1.0 Introduction and History

- Electron microscopes are scientific instruments that use a beam of energetic electrons to examine objects on a very fine scale.

- Electron microscopes were developed due to the limitations of Light Microscopes which are limited by the physics of light.

- In the early 1930's this theoretical limit had been reached and there was a scientific desire to see the fine details of the interior structures of organic cells (nucleus, mitochondria...etc.).

- This required 10,000x plus magnification which was not possible using current optical microscopes.
Principal features of an optical microscope, a transmission electron microscope and a scanning electron microscope, drawn to emphasize the similarities of overall design.

University of Tennessee, Dept. of Materials Science and Engineering

Dates

- The transmission electron microscope (TEM) was the first type of Electron Microscope to be developed and is patterned exactly on the light transmission microscope except that a focused beam of electrons is used instead of light to "see through" the specimen. It was developed by Max Knoll and Ernst Ruska in Germany in 1931.

- The first scanning electron microscope (SEM) debuted in 1938 (Von Ardenne) with the first commercial instruments around 1965. Its late development was due to the electronics involved in "scanning" the beam of electrons across the sample.
1.1 Characteristic Information: SEM

Topography
The surface features of an object or "how it looks", its texture; direct relation between these features and materials properties.

Morphology
The shape and size of the particles making up the object; direct relation between these structures and materials properties.

Composition
The elements and compounds that the object is composed of and the relative amounts of them; direct relationship between composition and materials properties.

Crystallographic Information
How the atoms are arranged in the object; direct relation between these arrangements and material properties.

e.g. Identification of Fracture Mode

SEM micrographs of fractured surface of two BaTiO$_3$ samples.
How Fine can You See?

- Can you see a sugar cube? The thickness of a sewing needle? The thickness of a piece of paper? ... 

- The resolution of human eyes is of the order of 0.1 mm, \(100\mu m \approx 4\) mils.

- However, something vital to human beings are of sizes smaller than 0.1mm, e.g. our cells, bacteria, microstructural details of materials, etc.
Microstructural Features which Concern Us

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

Scale and Microscopy Techniques

- XRD, TEM, SEM, OM
- Crystal Structure, Microstructure, Component
- Structure Determination, Fracture Mechanics
- Atomic microscopic, mesoscopic, macroscopic
- Quality control
- Basic Research, Applied Research

Microstructure ranging from crystal structure to engine components ($Si_3N_4$)
Advantages of Using SEM over OM

<table>
<thead>
<tr>
<th>Mag</th>
<th>Depth of Field</th>
<th>Resolution</th>
</tr>
</thead>
<tbody>
<tr>
<td>OM: 4x – 1400x</td>
<td>0.5mm</td>
<td>~ 0.2mm</td>
</tr>
<tr>
<td>SEM: 10x – 500Kx</td>
<td>30mm</td>
<td>1.5nm</td>
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</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.

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

2.1 Electron-Solid Interactions

When an electron beam strikes a sample, a large number of signals are generated.

We can divide the signals into two broad categories:

a) electron signals, b) photon signals
Specimen Interaction Volume

The volume inside the specimen in which interactions occur while interacting with an electron beam. This volume depends on the following factors:

- Atomic number of the material being examined; higher atomic number materials absorb or stop more electrons, smaller interaction volume.

- Accelerating voltage: higher voltages penetrate farther into the sample and generate a larger interaction volume.

- Angle of incidence for the electron beam; the greater the angle (further from normal) the smaller the interaction volume.

Below is an example of a typical interaction volume for:

- Specimen with atomic number 28, 20 kV
- 0° degrees tilt, incident beam is normal to specimen surface

noting the approximate maximum sampling depths for the

- Electron Beam
- Augers 10Å to 30Å
- Backscattered <1-2µm
- Range of L X-rays
- Range of K X-rays
- Range of Electron Penetration

5 µm
**Beam Interaction Simulations**

Theoretical SrS density: 3.7g/cm³, Long. range: 1062 Å, Long. straggle: 384 Å

- **15 kV**
  - 2 µm
  - 5000 Å

- **10 kV**
  - 1 µm

- **5 kV**
  - 2 µm

- **2 kV**
  - 0.5 µm

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**Electron Detectors and Sample Stage**

- **EDS Detector**
- **Secondary Detector**
- **Backscatter Detector**

**Objective lens**

**Sample stage**

**Example**
2.2 Electromagnetic Lenses

Lorentz force equation: \( F = q_0 v \times B \)

Nonaxial electrons will experience a force both down the axis and one radial to it. Only electrons traveling down the axis feel equal radial forces from all sides of the lens. The unequal force felt by the off-axis electrons causes spiraling about the optic axis.

after "Electron Beam Analysis of Materials", M.H. Loretto

Electromagnetic Lenses

The Condenser Lens

- For example with a thermionic gun, the diameter of the first cross-over point \( \sim 20-50 \mu m \).

