Scanning Electron Microscopy (SEM)

Do it with electrons!
Structure determines properties

We have discussed crystal structure (x-ray diffraction)

But consider now different level of structure

Microstructure - also affects properties

Grey cast iron - rather brittle

Ductile iron - highly ductile
Microscopy

Structure determines properties

We have discussed crystal structure (x-ray diffraction)

But consider now different level of structure

Microstructure - also affects properties

Cemented WC cutting tool
Microscopy

Structure determines properties

We have discussed crystal structure (x-ray diffraction)

But consider now different level of structure

Microstructure - also affects properties

Ferroelectric domains in BaTiO$_3$
Microscopy

Structure determines properties

We have discussed crystal structure (x-ray diffraction).

But consider now different level of structure.

Microstructure - also can be 'art'.
Electron microscopy

SEM - scanning electron microscopy

tiny electron beam scanned across surface of specimen

backscattered or secondary electrons detected

signal output to synchronized display
Electron microscopy

SEM - scanning electron microscopy

Magnification range 15x to 200,000x

Resolution of 50 Å

Excellent depth of focus

Relatively easy sample prep
SEM - scanning electron microscopy

Electron gun

Don't make x-rays - use electrons directly

Wavelength:

NOT $\lambda = h/c/E$

(massless photons)

$\lambda = h/(2m_{\text{electron}}qV_0)^{1/2}$

(non-relativistic)

$\lambda = h/(2m_{\text{electron}}qV_0 + q^2V_0^2/c^2)^{1/2}$

(relativistic)
SEM - scanning electron microscopy

$$\lambda = \frac{\hbar}{(2m_{\text{electrons}}qV_o + q^2V_o^2/c^2)^{1/2}}$$

$$\lambda = \frac{1.22639}{(V_o + 0.97845 \cdot 10^{-6}V_o^2)^{1/2}}$$

$\lambda$ (nm) & $V_o$ (volts)

10 kV $\rightarrow$ 0.12 Å

100 kV $\rightarrow$ 0.037 Å
SEM - scanning electron microscopy

Diagram showing the components of a scanning electron microscope:
- Gun
- Lens
- Scan coils
- Detector
- Amplifier
- CRT

Scan generator connects to CRT.
SEM - scanning electron microscopy

\[ \lambda = \frac{h}{(2m_{\text{electron}} q V_0 + q^2 V_0^2/c^2)^{1/2}} \]

Effects of increasing voltage in electron gun:

Resolution increased (\( \lambda \) decreased)

Penetration increases

Specimen charging increases (insulators)

Specimen damage increases

Image contrast decreases
SEM - scanning electron microscopy

Field emission electron source:

High electric field at very sharp tip causes electrons to "tunnel"
SEM - scanning electron microscopy

Field emission electron source:

High electric field at very sharp tip causes electrons to "tunnel"
SEM - scanning electron microscopy

Field emission electron source:

High electric field at very sharp tip causes electrons to "tunnel"

- cool tip $\rightarrow$ smaller $\Delta E$ in beam
- improved coherence

- many electrons from small tip $\rightarrow$ finer probe size, higher current densities ($100X >$)

- problems - high vacuum, more $$$, fussy
SEM - scanning electron microscopy

**Lenses**

electrons focused by Lorentz force from electromagnetic field

\[ F = qv \times B \]

effectively same as optical lenses

Lenses are ring-shaped

coils generate magnetic field
electrons pass thru hollow center

lens focal length is continuously variable

apertures control, limit beam
SEM - scanning electron microscopy

Specimen

Conducting -
- little or no preparation
- attach to mounting stub for insertion into instrument
- may need to provide conductive path with Ag paint

Non-conducting -
- usually coat with conductive very thin layer (Au, C, Cr)
Probes used

- **Visible light**
  - Optical microscopy (OM)
- **X-ray**
  - X-ray diffraction (XD)
  - X-ray photo electron spectroscopy (XPS)
- **Neutron**
  - Neutron diffraction (ND)
- **Ion**
  - Secondary ion mass spectrometry (SIMS)
  - Cleaning and thinning samples

