Outline

In this section of the course, we combine the discussion of physics of electron matter interactions, operation of SEM, the influence of the interaction volume, and the detector geometry to explain image formation, resolution in the images, and the contrast mechanisms. We end section with a discussion about charging and imaging non-conductive samples.

1) Review of SEM operation
2) Depth of field
3) Interaction volume
4) Secondary and back-scattered electrons
   A. Image Intensity
   B. Influence of interaction volume
   C. In-lens detectors
5) Contrast mechanisms
6) Charging effects
1) Review of SEM operation

- High Energy Electrons *(high spatial resolution!)*
- Condenser lens that define probe size and control current
- Apertures that control convergence angle *(depth of field)*
- Scanning coils above Objective lens raster beam on sample
- Objective lens focus probe on sample
- Detectors surrounding sample collected radiated signals *(In-lens detectors)*

SEM images are formed from the collected of radiated particles in a step by step manner by the sequential scanning of the sample with the electron probe

- **Low magnification**
  - The screen (or recording media) pixel size $d_{\text{screen}}$ 
  - $r \sim d_{\text{screen}}/\text{magnification}$
- **High magnification**
  - Resolution is a function of probe size ($r \sim d_{\text{probe}}$) and size of the interaction volume
1) Review of SEM operation

Incident electrons interaction with the sample produces:

- Secondary electrons SE: topography, low energy ≈ 0-50 eV
- Backscattered electrons BSE: atomic number Z, energy ≈ eV₀
- Auger Electrons: not detected in conventional SEM, surface analysis
- Cathodoluminescence: photons UV, IR, vis
- Absorbed current, electron-holes pairs creation, EBIC
- Plasmons
- Sample heating (phonons)
- Radiation damages: chemical bonding break, atomic displacement (knock-on) damage

2) Depth of Field

CdSe “flowers” that grow in clusters and have tubular-shaped branches

The power of SEM is the ability to observe and measure complex 3-D morphologies with nanometer resolution

High Depth of Field!
2) Depth of field

Influence of working distance and objective aperture on resolution

Increasing working distance will:
- increase depth of focus
- increase probe size and thus decrease resolution.
- increase the effects of stray magnetic fields and thus decrease resolution
- increase aberrations due to the need for a weaker lens to focus

Increasing aperture size will:
- increase current and produced signals – better signal to noise
- increase aberration and thus decrease resolution.
- decrease depth of field

2) Depth of field: Compromise with resolution

Light bulb filament

Resolution

Object height

Magnification

Influence of working distance and objective aperture on resolution

Increasing working distance will:
- increase depth of focus
- increase probe size and thus decrease resolution.
- increase the effects of stray magnetic fields and thus decrease resolution
- increase aberrations due to the need for a weaker lens to focus

Increasing aperture size will:
- increase current and produced signals – better signal to noise
- increase aberration and thus decrease resolution.
- decrease depth of field
2) Depth of field

Depth of field as a function of $d_{\text{probe}}$

- The depth of field is the depth for which the image can be focused.
- The depth of field increases when the convergence angle of the probe ($\alpha$) decreases.

- Increase the working distance
- Reduce objective aperture size

$$h_{\text{depth of field}} = \max \left\{ \frac{2d_{\lambda}}{\alpha}, \frac{\text{pixel size}}{\text{Mag}}, \frac{1}{\alpha} \right\}$$

Effect of working distance (WD) and aperture on the depth of field
2) Depth of field

Examples

Another example of the effect of the objective aperture diameter
2) Depth of field

Measuring depth of field: stereoscopy

\[ Z_L = \frac{(X_L \cos \alpha - X_R)}{\sin \alpha} = \frac{3.32 \mu m \cos 6° - 0.33 \mu m}{\sin 6°} = 28.4 \mu m \]

\[ Z_R = \frac{(X_L - X_R \cos \alpha)}{\sin \alpha} = \frac{3.32 \mu m - 0.33 \mu m \cos 6°}{\sin 6°} = 28.6 \mu m \]

\[ p = (X_L - X_R), \quad h = p/2 \sin \left( \frac{\alpha}{2} \right) = 3 \mu m/2 \sin(3°) = 28.7 \mu m \]