- If we want to focus the beam to a size < 10 nm on the specimen surface, the magnification should be \( \sim 1/5000 \), which is not easily attained with one lens (say, the objective lens) only.

- Therefore, condenser lenses are added to demagnify the cross-over points.
**Electromagnetic Lenses**  
**The Objective Lens**

- The objective lens controls the final focus of the electron beam by changing the magnetic field strength.

- The cross-over image is finally demagnified to a ~ 10nm beam spot which carries a beam current of approximately $10^{-9} - 10^{-13}$ A.

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**Electromagnetic Lenses**  
**The Objective Lens - Focusing**

- By changing the current in the objective lens, the magnetic field strength changes and therefore the focal length of the objective lens is changed.

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Electromagnetic Lenses
The Objective Lens - Stigmator

- The objective lens is machined to very high precision and the magnetic field pattern is very carefully designed.

- However, the precision attainable by machining cannot match that required for controlling a beam with a 10 nm diameter.

- The stigmator, which consist of two pairs of pole-pieces arranged in the X and Y directions, is added to correct the minor imperfections in the objective lens.

Electromagnetic Lenses
The Objective Lens - Aperture

- Since the electrons coming from the electron gun have spread in kinetic energies and directions of movement, they may not be focused to the same plane to form a sharp spot.

- By inserting an aperture, the stray electrons are blocked and the remaining narrow beam will come to a narrow “Disc of Least Confusion”
Electron vs. Optical Lenses

- e⁻'s don’t actually touch the lens
  - No definite interface
- e⁻’s rotate in the magnetic field
- e⁻’s repel each other
- $f \propto H \propto I$
  - Focus and magnification controlled electronically
  - No physical movements
- e⁻ lenses can only be positive elements (converging)
- Can’t correct e⁻ lens aberrations like you can with compound optical lenses
- e⁻ lenses always operate at small apertures

H = MFS

2.3 Breakdown of an Electron Microscope

In simplest terms, an SEM is really nothing more than a television. We use a filament to get electrons, magnets to move them around, and a detector acts like a camera to produce an image.
1) The "Virtual Source" at the top represents the electron gun, producing a stream of monochromatic electrons.

2) The stream is condensed by the first condenser lens (usually controlled by the "coarse probe current knob"). This lens is used to both form the beam and limit the amount of current in the beam. It works in conjunction with the condenser aperture to eliminate the high-angle electrons from the beam.

3) The beam is then constricted by the condenser aperture (usually not user selectable), eliminating some high-angle electrons.

4) The second condenser lens forms the electrons into a thin, tight, coherent beam and is usually controlled by the "fine probe current knob".

5) A user selectable objective aperture further eliminates high-angle electrons from the beam.
6) A set of coils then "scan" or "sweep" the beam in a grid fashion (like a television), dwelling on points for a period of time determined by the scan speed (usually in the microsecond range).

7) The final lens, the objective, focuses the scanning beam onto the part of the specimen desired.

8) When the beam strikes the sample (and dwells for a few microseconds) interactions occur inside the sample and are detected with various instruments.

9) Before the beam moves to its next dwell point these instruments count the number of e⁻ interactions and display a pixel on a CRT whose intensity is determined by this number (the more reactions the brighter the pixel).

10) This process is repeated until the grid scan is finished and then repeated, the entire pattern can be scanned 30 times/sec.
Summary of Electron Microscope Components

1. Electron optical column consists of:
   – electron source to produce electrons
   – magnetic lenses to de-magnify the beam
   – magnetic coils to control and modify the beam
   – apertures to define the beam, prevent electron spray, etc.

