Scanning Electron Microscopy
Techniques

2019-2020
Outline

• EM setup
  Electron sources
  Lenses
  Vacuum system
  Detection system

• SEM
  • Operation, Signals
  • Contrast mechanism
  • Interpretation of images, Challenges
How the SEM works?

- Electrons are accelerated to high energies
- Condenser lens system defines probe size and control probe current
- Scanning coils above Objective lens raster beam on sample
- Objective lens focus probe on sample
- Various detectors surrounding sample collect radiated signals
How the SEM works?

- Image formed step by step by the sequential scanning of the sample with the electron probe (using pair of deflector or scan coils, controlled by the scan generator)
- Monitor and scanning coils are synchronized
  - (one-to-one correspondence between the rastering pattern on the specimen and the rastering pattern used to produce the image on the monitor)
- Intensity of each pixel is proportional to signal received (collected SE/BSE electrons)
- When changing the magnification, we just change the raster size (no change in optics)

**Magnification = Area scanned on the monitor / Area scanned on the specimen**
Magnification

Measure the size of the particle and calculate the magnification

Magnification = Image size / Raster size
= Image pixel size / Raster pixel size

Size of the indicated particle is around:
- 200 nm on the image (scale bar)
- 1 cm on your handout/laptop screens
- 10 cm on the TV screen/projector

What is the magnification of this image?
- a) 50 kX
- b) 0.5 MX
- c) 35.46 kX

You should trust the scale bar and not the indicted magnification
Magnification

Pixel size on your sample? = 10 nm

Pixel size on your screen? = 1 cm

Magnification = $10^{-2} / 10^{-8} = 10^6$

Pixel size on your sample = 5 nm

Pixel size on your screen? = 1 cm

Magnification = $10^{-2} / 5 \times 10^{-9} = 2 \times 10^6$

What happens to the resolution?
Resolution (and visibility)

• **Fundamental**
  - Electron wavelength (beam energy) and diffraction limit:
    $\rightarrow$ Rayleigh criterion
  - Size of the probe $d_p$ (also current)
  - Aberrations: enlarges the probe size

• **Operational and sample**
  - Pixel size
  - Contrast and signal to noise ratio (visibility)
  - Beam energy $\rightarrow$ Interaction volume
  - System/Specimen stability
1. **Beam accelerating voltage (kV)**: the voltage with which the electrons are accelerated down the column;

2. **Probe current (ip)**: the current that impinges upon the specimen and generates the various imaging signals;

3. **Probe diameter or spot size (dp)**: the diameter of the final beam at the surface of the specimen;

4. **Probe convergence angle (αp)**: the half-angle of the cone of electrons converging onto the specimen.

Looking at the diagram it would seem that all we would have to do to maintain adequate probe current in a small probe diameter would be to increase the probe convergence angle.

But this is not the case due to aberrations in the optic system.

A small probe diameter always comes with a decrease in probe current.

**Doing SEM involves understanding the trade offs**
There are a number of points to emphasize about lenses when thinking about SEM:

Electromagnetic lenses are used to de-magnify the image of the beam source and to focus the beam on the specimen.

Condenser lenses are involved in demagnification of the image of the beam source.

The objective lens focuses on the specimen as well as de-magnifies.
Resolution | Probe size

A small probe diameter always comes with a decrease in probe current

Beam-related parameters are interrelated in other ways:
e.g. an increased accelerating voltage (shorter $\lambda$) will result in a more focused probe
\[ \text{small probe with high current, also smaller Airy disk size} \]
\[ \text{BUT a larger interaction volume (will see later)} \]
## Probe size | Effect of aperture

<table>
<thead>
<tr>
<th>Aperture size (micron)</th>
<th>Probe current</th>
<th>Purpose</th>
</tr>
</thead>
<tbody>
<tr>
<td>30</td>
<td>Low</td>
<td>High resolution; Low probe current; Large depth of field</td>
</tr>
<tr>
<td>70</td>
<td>Medium</td>
<td>Usual observation</td>
</tr>
<tr>
<td>100</td>
<td>High</td>
<td>Low resolution but high probe current; Reduced depth of field, more Cs</td>
</tr>
</tbody>
</table>
Resolution (and visibility)

Spatial resolution in the SEM depends on spot size

*Smaller spots give higher spatial resolution*

*But have less current (visibility issue)*

Shorter electron wavelengths mean smaller spot sizes

*Higher electron voltages give higher spatial resolution*

*But larger interaction volume (reduces resolution)*

Many different factors limit spatial resolution, “in practice”, with in the SEM, e.g., combined signals from multiple scattering, *size of interaction volume*, *aberrations*, specimen charging….
Resolution (and visibility)

