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Exceptional service in the national interest

Characterization Tools for Failure Analysis and for Enabling Materials Discovery

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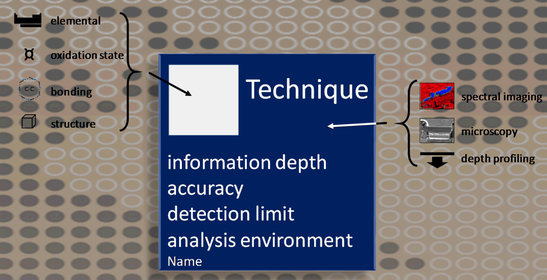


The Materials Center at Sandia National Laboratories draws from extensive experience in assessing aging and reliability relevant to the nuclear weapons stockpile. This core capability spans expertise in accelerated aging and testing to manufacturing troubleshooting to root-cause failure analysis. In DuraMat, fundamental analysis with advanced characterization tools will be critical for developing new materials and for forensic investigations of degraded modules. At hand are comprehensive surface science facilities including photoemission spectroscopy, Auger, AFM, profilometry, and SIMS. Surface analysis allows for characterization of the entire periodic table at nanometer scale depth resolution. Several aging-specific capabilities have been developed that are directly applicable to the DuraMat mission, including *in situ* thermal treatments and depositions. Depth profiling can be performed to evaluate buried layers and interfaces revealing information on diffusion, corrosion, or potential-induced degradation. A newly installed XPS is uniquely equipped with a gas cluster ion source for depth profiling organic materials, potentially relevant to understanding delamination or polymer degradation. The instrument also has IPES and UPS for examining electronic state information, as well as several X-ray sources for interrogating different depths in the near surface. Ambient pressure XRF for examining large area modules, interconnects, and solder has also recently been installed. Electron microscopy capabilities include several SEMs, FIB-SEMs, and WDS microprobe. SNL has state-of-the-art AC-STEM (with 0.8 Å resolution) and multi-STEM (for large area (~cm) imaging at nanoscale resolution). Electron microscopy can be used to assess structural changes in module materials, at interfaces, and at grain boundaries. Extensive X-ray diffraction, Raman, IR, ICP, and other capabilities add to the comprehensive capabilities in the Materials program. A unique core competency at SNL is the co-location of numerous advanced instruments coupled with a breadth of materials characterization experience and a culture of awareness for aging-related reliability.

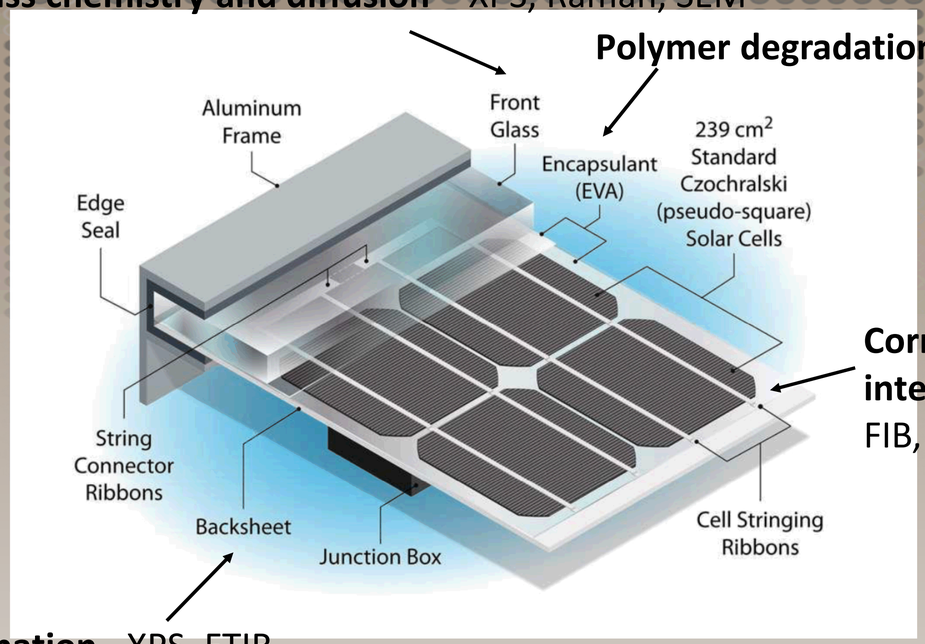
A Partial Periodic Table of Characterization at SNL