2. Vacuum systems consists of:
   – chamber which “holds” vacuum, pumps to produce vacuum
   – valves to control vacuum, gauges to monitor vacuum

3. Signal Detection & Display consists of:
   – detectors which collect the signal
   – electronics which produce an image from the signal
**Electron Beam Source**

- Single Crystal LaB₆
- Heating Cup
- Heating Wire (W or Re)
- Support Posts
- Lanthanum Hexaboride Single Crystal Filament

**Field Emission Gun**

- The tip of a tungsten needle is made very sharp (radius < 0.1 µm)
- The electric field at the tip is very strong (> 10⁷ V/cm) due to the sharp point effect
- Electrons are pulled out from the tip by the strong electric field
- Ultra-high vacuum (better than 7.5 x 10⁻⁹ Torr - 6 x 10⁻⁶ Pa) is needed to avoid ion bombardment to the tip from the residual gas.
- Electron probe diameter < 1 nm is possible
2.4 Signal Detection and Display

• If you change the target material, the high and low energy peaks remain (although their intensity may change) while the low intensity peaks change position and are characteristic of the sample.

• The reason we produce this type of profile is because the incident electrons we send into the sample are scattered in different ways.

• There are two broad categories to describe electron scattering:
  – elastic: Backscattered electrons
  – inelastic: Secondary electrons

Electron Detectors

Example
Secondary Electrons

These electrons arise due to inelastic collisions between primary electrons (the beam) and loosely bound electrons of the conduction band (more probable) or tightly bound valence electrons. The energy transferred is sufficient to overcome the work function which binds them to the solid and they are ejected.

The interaction is Coulombic in nature and the ejected electrons typically have $\approx 5 - 10$ eV. 50 eV is an arbitrary cut-off below which they are said to be secondary electrons.

Detection

Remember, secondary electrons are low energy electrons. We can easily collect them by placing a positive voltage (100 - 300V) on the front of our detector. Since this lets us collect a large number of the secondaries (50 - 100%), we produce a “3D” type of image of the sample with a large depth of field.

The type of detector used is called a scintillator / photomultiplier tube.
Detection Sequence

1. Secondary electrons (SE) are accelerated to the front of the detector by a bias voltage of 100 - 500 eV.

2. They are then accelerated to the scintillator by a bias of 6 - 12 keV, (10 KeV is normal).

3. Scintillator is doped plastic or glass covered with a fluorescent material (e.g. Europium). A thin (700Å) layer of Al covers it to prevent light from causing fluorescence. The 10keV potential allows the SE to get through the Al and fluoresce.

4. The light photons travel down the tube (guide) to a photocathode which converts them into electrons

5. The electrons move through the detector, producing more electrons as they strike dinodes. An output electron pulse is then detected.
Backscattered Electrons

Backscattered electrons (BSE) arise due to elastic collisions between the incoming electron and the nucleus of the target atom (i.e. Rutherford scattering). Higher Z, more BSE emitted.

As the name implies, elastic scattering results in little (< 1 eV) or no change in energy of the scattered electrons, although there is a change in momentum (p). Since $p = mv$ and the mass of the electron doesn’t change, the direction of the velocity vector must change. The angle of scattering can range from 0 to 180°.

Detection

Since BSE have high energies, they can’t be pulled in like secondaries. If you placed a potential on a grid to attract them, you would also attract the incident beam!!

The most common detector used is called a surface barrier detector. It sits above the sample, below the objective lens. BSE which strike it are detected.
Detection Sequence

Surface barrier detectors are solid state devices made up of semiconducting materials. A semiconducting material has a filled valence band and an empty conduction band—similar to ceramic materials.

1. When a BSE electron strikes the detector, electrons in the material move from valence to conduction band.
2. The electrons are now free to move in the conduction band or drop back into the valence band.
3. If a potential is applied, the e- and e+ can be separated, collected, and the current measured. The strength of the current is proportional to the number of BSE that hit the detector.

2.5 Operating Parameters

Magnification

An image is obtained by taking the signal from the sample and transferring it to a CRT screen. By decreasing the size of the scanned area (from which we get the signal), magnification is produced.