- **Fundamental**
  - Electron wavelength (beam energy) and diffraction limit: \( \rightarrow \) Rayleigh criterion
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- **Operational and sample**
  - Pixel size
  - Contrast and signal to noise ratio (visibility)
  - Beam energy \( \rightarrow \) Interaction volume
  - System/Specimen stability
Resolution | Pixel size

Nyquist sampling \( (f) = \frac{d}{2} \), where \( d \) = the smallest object, or highest frequency

Thus, the imaging sample rate (or pixel) size should be 1/2 the size of the smallest object you wish to record; e.g. if you need 100 nm resolution, then scan every 50 nm (at least).

**Ways to improve pixel resolution:**
Scanning more points (\( \rightarrow \) longer frame time: specimen drift may become an issue)
Reducing the raster area = increasing magnification
Scan parameters

- 5 pixels
  - 1px = 40 nm
  - 20 nm probe (Under sampling – Poor resolution)

- 10 pixels
  - 1px = 20 nm
  - 20 nm probe (Good sampling – Resolution?)

- 20 pixels
  - 1px = 10 nm
  - 20 nm probe (Over sampling – Blurry image)

- 20 pixels
  - 1px = 10 nm
  - 10 nm probe (Good sampling – Good resolution)

1 sec Dwell time

- 15 sec
  - More pixels

- 60 sec
  - More pixels

- 240 sec
  - Smaller probe
  - Less current

- 240 sec
  - Or more!

Images:
- 0.8 sec 1nA
- 0.8 sec 100pA
- 50 sec 100pA
You must find balance between spot size/pixel, current, and scan speed!

Sample Drift, Beam Damage, and Charging influence your choice of scan speed and current and thus are practical conditions that determine resolution.
Scan parameters

10 pA / 10 S  
Good resolution  
Low signal to noise ratio

10 pA / 160 S  
Good resolution  
Good signal to noise ratio

100 pA / 160 S  
Moderate resolution  
Better signal to noise ratio

1 nA / 160 S  
Poor resolution  
High signal to noise ratio

Low  
High  
Signal to noise ratio

High  
Poor  
Resolution
Visibility

Particles with positive contrast

Particles with negative contrast

100% Intensity change

50% Intensity change

5% Intensity change

Its easier to see small changes in intensity on a low background signal
If the detector's brightness and contrast are not properly adjusted, then it is not possible to correct with image processing; i.e. over/under saturation problem.

Do not throw away the information!
Visibility

5.1 % contrast  3.7 % contrast  2.2 % contrast  1 % contrast

Features on a noisy background:

signal to noise ratio 1:1  signal to noise ratio 1:3  signal to noise ratio 1:7
Focusing

Working distance

Objective lens (focus)

At focus

Sample

Stage

Over focus

Under focus

Emad Oveisi
Focus and depth of field

Under focus  In focus  Over focus

Pixel 1  Pixel 2  Pixel 3

Scan  Scan

Plane of optimum focus

D_f
Depth of field

\[ \tan \alpha = \frac{0.5\delta}{0.5d} = \frac{\delta}{d} \]

\[ d = \text{depth of field} \]
\[ \delta = \text{required spatial resolution} \]
\[ \alpha = \text{convergence angle} \]

For small angles, \( \tan(\alpha) = \alpha \)

\[ d = \frac{\delta}{\alpha} \]

Can control depth of field (\( d \)) with convergence angle (\( \alpha \))

\[ \tan \alpha = \frac{R_{ap}}{WD} \]
\[ d = \delta \frac{WD}{R_{ap}} \]
Depth of field

- 600 um OLA
  - WD: 10 mm
- 200 um OLA
  - WD: 10 mm
  - Depth of field: 20 mm
- 100 um OLA
  - WD: 10 mm
  - Depth of field: 30 mm
Depth of field

Specimen

100 µm

30 µm
SEM signals | Elastic scattering

- **No energy transfer**
- Low angle scattering: Coulomb interaction with the electron cloud
- High angle scattering, or back scattering: Coulomb interaction with nucleus
- Atom is not ionized
- Backscattered electrons have an energy range from 50 eV to nearly the incident beam energy.
- Most backscattered electrons retain at least 50% of the incident beam energy.
• An incident electron ejects a bound electron and scatters with an energy lowered by the electron bound energy.
• The ejected electrons having low energies (5-50 eV) are called secondary electrons (SE) and carry information about the surface topography.
• The incident electron can be scattered by Coulomb interaction with the nucleus.
• In the case of inelastic interaction, there is energy transfer, and the target atom can be ionized.