Distance between points: \( \sqrt{Z_L^2 + X_L^2} = 28.6 \mu m \)

\[ EDS(\text{angle}) = \tan^{-1} \left( \frac{Z_L}{X_L} \right) = 83.3° \]

Some Modern SEMs (Merlin) do not have a variable objective aperture so it is the column mode which changes the depth of field
4) Influence of the interaction volume

1 kV – surface features are resolved with high spatial resolution
30 kV – buried interfaces are visible though surface features are less resolved

- 30 kV imaging has higher spatial resolution but larger interaction volume, increasing edge effects that mask the fine features of the surfaces.
- 1 kV imaging has a smaller interaction volume and thus better for observing fine surface features.
- Sample Geometry is an important factor in choosing the accelerating voltage.

3) Interaction Volume

Number/Energy of backscattered electrons by Monte-Carlo simulations

W

1 kV
BSE 41%

3 kV
BSE 43%

30 kV
BSE 52%

C

1 kV
BSE 10%

3 kV
BSE 8%

30 kV
BSE 5%
3) Interaction Volume

Monte-Carlo simulations

1. Electron Flight Simulator (``Small World / D. Joy)
   - old… DOS !!!!
   - http://www.small-world.net

2. Single Scattering Monte Carlo Simulation (Freeware)
   - "Monte Carlo Simulation" Mc_w95.zip
   - by Kimio KANDA
   - http://www.nsknet.or.jp/~kana/soft/sfmenu.html

3. CASINO (Freeware)
   - " monte CArlo Simulation of electroN trajectory in sOlids "
   - by P. Hovongton and D. Drouin

Penetration and back-scattering vs elements (Z)

\[ V_{acc} = 20kV = cte \]

Depth of electron penetration vs Z and yield of backscatter electrons BSE (Monte-Carlo simulation):
3) Interaction Volume

Penetration and back-scattering vs elements (Z)

\[ V_{\text{acc}} = 5 \, \text{kV} = \text{cte} \]

Depth of electron penetration vs Z and yield of backscattered electrons BSE (Monte-Carlo simulation):

\[ V_{\text{acc}} = 1 \, \text{kV} = \text{cte} \]

Depth of electron penetration vs Z and yield of backscattered electrons BSE (Monte-Carlo simulation):
3) Interaction Volume

Penetration and backscattering vs energy ($E$)

$Z = \text{cte}$

Depth of electron penetration in Cu vs energy $E_0$ and yield of backscattered electrons BSE (Monte-Carlo simulation):

Cu 20 keV

Cu 5 keV

Cu 1 keV

Interaction volume versus $E_0$

Penetration depth in Cu as a function of incident energy $E_0$ and proportion of BSE (Monte-Carlo simulation)

$Z = \text{cte}$
4) Secondary Electrons and Backscattered Electrons

Wires with Nb<sub>3</sub>Sn core surrounded by a Cu matrix

SE: Intensity depends on inclination, i.e., surface topography and roughness
BSE: Intensity depends on atomic number, i.e., higher Z gives higher intensity

Yield for SE and BSE emission per incident electron vs atomic number Z

BSE: chemical contrast for all the elements
(sensitivity \( \approx DZ=0.5 \))
A fast way to phase mapping

SE: low or no chemical contrast but for light elements the topographical contrast will dominate on rough surfaces

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4) Secondary Electrons and Backscattered Electrons

Toner particle (penetration in light material)

4) Secondary Electrons and Backscattered Electrons

Dust on WC (different Z materials)
4) Secondary Electrons and Backscattered: *Interaction Volume*

"true" secondary electrons $SE_1$ and "converted BSE" secondaries $SE_2 + SE_3$

The SE signal always contain a high resolution part (SE1 from the probe) and a combined average (low resolution) part from SE2+SE3!