XPS 1-6 nm ±10% 0.1 at.% in ultra high vacuum X-ray Photoelectron Spectroscopy	**ToF-SIMS** 0.2-1 nm ±50% ppb ultra high vacuum Time of Flight Secondary Ion Mass Spectrometry	**AES** 1-5 nm ±30% 1 at.% ultra high vacuum Auger Electron Spectroscopy	**AFM** 1 nm N/A N/A air Atomic Force Microscopy	**OP** 50 nm N/A N/A air Optical Profilometry
SEM-EDS 0.3-3 µm ±10% 1 at.% high vacuum Secondary Electron Microscopy Energy Dispersive Spectroscopy	**mSEM** 0.3-3 µm N/A N/A high vacuum Multi - Secondary Electron Microscopy	**EBSD** 0.3-3 µm N/A N/A high vacuum Electron Backscatter Diffraction	**FT-IR** 0.5-2.0 µm ±10% 0.1 wt.% in air Fourier Transmission Infrared	**Raman** 0.5-5.0 µm ±10% 1 wt.% in air Raman Spectroscopy
FIB 0.1-100 µm ±10% 1 at.% high vacuum Focused Ion Beam	**AC-STEM** 2-100 nm ±20% 0.5 at.% ultra high vacuum Aberration Corrected Scanning Tunneling Electron Microscopy	**TEM-EELS** 0.8-100 nm ±20% 0.5 at.% ultra high vacuum Electron Energy Loss Spectroscopy	**GD-AES** no spatial resolution ±1% ppm vacuum Glow Discharge Atomic Emission	**ICP-OES** no spatial resolution ±1% ppm digested samples Inductively Coupled Plasma Optical Emission Spectroscopy
WDS 0.3-3 µm ±10% 0.1 at.% high vacuum MicroProbe Wavelength Dispersive Spectroscopy	**XRD** 10 µm ±5% 1 at.% in air X-ray Diffraction	**XRR** 10 nm ±1% N/A in air X-ray Reflectivity	**XRF** 0.1-5.0 µm ±10% ppt in air or vacuum X-ray Fluorescence	**ICP-MS** no spatial resolution ±1% ppb digested samples Inductively Coupled Plasma Mass Spectrometry

A few selected failure modes and possible routes towards investigation

Glass chemistry and diffusion – XPS, Raman, SEM

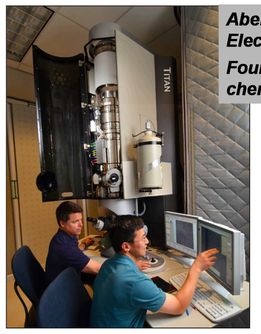
Polymer degradation – XPS, SIMS, FTIR



Corrosion/bonding/ intermetallics – SEM, FIB, TEM

delamination– XPS, FTIR

Unique capabilities in the Materials Characterization and Performance Department at Sandia

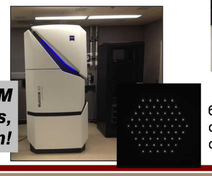


Aberration-Corrected Scanning Transmission Electron Microscope can image at 0.8Å resolution

Four in-column detectors provide 100X improvement in chemical mapping

Ga+ and Plasma FIB capabilities with 3D EBSD, Large area X-sections, Slice-N-View, and FIB-STEM

Surface analysis with gas-cluster sputtering, UPS, Al or Ag X-ray, heat/cool, IPES



Multi-Beam SEM High throughput, 1.22 GPixels/s, over mm areas at 4 nm resolution!

61 focused electron beams create secondary electrons collected in parallel

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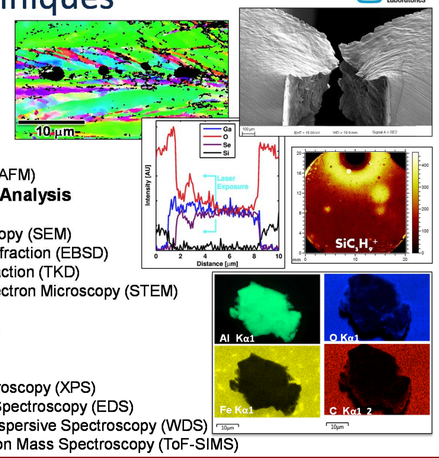
Breadth of Techniques