* Incident electron decelerated due to the electromagnetic field of the atom nuclei
* Energy released in the form of X-ray (White radiation)
SEM signals

Incident electrons interaction with the sample produces:

Electron signals:

**Secondary electrons SE**
Electrons ejected from material at low energies
Topography, low energy $\approx 5\text{--}50\ \text{eV}$

**Backscattered electrons BSE**
Incident electrons that elastically scatter and leave the sample
Atomic number $Z$
Energy $\approx eV_0$ (range from $50\ \text{eV}$ to an energy close to initial energy)

**Auger electrons**
Ejected electrons with an energy characteristic of target elements
Not detected in conventional SEM, surface analysis
SEM signals

https://myscope.training/legacy/sem/background/whatissem/detectors.php
We DO care where the signal (electron) comes from

**Surface signals:**
- Secondary electrons (topography)
- Auger electrons (electronic states, chemistry)

**Sub-surface signals:**
- Backscattered electron (Z contrast, crystallographic information)
- Characteristic X-ray (compositional information)
- Secondary florescence (Cathodoluminescence, band-gap)

**Spatial resolution depends on the size of the interaction volume**
Interaction volume differs with material, accelerating voltage, spot size
Monte Carlo simulations
Interaction volume - Effect of beam energy

The more the beam energy,

- The less the rate of energy loss with distance travelled.
  Electrons enter the specimen with more energy and lose it at lower rate.

- The trajectories near the surface become straighter, so, the more the penetration depth (i.e. larger interaction volume).

Cumulative effects of multiple elastic scatterings cause some electrons to propagate back towards the surface, thus widening the interaction volume.

Red trajectories = backscattering
Blue trajectories = Primary electrons
Top surface images of a TiO2/perovskite/FA-CN device acquired using electron beam energies of 3 and 1 keV, respectively demonstrating the perovskite and covering HTM layer.
Interaction volume - Effect of beam energy

Effect of the accelerating voltage on penetration depth and signal

20 kV:
- Strong penetration
- 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 signal is produced in the C film.
- The C film and its defects become visible

From D.C. Joy, Hitachi News 16 1989
Interaction volume - Effect of beam energy

1kV - Surface features are resolved with high spatial resolution

30 kV – buried interfaces are visible though surface features are less resolved

30 keV imaging should in principle offer higher spatial resolution. However, the larger interaction volume and increased edge effects masks the fine features of the surfaces.
The more the “Z”,

- the more the probability for elastic scattering (shorter mean free path)
- the shorter the penetration depth (i.e. smaller interaction volume).
SEM signal | BSE yield

\[ \eta = \frac{N_{\text{BSE}}}{N_b} \]

- \( \eta \) shows a monotonic increase with atomic number. This relationship forms the basis of atomic number (Z) contrast.
- Areas of the specimen composed of higher atomic number elements emit more backscatter signal and thus appear brighter in the image.
- Z contrast is relatively stronger at lower atomic numbers (see the slope of the line).
- \( \eta \) is almost insensitive to beam energy (expect for lower than 1 kV).
NOTE: There is only a small change in $\eta$ with accelerating voltage. As the accelerating voltage is reduced toward the very lower end (1 keV), $\eta$ increases for low Z elements and decreases for high Z elements.
SEM signal | BSE contrast

Detector A

Detector B
SEM signal | BSE contrast

A + B

Composition (mainly)

A - B

Topography
Which segment configuration for this image?
**BSE energy range:** BSEs follow trajectories which involve very different distances of travel in the specimen before escaping. The energy range for BSEs is thus wide (from 50 eV to that of the incident beam energy). The majority of BSEs, however, retain at least 50% of the incident beam energy ($E_0$).

Generally speaking, higher atomic number elements produce a greater number of higher energy BSEs and their energy peak at the higher end is better defined.
BSE yield depends on “Z” of material, and SE yield depends mainly on the voltage.
**SEM signal | SE yield**

**SE1**: from interactions of the incident probe with specimen atoms.
- SE1s are produced in close proximity to the incident beam and thus represent a high lateral resolution signal.

**SE2** from interactions of the high energy BSEs with specimen atoms.
- Both lateral and depth distribution characteristics of BSEs are found in the SE(II) signal and thus it is a comparatively low resolution signal.