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Al$_x$Ga$_{1-x}$As/GaAs "quantum wire" (2-D quantum well)

SE mode image on a cleaved surface. The SE$_2$ (BSE chemical) contrast dominates this image in absence of topographical contrast

(by courtesy of Dr. K. Leifer, IPEQ/EPFL)
4) Secondary Electrons and Backscattered: 

*Interaction Volume*

True secondary electrons **SE1** and "converted BSE" secondaries **SE2+SE3**

Various SE types from
- SE1: incident probe
- SE2: BSE leaving the sample
- SE3: BSE hitting the surroundings

although this signal is gathered around the probe, its intensity is only attributed to the pixel corresponding to the actual probe position

Relative contribution of SE1 and SE2 (+SE3) vs primary energy

The total intensity (green and brown) is attributed to the (x,y) pixel, here at 0 nm on this 1-D model

(adapted from D.C. Joy 
Hitachi News 16 1989)

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4) Secondary Electrons and Backscattered Electrons

In-lens Backscattered Electron Detector EsB

In-lens Secondary Electron Detector

Everhart Thornley Secondary Electron Detector

Solid State Backscattered Electron Detector

Variable Pressure Secondary Electron Detector

Sample

In-lens SE detector

Everhart Thornley detector
4) Secondary Electrons and Backscattered Electrons

Energy spectrum of electrons leaving the sample

 BSD  ET-SE
 In lens-SE In lens-EsB
4) Secondary Electrons and Backscattered Electrons

Shorter work distance improves spatial resolution of fine topographical features (But at lower depth of field!)

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4) Secondary Electrons and Backscattered Electrons

Voltage has 2 important effects on image contrast
1) Size of interaction volume
2) Detector sensitivity/energy selectivity

Contrast reversal in BSE mode at low accelerating voltage

Fig. 4.1. Dependence of the backscattering coefficient \( \eta \) at normal incidence \( (\phi=0) \) on atomic number \( Z \) for different electron energies.

from L. Reimer, Image formation in low-voltage SEM
4) Secondary Electrons and Backscattered Electrons

**low kV imaging**

<table>
<thead>
<tr>
<th>SE image</th>
<th>EsB image</th>
<th>SE image</th>
<th>EsB image</th>
</tr>
</thead>
<tbody>
<tr>
<td><img src="image1.png" alt="Image 1" /></td>
<td><img src="image2.png" alt="Image 2" /></td>
<td><img src="image3.png" alt="Image 3" /></td>
<td><img src="image4.png" alt="Image 4" /></td>
</tr>
</tbody>
</table>

10 kV  
0.5 kV

5) Contrast mechanisms

**Size and edge effects**

![Image 5](image5.png)
5) Contrast mechanisms

- Secondary electron escape for near surface and thus give information about topography
- If a sample is titled, the interaction volume is tilted and closer to the surface. Thus, more SE escape from the surface, giving higher signals
- The same principle is true for rough surfaces – Sloped surfaces and edges have an interaction volume that is effectively tilted and have higher SE signals

1. Rough Surfaces have high SE image contrast
2. Titling can improve SE image contrast

Topographical contrast in SE mode: Effect of sloped surfaces

penetration depth ("range") >> SE escape length

Relative yield of SE vs angle of incidence on the sample surface

(adapted from D.C. Joy Hitachi News 16 1989)

\[ I(\theta) = I_0 \delta(\theta) \equiv \frac{I(0)}{\cos \theta} \]

Relative yield of SE vs angle of incidence on the sample surface
5) Contrast mechanisms

Topographical contrast at low energy
Effect of the incidence angle

But at low energy....

Range

yield will be nearly independent of tilt

(adapted from D.C. Joy Hitachi News 16 1989)

5) Contrast mechanisms

Size and edge effects

5) Contrast mechanisms

Size and edge effects

Do not forget, in SEM:
The signal is displayed at the
probe position, not at the actual
SE production position!!!

Fig. 6.6. SE signal intensity across spheres with diameters larger and smaller than the electron range \( R \)
and increase of the SE signal at an edge caused by diffusion contrast.
5) Contrast mechanisms: BSE topographical contrast

BSE topographical contrast is not negligible! Chemical contrast is better observed only on flat, polished surfaces

5) Contrast mechanisms

Comparison of SE and BSE contrast modes

If you look down to the column, the "light" seems to come from the Everhardt-Thornley detector.
5) Contrast Mechanisms

Everhart Thornley detector

In-lens SE detector

SE2 Detector located on the lower right

In-lens SE Detector located directly above and centered

6) Contrast Mechanisms

The bias on the Faraday cage can be adjusted from +400 V to -250 V
5) Contrast Mechanisms

