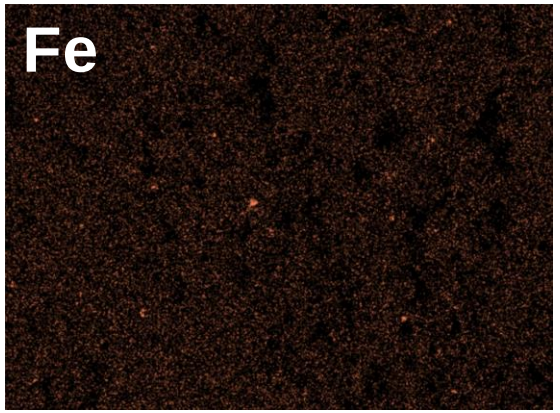


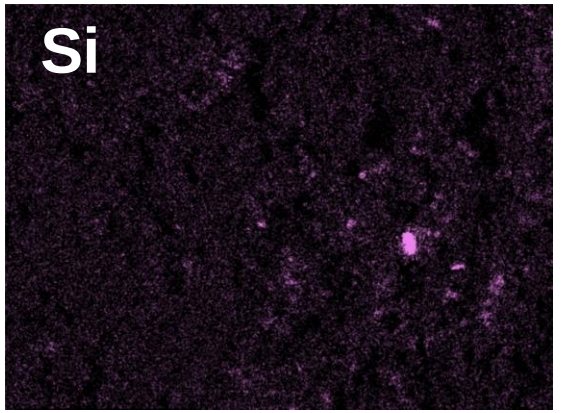








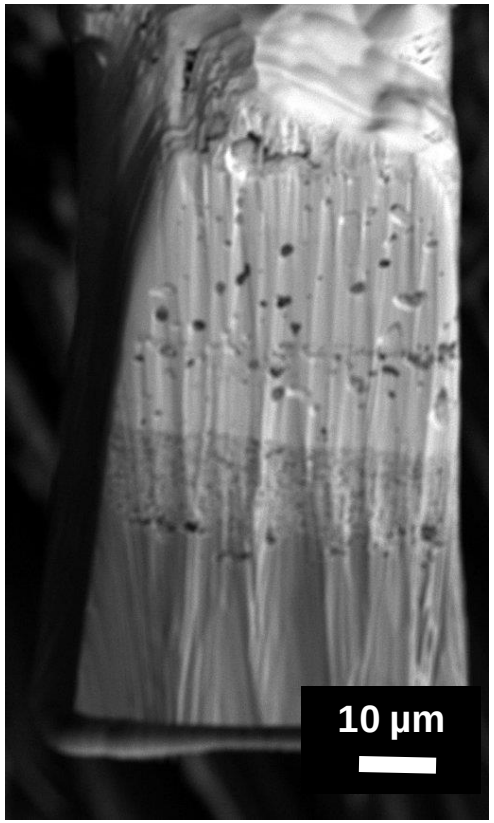


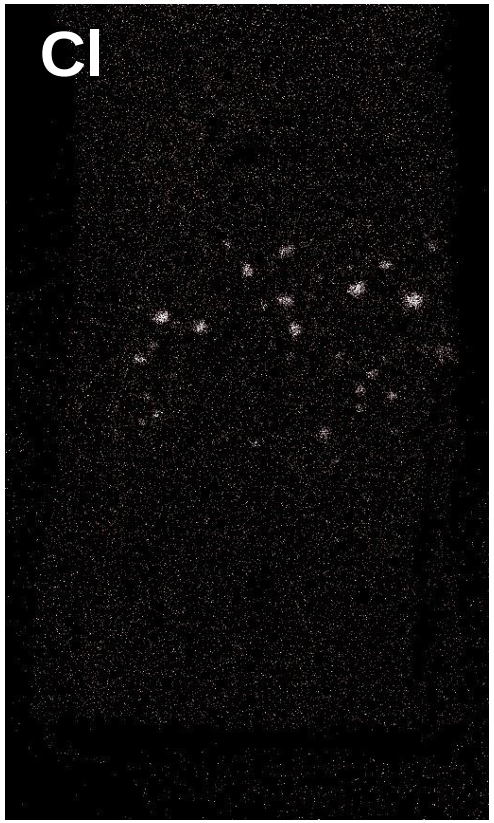


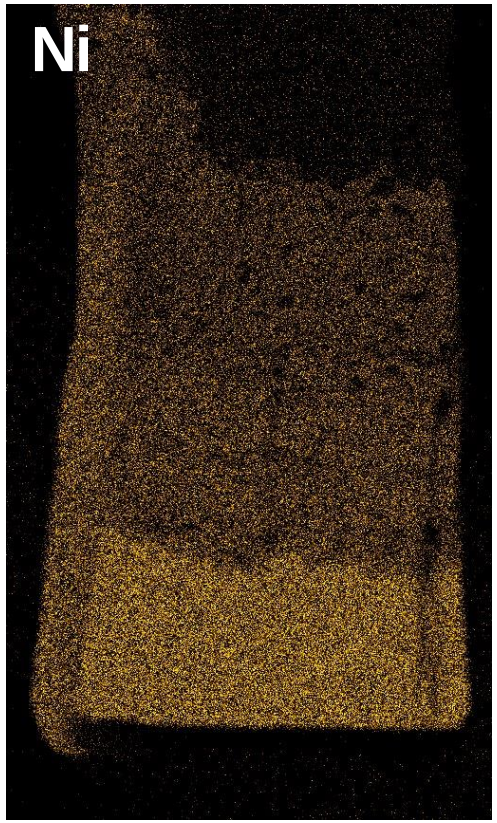
SEM surface analysis illustrates features primarily U based on elemental analysis.