**SE3** are produced by high energy BSEs which strike the pole pieces and other solid objects within the specimen chamber.

**NOTE**: Image signal is displayed at the probe position NOT at the actual SE production position.

**SE2 + SE3 reduce resolution**
Less delocalized contrast at lower voltage
Fracture surface in Ni-Al alloy
SEM signal | SE and BSE yield

Positive charging

Negative charging

Accelerating voltage
• If a sample is titled, the interaction volume is tilted and closer to the surface. Thus, more SE escape from below the surface, giving higher signals.

• The same principle is true for rough surfaces – Sloped surfaces and edges have an interaction volume that is effectively titled and have higher SE signals.

1. Rough Surfaces have high SE image contrast
2. Titling can improve SE image contrast
Do not forget, in SEM:
The signal is displayed at the probe position, not at the actual SE production position!!!
SEM signal | Edge effect

5 kV

30 kV
SEM signal | Edge effect

Images show the effect of different electron voltages (kV) on SEM images:
- 30 kV (a)
- 5 kV (c)
- 15 kV (b)
- 1 kV (d)

Each image demonstrates the variation in detail and contrast of the same sample at different electron beam energies.
**SE vs BSE imaging**

**SE image**

**BSE image**
SE have low energies (5-50eV), and thus are emitted only from surface and possess information about topographical features.

BSE emission depends on “Z”, thus intensity in the BSE images scales with atomic number and depends on local composition.

Specimen: Ferritic stainless substrate initially coated with a porous MnCo$_2$O$_4$ This materials were coupled at high temperature (750° C) with a perovskite material.

Images courtesy of Manuel Bianco, GEM-EPFL
SE vs BSE imaging

- flat material
- rough material
- low Z material
- thin layer of contamination
- low Z material

Dust on WC (different Z materials)
The fact that SEM images can be (somehow!) readily interpreted by viewers derives from the “Light optical analogy”.

![Diagram showing electron beam, detector, specimen, and viewer's line of sight]

The direction of the detector within the image is analogous to where the “illumination” appears to come from.

Since we are used to having illumination from the overhead, so we should rotate the scanned image so that the detector (= sun) appears to be at the top of the image.
Interpretation requires to know where the detector is located.

Since we are used to having illumination from the overhead, we should rotate the scanned image so that the detector/sun appears to be at the top of the image.
SEM contrast | Detector position

In-lens SE detector

Everhart Thornley detector

In-lens SE Detector located directly above and centered

SE2 Detector located on the lower right

Why they are different?

Where is the detector for each image?
Beam induced changes to the sample:

- atom displacement ("knock on")
  - Radiation damage

- chemical bound breaking
  - Radiolysis

- lattice atom vibrations (phonons)
  - Sample heating
Contamination

- Primary example: Hydrocarbon build-up on surface

- *Masks surface features and information about the sample*

**Sources**
- Sample surface
- SEM chamber
- Beam induced degradation and migration of sample compounds

- Plasma cleaning sample prior to observation
- Use gloves when handling samples
Occurs in non-conducting samples (also in samples that are not well grounded)

Charging deflects the low-energy secondary electrons causing image distortions and contrast changes

Ways to mitigate charging
- Coat the sample
- Work at low kV
- Use low currents (noisy images)
- Use the “magic” charge neutrality voltage
- Use high working chamber pressures (environmental SEM)
- Charge compensation devices
Challenges | Charging

Dust particles on a metallic substrate
Challenges | Charging

Depends on:
- material properties (surface resistivity)
- beam conditions (beam energy, current, and scan rate)

Uncoated quartz fragment – 1kV ETD with positive bias
Challenges | Charging

1 kV

Fiberglass*

V close to $E_2$

$\text{SiO}_2$ substrate

5 kV

$V >> E_2$

* Images courtesy B. Senior
Challenges | Charging

Paper under vacuum

Paper in low-vacuum (40 Pa)
Challenges | Charging

Surface charging affects the appearance of the spherical particles!

Close to charge neutrality voltage
Spherical shape of particles is back

SiO$_2$ spherical particles

Images courtesy P.A. Buffat
Some useful literature


- **Physics of image formation and microanalysis**, Springer, by L. Riemer

- **Optique: Fondements et applications**, Dunod, by J.S. Perez
Detector position/geometry (BSE)

A

B

A + B

A - B

Composition (mainly)

Topography
Which segment configuration for this image?

Segments A+B

A-B
B-A

A
B

B
A

A
B

B
A

A
B

B
A

Which segment configuration for this image?