# Electron Microscopy of Materials: 1. Instrumentation

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## Introduction

Electron microscopy methods are an important part of materials research and development in the Chemical Engineering Division (CMT). With ever increasing demands on materials performance, it is essential to relate microstructure and composition to materials properties. Scanning and transmission electron microscopy (SEM and TEM) methods allow for the observation and characterization of materials at a level of resolution that meets these demands. This display demonstrates the application of SEM/TEM methods in solving problems of current scientific and technological importance at CMT.

## Capabilities

**SCANNING ELECTRON MICROSCOPY**

- **Secondary Electron Imaging** for sample topography
- **Backscattered Electron Imaging** for atomic number contrast
- **Energy Dispersive Spectroscopy** for elemental analysis

**TRANSMISSION ELECTRON MICROSCOPY**

TEM analysis has made significant contributions in support of programs throughout CMT. The TEM is one of the division's most powerful research tools and also has the distinction of being one of the few radiological TEMs in the country.

- **Brightfield/Darkfield Imaging** for conventional imaging (brightfield) or imaging from selected diffracted beams (darkfield)
- **Electron Diffraction** for phase/structure determination and orientation relationships
- **Energy Dispersive Spectroscopy for** elemental analysis
- **Electron Energy Loss Spectroscopy** for elemental composition and atomic bonding state. Greater sensitivity than EDS and better detection of light elements.
- **Specialized sample preparation techniques:**
  - Custom made substrates (e.g. hydrophilic substrates for nanoparticle dispersions)
  - Novel sample preparation techniques for ultramicrotomy
  - Micromanipulation methods for handling small radioactive samples

## ACL Electron Microscopy Services

- **2 SEM USER FACILITIES (training provided)**
- **ELECTRON MICROSCOPY ANALYSIS including sample preparation, interpretation and documentation.**

## Instrumentation

### Scanning Electron Microscopes

**Topcon ABT-60**

Resolution: 4 nm ( 30kV, working distance: 3mm, secondary electron image)

Accelerating Voltages 0.5 to 3kV (100V steps), 3 to 30 kV (1kV steps)

EDS System: ThermoNORAN Vantage with Norvar window for light element analysis. Resolution: 138 eV

Capabilities:

- X-Ray Mapping
- Line Scan
- Spot Analysis
- Digital Imaging

**JEOL 6400**

Resolution: 3.5 nm (35kV, working distance: 8mm, secondary electron image)

Backscattered resolution: 10.0 nm attainable (working distance: 8 mm, 35kV)

EDS System: ThermoNORAN Vantage with Norvar window for light element analysis. Resolution: 148 eV

Capabilities:

- X-ray Mapping
- Line Scans
- Spot Analysis
- Digital Imaging

### Transmission Electron Microscope

**JEOL 2000FX**

Resolution: Point-to-point: 0.28 nm
Line image: 0.14 nm
Accelerating Voltage: 80 to 200
Minimum step: 50V
EDS System: iXRF
EELS System: Gatan

**Ultramicrotome Ultracut UTC**

Uniform section thickness down to ~15 nm

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# Electron Microscopy of Materials: 2. Applications

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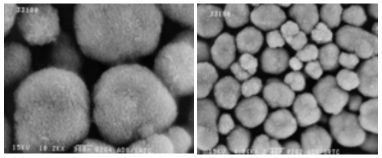
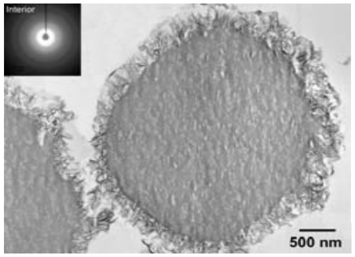
## Applications

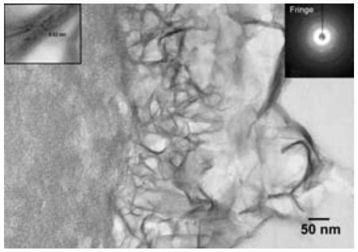
### Monosodium Titanate Particles for Sorption of Actinides [1]



SEM images of monosodium titanate particles (secondary electron image).