- **Electron**
  - Scanning electron microscopy (SEM)
  - Transmission electron microscopy (TEM)
  - Electron holography (EH)
  - Electron diffraction (ED)
  - Electron energy loss spectroscopy (EELS)
  - Energy dispersive x-ray spectroscopy (EDS)
  - Auger electron spectroscopy (AES)
(SEM) and TEM
JEOL 6700F Ultra High Resolution Scanning Electron Microscope
**Kanaya-Okayama Depth Penetration**

**Formula**

\[
R = \frac{0.0276 \ A \ E^{1.67}}{(Z^{0.89} \ \rho)} \ 	ext{µm}
\]

- **R** = Depth Penetration
- **A** = Atomic Weight (g/mole)
- **E** = Beam Energy (KV)
- **Z** = Atomic number
- \( \rho \) = density (g/cm^2)

**The Affect of Accelerating Voltage**

- **30KV**: 3.1 µm
- **15KV**: .99 µm
- **5KV**: .16 µm (100A)
- **1KV**: .01 µm
- **.5KV**: 35 A

*Depth Penetration in Iron*

*(predictions from the KO formula)*
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.
Scanning electron microscopy is used for inspecting topographies of specimens at very high magnifications using a piece of equipment called the scanning electron microscope. SEM magnifications can go to more than 300,000 X but most semiconductor manufacturing applications require magnifications of less than 3,000 X only. SEM inspection is often used in the analysis of die/package cracks and fracture surfaces, bond failures, and physical defects on the die or package surface.

During SEM inspection, a beam of electrons is focused on a spot volume of the specimen, resulting in the transfer of energy to the spot. These bombarding electrons, also referred to as primary electrons, dislodge electrons from the specimen itself. The dislodged electrons, also known as secondary electrons, are attracted and collected by a positively biased grid or detector, and then translated into a signal.

To produce the SEM image, the electron beam is swept across the area being inspected, producing many such signals. These signals are then amplified, analyzed, and translated into images of the topography being inspected. Finally, the image is shown on a CRT.
Scanning Electron Microscopy (SEM)

- The energy of the primary electrons determines the quantity of secondary electrons collected during inspection. The emission of secondary electrons from the specimen increases as the energy of the primary electron beam increases, until a certain limit is reached. Beyond this limit, the collected secondary electrons diminish as the energy of the primary beam is increased, because the primary beam is already activating electrons deep below the surface of the specimen. Electrons coming from such depths usually recombine before reaching the surface for emission.

- Aside from secondary electrons, the primary electron beam results in the emission of backscattered (or reflected) electrons from the specimen. Backscattered electrons possess more energy than secondary electrons, and have a definite direction. As such, they can not be collected by a secondary electron detector, unless the detector is directly in their path of travel. All emissions above 50 eV are considered to be backscattered electrons.
Scanning Electron Microscopy (SEM)

- Backscattered electron imaging is useful in distinguishing one material from another, since the yield of the collected backscattered electrons increases monotonically with the specimen's atomic number. Backscatter imaging can distinguish elements with atomic number differences of at least 3, i.e., materials with atomic number differences of at least 3 would appear with good contrast on the image. For example, inspecting the remaining Au on an Al bond pad after its Au ball bond has lifted off would be easier using backscatter imaging, since the Au islets would stand out from the Al background.

- A SEM may be equipped with an EDX analysis system to enable it to perform compositional analysis on specimens. EDX analysis is useful in identifying materials and contaminants, as well as estimating their relative concentrations on the surface of the specimen.
Comparison of OM, TEM and SEM

Principal features of an optical microscope, a transmission electron microscope and a scanning electron microscope, drawn to emphasize the similarities of overall design.
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.
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.
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.
Scale and Microscopy Techniques

- **XRD, TEM**: Crystal Structure
- **SEM**: Microstructure
- **OM**: Component

**Structure Determination** - **Fracture Mechanics**

**Quality Control**

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>
</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.