Effect of collector bias voltage on the SE image contrast

-250V  -100V  0V  400V

Contrast in image suggest the object in the center has a “stepped” pyramid morphology

Contrast mechanisms
5) Contrast mechanisms

Rotated by 180 degrees, and now it looks like an etched pit on the surface

Detector position?  Image rotated 180 degrees  Detector position?

pyramid?  ..... OR .....  etch-pit?
5) Contrast mechanisms

Influence of voltages on the resolution and contrast mechanisms

- Low voltage better resolution and imaging of fine surface features low Z materials (smaller interaction volume and less SE2 signal)
- High voltage better spatial resolution for isolated particles
5) Contrast mechanisms

Effect of the accelerating voltage on penetration and SE signal

20 kV:
- Strong penetration, SE3 is a much larger signal than SE1/SE2.
- It reveals the copper grid under the C film via the electron backscattering, but the structure of the film itself is hidden.

2 kV:
- Low penetration, only a few electrons reach the copper grid and most of the SE3 are produced in the C film together with SE1/SE2.
- The C film and its defects become visible.


5) Contrast mechanisms

Enhancement topograpical contrast at low voltage: less delocalization by SE2.
5) Contrast mechanisms

Change in SE topological contrast with the voltage

(from L. Reimer, Image formation in the low-voltage SEM)

Interaction volume size is very important for contrast mechanisms. Choice of HT voltage is important for proper visualization of surface features
5) Contrast mechanisms

Higher voltage gives more edge effects and increased topographical contrast in BSE images. However, it gives lower resolution of fine surface features (larger interaction volume).

20 KV

5 KV
6) Charging effects

Fiberglass on epoxy

(by courtesy of B. Senior CIME/EPFL)

Charging-up of an insulating particle of dust

Negative charges left on the particle create an electric field that repels the SE toward the substrate around the dust

(adapted from L. Reimer Scanning Electron Microscopy)
6) Charging effects

Extreme charging-up: electrons are reflected by the sample and hit the microscope sample chamber!!!

In-lens SE detector

In-lens detectors collect SE1 electrons, those that emanate from the near surface, which are more sensitive to surface charging

Everhart Thornley detector
6) Charging effects

Total yield for electron emission (SE + BSE) on insulators

$E_1$ and $E_2$ are critical energies for which 1 electron leaves the surface for each incident electron: *neutrality*

$eV_{\text{acc}} = E_1$ is unstable
$eV_{\text{acc}} = E_2$ is stable

when $eV_{\text{acc}} < E_2$ positively charged
when $eV_{\text{acc}} > E_2$ negatively charged
when $eV_{\text{acc}} = E_2$ charging disappears

Caution: this extremely simple model is not quantitative for insulators because charge implantation and removal depends on the scanning speed and precise sample geometry.
6) Charging effects

Some values of the neutrality $E_2$ energy

$E_2$: upper neutrality energy

$E_m$: maximum emission energy

$\delta_m$: maximum yield at $E_m$

adapted from:
E. Plies, Advances in Optical and Electron Microscopy, 13 (1994) p 226