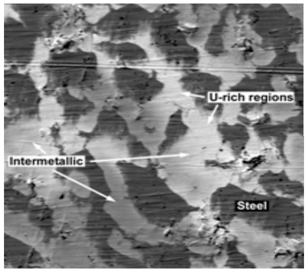



TEM brightfield image showing cross section through two particles. Electron diffraction pattern (inset) of the particle interior shows that it is amorphous or nanocrystalline.

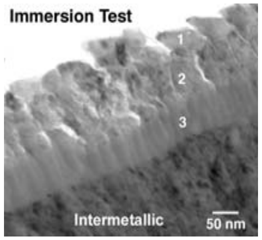



TEM brightfield image of particle exterior. Inset (upper-right) shows basal spacing of outer fringed microstructure.

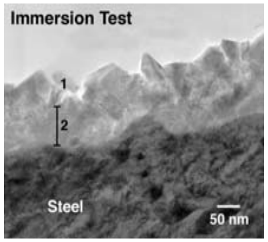
### Metallic Nuclear Waste Form from Electrometallurgical Processing of EBR-II Spent Fuel [2]



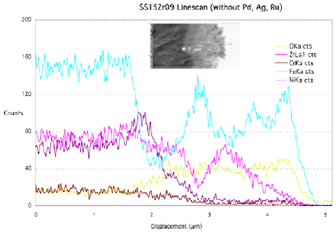

SEM image of polished bulk metal waste form showing a eutectic microstructure consisting of steel and intermetallic phases. (Backscattered electron image formed by atomic number contrast.)




TEM brightfield image of corroded sample showing cross section through the surface and underlying intermetallic phase. Image shows three distinct surface layers formed during corrosion.

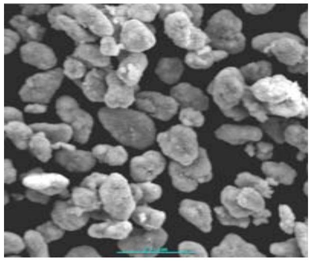



TEM brightfield image of corroded sample showing cross section through the surface and underlying steel phase. Image shows iron oxide bi-layer formed during corrosion.

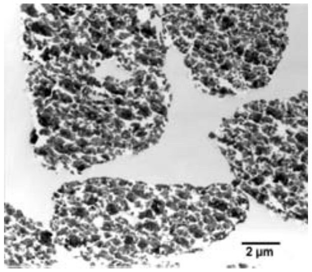



EDS Linescan showing elemental distribution along line of analysis (inset). Analysis started in the bulk intermetallic and proceeded through the surface alteration layers.

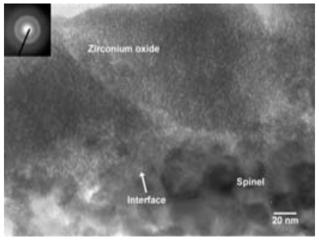
### $ZrO_2$-Coated $LiMn_2O_4$ Electrode Particles for Li-Ion Batteries [3]



SEM image of particles (secondary electron image).




TEM brightfield image showing cross section through several particles.




TEM brightfield image showing enlargement of particle surface. Electron diffraction pattern (inset) shows that the thin Zr-oxide particle coating is amorphous.

NOTE: ALL TEM SAMPLES PRODUCED BY ULTRAMICROTOMY. SAMPLE SECTION THICKNESS ~50NM

[1] This study was performed in connection with work done under Contract Number DE-AC09-96SR18500 for the U.S Department of Energy. [2] This work was supported by the U.S. Department of Energy Office of Nuclear Energy, Science and Technology. [3] This work was supported by the U.S. Department of Energy, Office of Science, Basic Energy Sciences, and FreedomCAR and Vehicle Technologies Program.

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