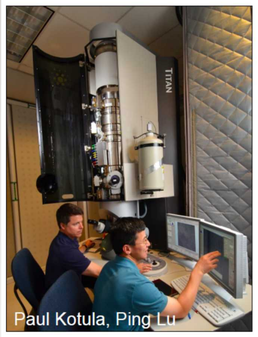
- **Sample Preparation**
  - Cleaning Lab
  - Metallography Lab
  - Focused Ion Beam (FIB)
- **Surface Topography**
  - Profilometry
  - Interferometry
  - Atomic Force Microscopy (AFM)
- **Microstructural Imaging & Analysis**
  - Optical Microscopy
  - Scanning Electron Microscopy (SEM)
  - Electron BackScattered Diffraction (EBSD)
  - Transmission Kikuchi Diffraction (TKD)
  - Scanning Transmission Electron Microscopy (STEM)
- **Compositional Analysis**
  - Auger Spectroscopy (AES)
  - X-ray Diffraction (XRD)
  - X-ray Fluorescence (XRF)
  - X-ray Photoelectron Spectroscopy (XPS)
  - Energy Dispersive X-Ray Spectroscopy (EDS)
  - MicroProbe Wavelength Dispersive Spectroscopy (WDS)
  - Time of Flight Secondary Ion Mass Spectroscopy (ToF-SIMS)

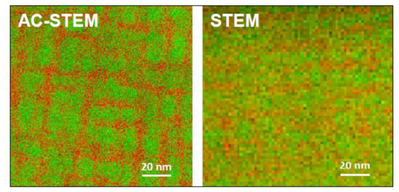


AC-STEM

Our state-of-the-art, Aberration-Corrected Scanning Transmission Electron Microscope can image at 0.8Å resolution!

Four in-column detectors provide 100X improvement in chemical mapping



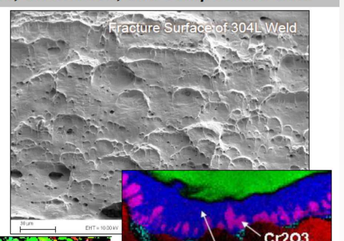


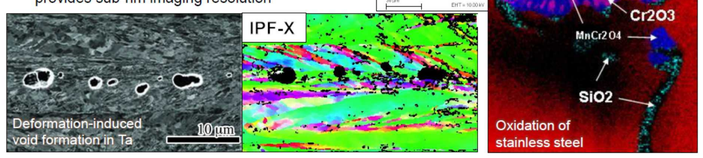
- High resolution chemical imaging on the AC-STEM far exceeds the previous generation of analytical capability.
- Improvement is demonstrated on electrical contact materials.

Materials Analysis of Bulk Samples

Scanning Electron Microscopes (SEM) allow for imaging of features and topography alongside collection of phase, orientation, and composition data

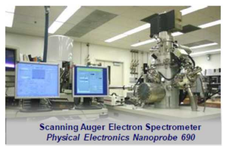
- Electron Backscattered Diffraction (EBSD) and Transmission Kikuchi Diffraction (TKD) give phase and orientation maps down to 2nm steps
- Electron Dispersive Spectroscopy (EDS) and Wavelength Dispersive Spectroscopy (WDS) provide quantitative composition analysis
- Backscattered Electron (BSE) and Secondary Electron (SE) Detectors provide contrast of surface features
- "Extreme High Resolution" Magellan provides sub-nm imaging resolution





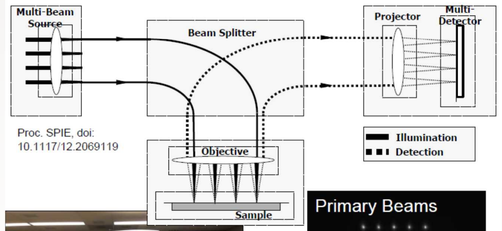
Surface Analysis Suite

Quantitative composition, oxidation state, trace level detection, electronic information, and topography. Total periodic table analysis at nm-Å depths!



Large Area, High Res: mSEM

Multi-Beam SEM is designed for needle-in-a-hay-stack problems High throughput, 1.22 GPixels/s, over mm areas at 4 nm resolution!



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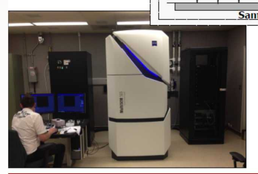

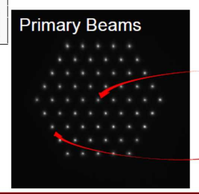

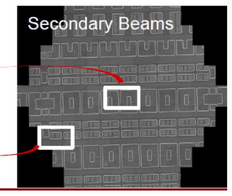

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