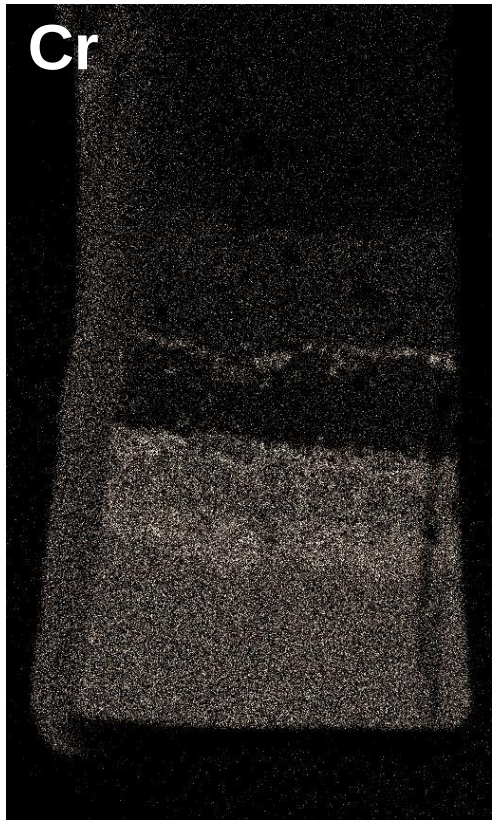
3D Reconstruction

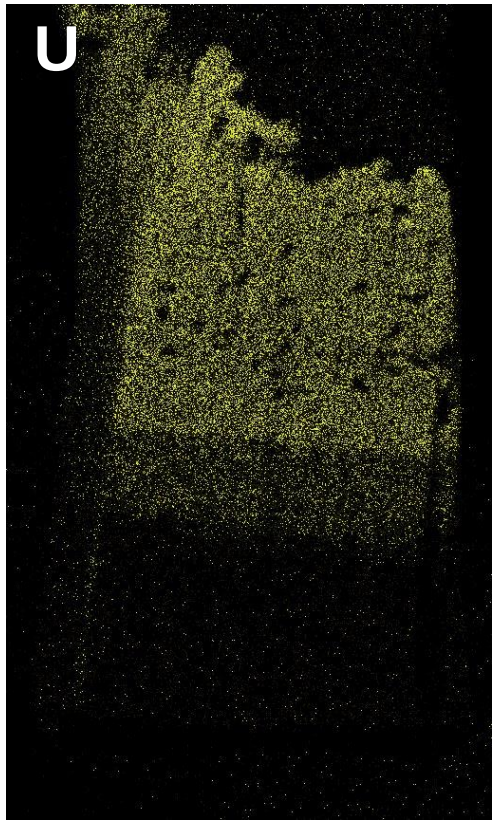
Elemental Analysis





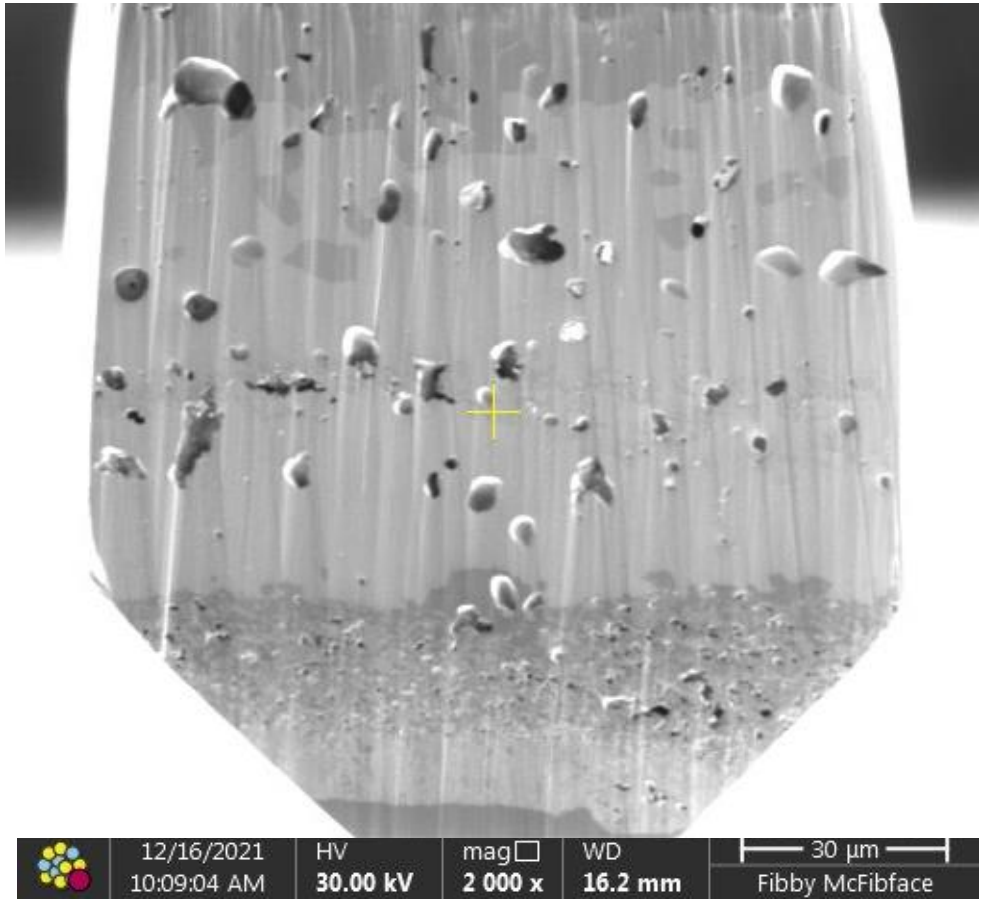


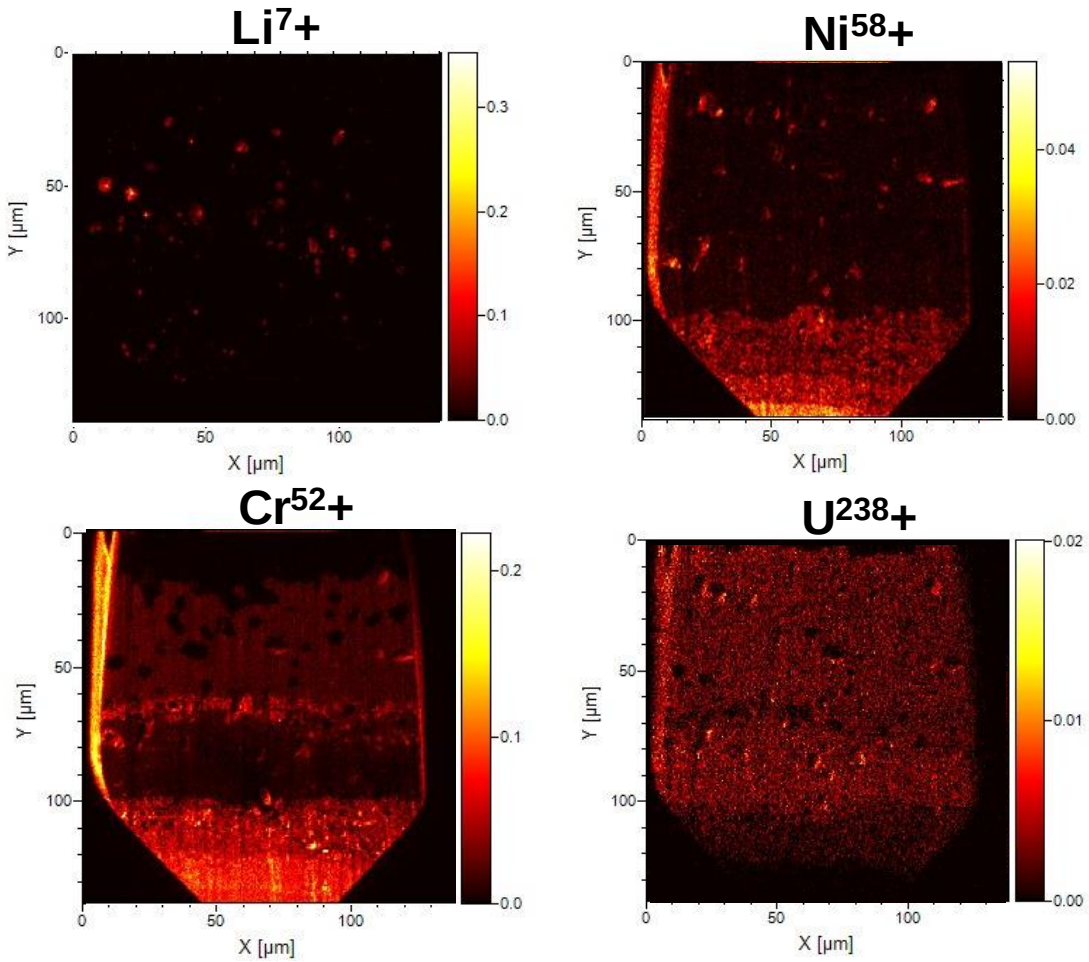




3D FIB reconstruction notes consistent presence reveals that features observed at the surface propagate extensively into the bulk material, corresponding to a porous U-based alloy with selective regions of Cr enrichment.