<table>
<thead>
<tr>
<th>Material</th>
<th>$\delta_m$</th>
<th>$E_m$ (keV)</th>
<th>$E_2$ (keV)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Carbon</td>
<td>0.94–1.0</td>
<td>0.3–0.55</td>
<td>0.65</td>
</tr>
<tr>
<td>Aluminium</td>
<td>0.97–1.17</td>
<td>0.30–0.40</td>
<td>1.05</td>
</tr>
<tr>
<td>Silicon</td>
<td>0.9–1.10</td>
<td>0.25–0.40</td>
<td>1.15</td>
</tr>
<tr>
<td>Chromium</td>
<td>1.0–1.16</td>
<td>0.48–0.60</td>
<td>1.8–2.0</td>
</tr>
<tr>
<td>Iron</td>
<td>1.1–1.3</td>
<td>0.40</td>
<td>1.27</td>
</tr>
<tr>
<td>Copper</td>
<td>1.1–1.3</td>
<td>0.50–0.75</td>
<td>2.74–2.8</td>
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<tr>
<td>Be–Cu bronze</td>
<td>2.20–5.0</td>
<td>0.3–0.4</td>
<td>2.23–3.0</td>
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<td>Molybdenum</td>
<td>1.0–1.24</td>
<td>0.40–0.65</td>
<td>2.23–3.0</td>
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<tr>
<td>Silver</td>
<td>1.0–1.4</td>
<td>0.70–0.80</td>
<td>3.2–5.8</td>
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<tr>
<td>Gold</td>
<td>1.31–1.45</td>
<td>0.7–0.8</td>
<td>7.8–8.2</td>
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<tr>
<td>Al2O3</td>
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<td>0.30–0.4</td>
<td>3.0</td>
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<tr>
<td>SiO2</td>
<td>2.5</td>
<td>0.42–0.5</td>
<td>3.0</td>
</tr>
<tr>
<td>Glass passivation</td>
<td>2–3</td>
<td>0.3–0.42</td>
<td>2.0</td>
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<tr>
<td>Ni silicide</td>
<td>1.97</td>
<td>0.8</td>
<td>6.5</td>
</tr>
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<td>GaAs</td>
<td>1.20</td>
<td>0.6</td>
<td>2.6</td>
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<tr>
<td>PVC</td>
<td>1.65</td>
<td></td>
<td></td>
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<tr>
<td>Teflon–FEP</td>
<td>2.21–3.0</td>
<td>0.3–0.4</td>
<td>1.82–1.9</td>
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<tr>
<td>Kapton (polyimide)</td>
<td>2.10</td>
<td>0.15</td>
<td></td>
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<tr>
<td>HPR resist</td>
<td>1.09</td>
<td>0.37</td>
<td>0.55</td>
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<td>PBS resist</td>
<td>0.70</td>
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<tr>
<td>AZ1470 resist</td>
<td>0.9–1.10</td>
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</tr>
</tbody>
</table>

6) Charging effects

Charging-up on a mask for microelectronic

(V$_{acc}$ >> $E_2$) $V_{acc}$=E$_2$
6) Charging effects

Charging-up on spherical silica particles

Charging of particle surface lead to anomalous contrast given a “flying saucer” like morphology at 1.5 kV, close to the neutrality point, particles recover their spherical type contrast.

5 kV

1.5 kV

Contrast reversal in SE mode close to the neutrality point

SiO₂-Cr mask for TEG-FET transistors production

SiO₂ (E₂~3.0keV)  Cr (E₂~1.8keV)

3.0 kV  1.8 kV

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6) Charging effects

Surface potential (voltage) contrast

Charging effects

fiberglass on epoxy

Which polarity ??????

SE contrast is improved and distortions are minimized at low voltages

*TABLE 1. Truth Table for NAND Gate*

<table>
<thead>
<tr>
<th>Condition</th>
<th>Inputs</th>
<th>Output</th>
</tr>
</thead>
<tbody>
<tr>
<td>a</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>b</td>
<td>0</td>
<td>1</td>
</tr>
<tr>
<td>c</td>
<td>1</td>
<td>0</td>
</tr>
<tr>
<td>d</td>
<td>1</td>
<td>1</td>
</tr>
</tbody>
</table>

(from Golstein et al. Practical SEM (1975))
6) Charging effects

Initial first overview scan (time zero)  After scanning region for 5 minutes

Initial surface charging dissipates with time near the charge neutrality point of the material

Charging-up is reduced or even cancelled when working at $E_2$, though finding this can be difficult especially in complex inhomogeneous samples!

Charging can be cancelled under partial atmosphere in a "low vacuum" or "low pressure" Environmental SEM

- Problem: incident electrons from the probe are scattered by the atmosphere ("skirting effect")
- reduced resolution and contrast
- delocalized microanalysis (resolution may decrease to mm!)

Using a charge compensator device may reduce skirting effect by localizing gas near sample surface
8) Charging effects

The variable pressure (VP) mode of the Zeiss Gemini 300 SEM allows the observation of non-conductive samples though resolution is reduced due to “skirting” effects and noise.

6) Charging effects

Charge compensator device on the Zeiss Merlin

- Gas introduced near the sample
- Gas is ionized by electron beam
- Surface charge is reduced due to ion bombardment
- SEM is operated at high voltages instead low voltages, i.e., ionization of the gas and thus surface charge compensation is better at higher voltages
QUESTIONS?