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.
Electron-Solid Interactions

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

- Incident $e^-$ Beam
- Backscattered $e^-$
- Cathodoluminescence
- Secondary $e^-$
- Inelastically Scattered $e^-$
- Elastically Scattered $e^-$
- Unscattered $e^-$
- Transmitted Electrons
- Auger $e^-$
- X-rays

We can divide the signals into two broad categories:

a) electron signals, b) photon signals
Scanning Electron Microscope

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.
Scanning Electron Microscope

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.
Scanning Electron Microscope

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\textsuperscript{-} 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.
A Look Inside the Column

- Electron gun
- Gun Alignment Control
- Pneumatic Air Lock Valve
- Condenser Lens
- Objective Aperture
- Scanning Coil
- Objective Lens
- Motorized Stage
- Sample Chamber

Stage

Column
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
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:

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

at low current:

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

\( J_c \) = 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)
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
Ion Beam Coating

Gatan PECS Model #682
BTO and Ag powders written by a laser on Kapton
BTO and Ag powders written by a laser on Kapton
Chemical Analysis with the SEM

- Qualitative analysis
- Quantitative analysis
- Mapping of locations of elements
Figure 3.20. Schematic illustration of the origin of two sources of secondary electron generation in the sample. Incident beam electrons (B) generate secondary electrons ($SE_1$) upon entering the sample. Backscattered electrons (BSE) generate secondary electrons ($SE_2$) while exiting the sample. $\lambda$ is the mean free path for secondary electrons.

$\lambda \approx 1 \text{ nm for metals up to } 10 \text{ nm for insulators}$
...a little more accurately

Shamelessly stolen from
Northern Arizona University
Microprobe Lab
X-ray generation from inner shell transitions

Emission energy is “characteristic” of energy level differences and hence element

SEM electron (e.g. 20 keV) “kicks out” electron from “K-shell”
“Kinds of X-rays”

• “Characteristic X-rays”
  – Characteristic of element from which they are emitted
  – Nominally sharp lines

• Brehmstrahlungen
  – Broad, featureless background
  – Sanity check: must asymptote to zero at Duane-Hunt limit
    • Otherwise: evidence of charging!
X-ray spectrum of sapphire

Characteristic x-rays

Brehmstrahlung (baseline/background)
Fluorescence yield

Figure 6.6. Fluorescence yield $\omega$ as a function of atomic number for electron ionization within the $K$, $L$, and $M$ electron shells.
Fluorescence yield

- Fluorescence yield
  - \( \omega_K \) = K-shell photons emitter per K-shell ionization
  - For each shell: \( \omega_K > \omega_L > \omega_M \)
  - For \( Z < 20 \), the yield drops like a stone!
  - For carbon, \( Z = 6 \), \( \omega_K \approx 0.005 \); for zinc, \( Z = 30 \), \( \omega_K \approx 0.5 \)
  - Quantitation simply by looking at peak heights, or even areas, is a disaster!
Kinds of X-ray analyzers

• Energy dispersive (EDS)
  – Operate just like a solar cell!
  – Charge generated by one X-ray: X-ray energy divided by band gap of silicon...well, not really

• Wavelength dispersive (WDS)
  – Bent LiF crystal acts like a conventional glass lens
  – Degree of deviation in bent crystal depends on X-ray wavelength
X-ray detector "Energy Collection Efficiency"

Glenn F. Knoll,
Radiation Detection and Measurement,
Wiley (1979) p. 503

FIGURE 13-22. The average energy required to form one electron-hole pair ($\epsilon$) versus bandgap energy for a number of semiconductor materials. (From Klein\textsuperscript{46}.)
X-ray detector “Energy Collection Efficiency”

TABLE 13-3. Properties of Semiconductor Materials

<table>
<thead>
<tr>
<th>Material</th>
<th>Z</th>
<th>Band gap (eV)(^a)</th>
<th>Energy per e-h pair (eV)(^a)</th>
<th>Best γ-Ray Energy Resolution (FWHM)(^b)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Si (300 K)</td>
<td>14</td>
<td>1.12</td>
<td>3.61</td>
<td>420 eV @ 100 keV</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.17 @ 77K</td>
<td></td>
<td>920 eV @ 660 keV</td>
</tr>
<tr>
<td>Ge (77 K)</td>
<td>32</td>
<td>0.74</td>
<td>2.98</td>
<td>1300 eV @ 1330 keV</td>
</tr>
<tr>
<td>CdTe (300 K)</td>
<td>48–52</td>
<td>1.47</td>
<td>4.43</td>
<td>3800 eV @ 122 keV</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>7500 eV @ 661 keV</td>
</tr>
<tr>
<td>HgI(_2) (300 K)</td>
<td>80–53</td>
<td>2.13</td>
<td>6.5</td>
<td>850 eV @ 6 keV</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>3500 eV @ 122 keV</td>
</tr>
<tr>
<td>GaAs (300 K)</td>
<td>31–33</td>
<td>1.43</td>
<td>4.2</td>
<td>650 eV @ 60 keV</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>2600 eV @ 122 keV</td>
</tr>
</tbody>
</table>