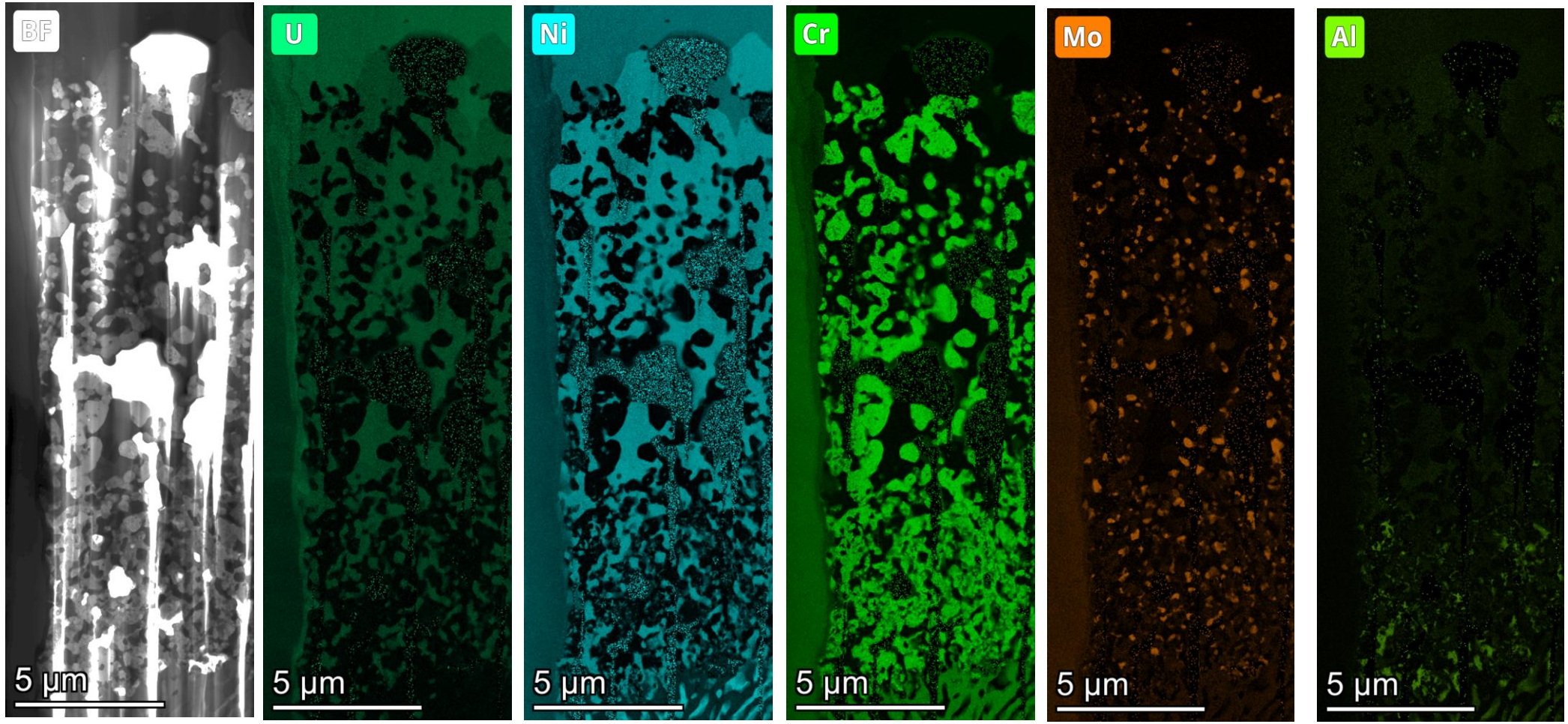
Verifying Elemental Analysis with SIMS





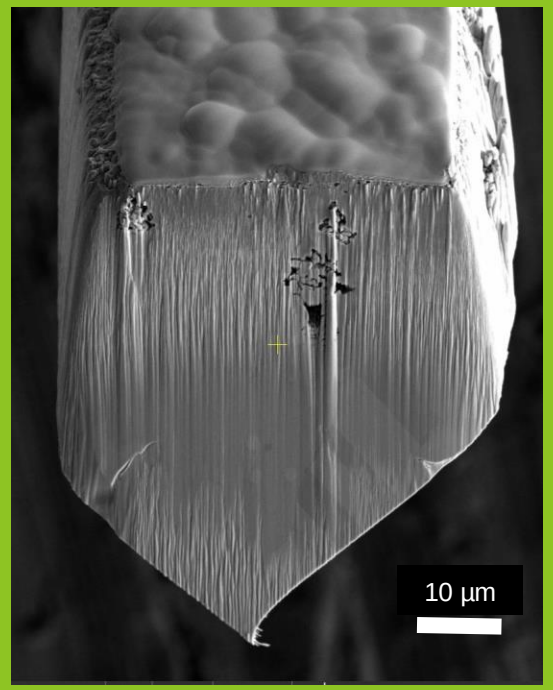
Chemical analysis of isotopic species using SIMS corroborates and enhances EDS elemental analysis, detecting lighter species, such as Li, originating from the salt still residing within pores at higher resolution and sensitivity.

TEM Analysis



TEM analysis notes phase separation of Ni, Cr, Mo, and Al within the U-Ni alloy region.

Results



$NaCl\text{-}MgCl_2$ @ 700 °C (1000 h)

- Presence of intergranular Cr-O
- Fe and Ni dealloying
- Mg-Al-O products in porous network

$LiCl\text{-}KCl\text{-}UCl_3$ @ 700 °C (1000 h)

- U-Ni alloy formation
- Cr, Mo, Al phase separation in U-Ni alloy regime

Acknowledgements

- Laboratory Directed Research & Development (LDRD) Office
- INL Glenn T. Seaborg Institute
- Characterization and Advanced Post-Irradiation Examination (CAPIE) Division
  - Advanced Ion Characterization and Micro-mechanics (AICM) Group
  - Materials Informatics & Transmission Electron Microscopy (MI&TEM)
  - Nuclear Structural Materials (NSM) Group
- Fuel Cycle Science & Technology Division
  - Pyrochemistry & Molten Salt Systems Department
    - Ruchi Gakhar
    - Guoping Cao
    - Michael Woods