MSE 262
Materials Characterization Techniques

2 Credit Hours

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Introduction

• Limitation of the TEM

• Unless the specimen is made very thin, electrons are strongly scattered within the specimen, or even absorbed rather than transmitted.

• This constraint has provided the incentive to develop electron microscopes that are capable of examining relatively thick specimens.

What is SEM?

• SEM is a type of electron microscope that images the sample surface by scanning it with a high-energy beam of electrons.

• SEM uses electrons rather than light to form an image.

• The electrons interact with the atoms in the sample producing information such as sample’s surface topology, composition and other properties such as electrical conductivity.

• A scanning electron microscope, like the TEM, consists of
  • Electron optical column
  • A vacuum system
  • Electronics
Characteristic Information

- **Topography** – The surface features of an object or "how it looks", its texture.
- **Morphology** – The shape and size of the particles making up the object.
- **Composition** – The elements and compounds that the object is composed of and the relative amounts of them.
- **Crystallography** – How the atoms are arranged in the object.

Direct relation between these information and materials properties and performance.

Identification of Fracture Mode

SEM micrographs of fractured surface of two BaTiO$_3$ samples.

Important Microstructural Features

- **Grain size**: from < mm to the cm regime
- **Grain shapes**
- **Precipitate size**: mostly in the mm regime
- **Volume fractions and distributions of various phases**
- **Defects such as cracks and voids**: < mm to the cm regime

Optical Microscope vs SEM

OM - 2D

SEM - 3D

Depth of Field

- The distance between the nearest and farthest objects in a scene that appear acceptably sharp in an image.

<table>
<thead>
<tr>
<th>Magnification</th>
<th>Depth of Field</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>OM</td>
</tr>
<tr>
<td>10</td>
<td>60 µm</td>
</tr>
<tr>
<td>100</td>
<td>8 µm</td>
</tr>
<tr>
<td>1000</td>
<td>0.2 µm</td>
</tr>
<tr>
<td>10000</td>
<td>--</td>
</tr>
</tbody>
</table>

- The SEM has a large depth of field, which allows a large amount of the sample to be in focus at one time and produces an image that is a good representation of the three-dimensional sample.

Optical Microscopy vs. SEM

**OM**

Small depth of field
Low resolution

**SEM**

Large depth of field
High resolution
Advantages of SEM over OM

<table>
<thead>
<tr>
<th>Mag</th>
<th>Resolution</th>
</tr>
</thead>
<tbody>
<tr>
<td>OM: 4x – 1000x</td>
<td>~ 0.2 mm</td>
</tr>
<tr>
<td>SEM:10x – 500Kx</td>
<td>1 – 10 nm</td>
</tr>
</tbody>
</table>

✓ The SEM produces images of high resolution, which means that closely features can be examined at a high magnification.

The combination of higher magnification, larger depth of field, greater resolution and compositional and crystallographic information makes the SEM one of the most heavily used instruments in research areas and industries, especially in semiconductor industry.

Basic Principles of SEM

Use a filament to get electrons, magnets to move them around, and a detector to act like a camera to produce an image.

How the SEM Works

Interaction with specimen

- Secondary electrons
  - topography
- Back scatter electrons
  - compositional
- X-rays
  - chemistry

Electron-Specimen Interactions

SEM Setup

Electron/Specimen Interactions

When the electron beam strikes the sample, both photon and electron signals are emitted.
**SE versus BSE**

- SE images show **morphology and topography** of a sample.
  - The more the number of electrons reaching the detector, the brighter the image is.
- SE are produced by **inelastic interaction** of electron beam with electrons in the atom rather than the nucleus. There is energy loss.

![SE and BSE images](image)

**BSE**

Ti wires wrapped around a thicker Ni wire. Ni has a greater atomic number and therefore brighter in the BSE image.

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**BSE images show** **difference in composition** or difference in atomic number in a sample.

- The higher the atomic numbers of the atom, the more backscattered electrons are bounced back, making the image brighter for larger atoms.
- BSE are produced by the **elastic interaction** of the electron beam with nuclei of atoms in the specimen. No energy is lost.

![BSE images](image)

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**The Microscope Column**

- Electron gun
- First condenser lens
- Second condenser lens
- Deflection coils
- Backscatter electron detector
- Objective lens
- X-ray detector
- Vacuum pump

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**Sample Preparation**

- In contrast to TEM or BF imaging in OM (sample thicknesses => 10 nm–1 μm), sample depths for SEM is 10–50 mm range.
- Samples must be of appropriate size to fit into chamber.
- For conventional imaging, specimens must be electrically conductive.
- Non-conductive specimens tend to charge due to presence of electrons => coating.
  - Sputter coating with C, Cr, or Au-Pd
  - Carbon tape, carbon paint
- Charging leads to
  - Deflection of scattered electrons
  - Increased emission of scattered electrons in cracks
  - Periodic scattered bursts and beam deflection

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**Requirement for Sample Preparation**

1. Remove all water, solvents, or other materials that could vaporize while in the vacuum.
2. Firmly mount all the samples.
3. Non-metallic samples like ceramics, should be coated so they are electrically conductive.
   Metallic samples can be placed directly into the SEM.

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**SEM images of amorphous carbon nanofibers (nonconductive)**

(a) as-formed nanofibers, without a gold coating and (b) after sputtering a thin conductive gold coating on the surface.
### Comparing OM and SEM

<table>
<thead>
<tr>
<th>Characteristics</th>
<th>SEM</th>
<th>OM</th>
</tr>
</thead>
<tbody>
<tr>
<td>Illumination</td>
<td>Electron beam</td>
<td>Light beam</td>
</tr>
<tr>
<td>Lens</td>
<td>Electrostatic lens for de-magnification crossover and electro-magnification lens for magnification</td>
<td>Optical lens for magnification</td>
</tr>
<tr>
<td>Resolution</td>
<td>High</td>
<td>Low</td>
</tr>
<tr>
<td>DOF</td>
<td>High</td>
<td>Low</td>
</tr>
<tr>
<td>Magnification</td>
<td>10x – 500Kx</td>
<td>4x – 1000x</td>
</tr>
<tr>
<td>Focusing</td>
<td>electrical</td>
<td>mechanical</td>
</tr>
<tr>
<td>Obtainable images/data</td>
<td>several</td>
<td>Transmitted and reflected</td>
</tr>
<tr>
<td>Contrast</td>
<td>Sharp and chemical property controlled</td>
<td>Color/brightness controlled</td>
</tr>
</tbody>
</table>

### Energy Dispersive X-ray spectroscopy

- Energy dispersive X-ray analysis (EDXA) or Energy dispersive X-ray microanalysis (EDXMA)
- This is an analytical technique used for the elemental analysis or chemical characterization of a sample
- This is derived from the characteristic x-ray
- Each element has a unique atomic structure allowing unique set of peaks on its X-Ray emission spectrum
  - For that purpose, we need a signal that is highly Z-specific
- Auger electron spectroscopy (AES)

### Energy Dispersive X-ray spectroscopy

- EDS detectors on SEM's cannot detect very light elements (H, He, and Li)
  - many instruments cannot detect elements with atomic numbers less than 11 (Na)
- Solid state x-ray detector: very fast and easy to utilize, have relatively poor energy resolution and sensitivity to elements present in low abundances
- Wavelength dispersive x-ray detectors (WDS)
- Electron probe microanalyzers (EPMA)