\(^a\) Data from Bertolini, Cappellani, and Restelli\(^1\)

\(^b\) Glenn F Knoll, Radiation Detection and Measurement, Wiley (1979)

Value increases to 3.8 eV at 77 K

n.b.!!
X-ray analyzer “failure modes”

- X-rays from heavier elements (higher energy) can ionize silicon
  - “Escape peaks” are at energy lower than major peaks by 1.74 keV
  - Some energy of incident X-ray was used to ionize silicon in detector

- Sum peaks
  - Two X-rays enter detector while counting window is open
  - Small peak whose energy is the sum of two lower energy peaks.
Sum peak!

Aluminum at 1.48 keV

Sum peak at 1.98 keV
Spatial Resolution

X-ray Spatial Resolution

- X-ray range is related to, but less than, electron range $R_{K-O}$
  - Electron scattering volume and $R$ increases with $E_o$
  - $R$ defined as depth where electron $E \sim 0$

Monte Carlo calculations presumably done for some intermediate Z element

Stolen from Goldstein’s lecture
Lab Lecture: How to Actually use the EDAX EDS system

• Main software has three tabs:
  • Spectrum shows only the spectrum
    – What is a spectrum?
  • Image
    – Shows SEM image of area being analyzed
    – Shows X-ray spectrum
    – Shows quantitative analysis or histogram of brightness
  • Mapping
    – Allows selection of elements to be mapped
    – Shows spatial distribution of elements in sample
Setting up the NovaNano

- Be sure the CCD camera (quadrant) is deselected/not active
- Lower the EDS detector to 36 mm
- Raise the spot size to 5 or 6
- In the mode setup, choose “EDX mode”
- Boot the Genesis software on the support computer (left monitor)
X-ray Detector Insertion Gauge
• Important tabs on the Genesis window
  – EDS is an attachment to the SEM
    • Both Quanta and NovaNano have EDAX systems
    • Leo has ancient Oxford system.
  – Genesis is EDAX trade name for EDS operating system
Important icons in Genesis

- **Import spectrum**
- **Start/Stop EDS collection**
- **Clear spectrum**
- **Expand/compress Spectrum horizontally**
- **Add text**
Genesis icons continued

- Expand/compress scale vertically
- Home: Restore Horizontal and vertical scales
- Export to Word
Genesis icons continued
Preset options

What kind of time?
Live?
Clock?
RoI?

Determines “Live Time” during which data are collected

“None” is also an option
Dead time

• Kinds of time
  – Live time: time during which detector takes data
  – Dead time: time during which current accumulation window is closed
  – Clock time
    • Sum of live and dead time
    • Actual time we all know and love
Amp time: adjust for dead time of 20 - 40%

1.6 μsec – 102.4 μsec
Microscope control panel and return!

Return to normal operating screen
Peak ID

Halographic Peak Determination: fits spectrum with theoretical sum

Possible matches for energy where cursor is located

Move to list of labeled peaks

Labels from spectrum
Lower tray information

Red while taking data
Black when finished

O Ka

CPS:21265  DT%:23  Lsec:50  Cnts:429  keV:2.650  FS:46637
You’re image here!

Image Tab
Top Center Icons

- Return control to microscope!
- Raster
  - Full Reduced Spot
  - User drawn
Collect Image

Collected Image

Reso: 1024x800
Strips: 25
Conti:

<table>
<thead>
<tr>
<th>Label</th>
<th>Det</th>
<th>Smin</th>
<th>Smax</th>
<th>Reads</th>
</tr>
</thead>
<tbody>
<tr>
<td>SE1</td>
<td>1</td>
<td>0</td>
<td>4095</td>
<td>16</td>
</tr>
<tr>
<td>BSE</td>
<td>1</td>
<td>0</td>
<td>4095</td>
<td>16</td>
</tr>
</tbody>
</table>

Autoscale
Default
Brightness and contrast adjust in counterintuitive direction. Use low resolution and few strips for faster feedback.