What is Microscopy?

As Wikipedia defines it: Microscopy is the technical field of using microscopes to view objects and areas of objects that cannot be seen with the naked eye (objects that are not within the resolution range of the normal eye).
**Light Microscopes and SEMs are similar in design**

![Light microscope (LM) vs. Scanning electron microscope (SEM)](image)

**Why use SEMs: Large depth of field allows visualization of 3-D morphologies**

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
Electron Microscopes use the interaction of electrons with matter to characterize a material’s structural and composition

- Electrons are accelerated to high energies *(high spatial resolution!)*
- Electron beams are monochromatic *(having the same energy!)*
- Electron beams are shaped *(e.g., focused, collimated etc.)* and directed onto the samples using electron static and magnetic lens and deflector coils
- **The interaction of the high energy electrons produces secondary signals that have intrinsic information, specific to the sample’s properties**

SEM operation: interaction volume

- At high magnification, spatial resolution depends on the size of the interaction volume
- Interaction volume differs material, accelerating voltage, spot size
- Different particles have different interaction volumes sizes and thus different resolution for the same microscope settings
Outline:

This section of the course describes mechanisms by which secondary signals (the ones which we use to characterize our samples) are produced in the SEM through interaction of high energy electron interactions with matter.

1) Advantage of using high energy electrons for characterization

2) High energy electron beams: wavelength and resolution

3) Electron-matter interactions

4) Electron Scattering
   A. Inelastic scattering (Secondary electrons)
   B. Electron scattering (Backscattered electrons)
   C. X-ray generation
   D. Other signals

5) Challenges in imaging with electrons

1) Advantage of using high energy electrons: strongly interacts with matter

High energy electrons are backscattered and produce secondary particles (signals) that provide detailed information about the features of the samples and the material’s properties.
1) Advantage of using high energy electrons: **Resolution**

High energy electrons have short wavelengths that allow us to observe nanoscale features in samples.

Wavelength and thus spot size decrease with increasing electron energy!!

High energy = short wavelength = high spatial resolution

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2) Energy and wavelength (spatial resolution) are proportional
2) High energy electrons have short wavelengths

Electromagnetic radiation: \( E = \frac{hc}{\lambda} \) so if \( \lambda < 5 \text{ nm} \), \( E > 1 \text{ keV} \)

Electron wavelength according to de Broglie equation: \( \lambda = \frac{h}{p} \)

with \( p = m_o v \), \( eV = m_o v^2/2 \), and \( p = (2m_o eV)^{1/2} \)

- **non relativistic** (<50keV):
  \[
  \lambda = \frac{h}{(2m_o eV)^{1/2}}
  \]

- **Relativistic correction** (>50keV or >1% speed):
  \[
  \lambda = \frac{h}{\left[2m_o eV \left(1 + \frac{eV}{2m_o c^2}\right)\right]^{1/2}}
  \]

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2) Wavelength of High Energy Electrons

<table>
<thead>
<tr>
<th>Accelerating voltage [KV]</th>
<th>Nonrelativistic ( \lambda ) [nm]</th>
<th>Relativistic ( \lambda ) [nm]</th>
<th>Mass [x m(_o)]</th>
<th>Velocity [x 10(^8) m/s]</th>
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</tbody>
</table>

- \( e = 1.602 \times 10^{-19} \text{ C} \)
- \( 1 \text{ eV} = 1.602 \times 10^{-19} \text{ J} \)
- \( m_o = 9.109 \times 10^{-31} \text{ kg} \)
- \( m_o c^2 = 511 \text{ keV} \)
- Kinetic energy = \( eV \) (per 1 volt)
- \( h = 6.626 \times 10^{-34} \text{ N-m-s} \)
- \( 1 \text{ amp} = 1 \text{ C/sec} \)
- \( c = 2.998 \times 10^8 \text{ m/sec} \)
2) Spatial resolution of high energy electrons

- Abbe's definition of maximum resolution of an optical system states that the smallest feature resolved is limited by diffraction. That is, the resolving power of a microscope is taken as the ability to distinguish between two closely spaced diffraction (Airy) disks.
  \[ d = \frac{\lambda}{n \sin \theta} \]
- Visible light: \( \lambda \approx 500 \text{ nm} = 5 \times 10^{-7} \text{ m} \)
  resolution \( \approx 300 \text{ nm} \) (1000 atomic diameter)
- Electrons 10 keV: \( \lambda \approx 1.2 \times 10^{-11} \text{ m} \)
  resolution \( \approx 1 \text{ nm} \)
- Spatial resolution in the SEM depends on spot size: Smaller spots have higher spatial resolution
- Shorter electron wavelengths mean smaller spot sizes: Higher electron voltages have higher spatial resolution

Many different factors limit spatial resolution, “in practice”, with in the SEM, e.g., combined signals from multiple scattering, size of interaction volume, coulombic interaction, aberrations, specimen charging....