Magnification is determined by taking the ratio of the lengths of the scans:
Mag. = L/l

<table>
<thead>
<tr>
<th>Mag.</th>
<th>Area</th>
</tr>
</thead>
<tbody>
<tr>
<td>10x</td>
<td>(1cm)$^2$</td>
</tr>
<tr>
<td>100x</td>
<td>(1mm)$^2$</td>
</tr>
<tr>
<td>1Kx</td>
<td>(100 μm)$^2$</td>
</tr>
<tr>
<td>10Kx</td>
<td>(10 μm)$^2$</td>
</tr>
<tr>
<td>100Kx</td>
<td>(1 μm)$^2$</td>
</tr>
<tr>
<td>1Mx</td>
<td>(100 nm)$^2$</td>
</tr>
</tbody>
</table>
Resolution

Resolution is the ability to resolve two closely spaced points. While you may have to be at a high magnification to see small features, resolution is NOT the same as magnification.

One way to improve resolution is by reducing the size of the electron beam that strikes the sample:

\[ d_{\text{min}} = 1.29 C_s^{1/4} \lambda^{3/4} [7.92 (iT/J_c) \times 10^9 + 1]^{3/8} \]

at low current:

\[ d_{\text{min}} = 1.29 C_s^{1/4} \lambda^{3/4} \]

\( J_c \) = current density of the source, \( \lambda \) = electron wavelength
\( C_s \) = spherical aberration, \( i \) = current, \( T \) = temperature,

Resolution

We can also improve the resolution by:

- Increasing the strength of the condenser lens
- Decreasing the size of the objective aperture
- Decreasing the working distance (WD = the distance the sample is from the objective lens)
Depth of Field (I)

The height over which a sample can be clearly focused is called the Depth of Field. The SEM has a large depth of field which produces the images that appear 3-dimensional in nature.

Depth of field is improved by:

• Longer working distance
• Smaller objective apertures
• Lower magnifications

Depth of Field (II)

A beam having a semi-angle of convergence $\alpha$ will converge from a radius $r$ to a focus and diverge again in a vertical distance $D$. For small $\alpha$:

$$D = \frac{2r}{\alpha}$$

In a current high-resolution CRT's (spot size = 0.1 mm = 100 microns), the focusing becomes objectionable when two pixels are fully overlapped, where the pixel size on the specimen is $0.1/M$ mm, where $M$ is the magnification, which gives us the practical expression for the depth of focus/field:

$$\text{DOF} \approx \frac{0.2}{\alpha M} \text{ (mm)}$$
Depth of Field (III)

The angle $\alpha$ is determined by:

$$\alpha = \frac{r}{WD}$$

$r$ = radius of the aperture used, and WD is the working distance of the aperture from the specimen.

<table>
<thead>
<tr>
<th>Table 34.2 Depth of field in $\mu$m with 10-mm working distance</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Magnification</strong></td>
</tr>
<tr>
<td>-------------------</td>
</tr>
<tr>
<td>100</td>
</tr>
<tr>
<td>1000</td>
</tr>
<tr>
<td>10000</td>
</tr>
<tr>
<td>100000</td>
</tr>
</tbody>
</table>

Depth of Field vs. Resolution

Depth of field and resolution have a reciprocal relationship:

- Improving resolution in conventional SEM’s leads to a smaller depth of field

- While increasing depth of field decreases resolution. Useful for each particular sample.
3.0 Instrumentation

JEOL 6700F Ultra High Resolution Scanning Electron Microscope

3.1 Sample Preparation

Sample Coating

Q: Why?
A: Charging:

- Deflection of SE’s
- Increased emission of SE's in cracks
- Periodic SE bursts
- Beam deflection

Solutions:
- Sputter coating with C, Cr, or Au-Pd
- Carbon tape, carbon paint, In foil
Sputtering

Ion Beam Coating

Gatan PECS Model #682
4.0 Artifacts and Examples

- Surface Etching
- Thermal damage
  - Sample temperature can rise up to 40°C
- Coating Film Adhesion: Not sticking, temperature sensitive
- Contamination
- Morphology Modification
- Stigmation
- Focus
- Depth of field
- Voltage
Silica Spheres, 0.5 µm, Hereisus

Native BS Compo Fe Alloy (Fe-Mo-Mn-Cr B)
5.0 Summary

• The basic layout and operation of an SEM.

• The different imaging modes of the SEM.

• How an image is obtained

• Sample preparation

• Materials

• Information: Imaging, composition (EDS, WDS, CL)

• Fabrication with EBL