3) The incident high-energy electron beam produces multiple secondary particles (signals)

Electron Signals

- Secondary electrons (SE): electrons ejected from material at low energy (5 to 50 eV)
- Back scattered electrons (BSE): incident electrons that elastically scatter and leave the sample: range from 50 eV to an energy close to initial energy \( E_0 \)
- Auger Electrons: ejected electrons with an energy characteristic of target elements

Other Electromagnetic radiation

- X rays with continuous energy, resulting from deceleration of incident electrons ("Bremsstrahlung" or "breaking radiation")
- Characteristic X-rays with a distinct energy associated to the target atoms.
- Cathodoluminescence: Visible radiation mainly emitted by insulating or semi-conducting materials.
3) The incident high-energy electron beam produces multiple secondary particles (signals) continued

- Surface signals –
  - Secondary electrons (topography)
  - Auger electrons (chemistry)

- Sub-surface signals
  - Backscattered electrons (phase and crystallographic information)
  - Characteristic X-rays (compositional information)
  - Secondary fluorescence (Cathodoluminescence- band gap)

- Spatial resolution depends on the size of the interaction volume
  - Interaction volume differs material, accelerating voltage, spot size
  - Marco Cantoni will discuss how to determine interaction volumes with Monte Carlo simulations

3) In reality, the interaction volume is not “tear drop” shaped

Monte Carlo Simulation of 5 keV electrons in Copper using 10nm initial beam size

Blue – primary electrons
Red – backscattered electrons
4) High energy electron interaction with matter in the SEM

- 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.

4) Inelastic scattering

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.
4) Backscattered electrons

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 electron energies 50eV – accelerating voltage (most are half of the incident energy)

4) Secondary electron yield depends mainly on the voltage and BSE yield depends on Z of material

BSE yield increases with Z, but this relationship deviates at very low incident electron energies

Ionization occurs more readily at lower incident electron energies
4) Secondary and Backscattered electron images

SE images have low energies (5-50 eV), and thus are emitted only from the 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.

5) Other Inelastic Scattering events

Relaxation processes of the excited state

Characteristic X ray
- X-ray generation
  - X-ray energy characteristic interorbital electron transitions and thus of the element

Visible photon
- Fluorescence
  - Low transition energy, visible or UV photon is emitted

Auger electron
- Emission Auger
  - The relaxing process interacts with an electron with a characteristic energy
5) Other Inelastic Scattering events

\[ E = (E_{K} - E_{L1} - E_{L2}) \]

4) Other Inelastic Scattering events:
Characteristic X-ray generation

Energy Dispersive Spectroscopy

K-edges    L-edges
4) Other signals

Electron-holes creation:
An incident electron onto a semiconductor can excite a valence electron to the conduction band, creating an electron-hole pair.

Cathodoluminescence:
The excited electron recombines with its hole, emitting a photon with the gap energy, in the visible. This technique allows to put forward the defects that modify the gap energy.

Electron incident

\[ E_{\text{gap}} \]

Conduction band
Valence band
Electron that has lost energy

\[ E_{\text{gap}} = h\nu \]

4) Other signals: Examples of CL Images of perovskite solar cell materials using Attolight ROSA SEM-CL

SEM-CL is useful for correlating/detecting:
- phase distribution and composition with luminescent properties
- defect structure with optical properties
- detection of trace elements, their valence and structural position

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5) Challenges in imaging

Beam induced changes to the sample:

- atom displacement ("knock on")
  - Radiation damage
- chemical bound breaking
  - Radiolysis
- lattice atom vibrations (phonons)
  - Sample heating

5) Challenges for imaging

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
5) Challenges for imaging

Charging

- 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
  - 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

5) Challenges for imaging: other effects

1) Leakage:
   - Magnet field from distribution board
   - Stray fields from power lines in the surrounding walls
   - High-tension line located too close to the instrument
2) Low floor strength (acoustic vibrations)
3) Improper grounding
4) Detector discharging and/or contaminated
QUESTIONS?