MS414 Materials Characterization
(소재분석)
Lecture Note 9: SEM

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# Syllabus

1. Overview of various characterization techniques (1 lecture)
2. Chemical analysis techniques (9 lectures)
   2.1. X-ray Photoelectron Spectroscopy (XPS)
   2.2. Ultraviolet Photoelectron Spectroscopy (UPS)
   2.3. Auger Electron Spectroscopy (AES)
   2.4. X-ray Fluorescence (XRF)
3. Ion beam based techniques (6 lectures)
   3.1. Rutherford Backscattering Spectrometry (RBS)
   3.2. Secondary Ion Mass Spectrometry (SIMS)
4. Diffraction and imaging techniques (7 lectures)
   4.1. Basic diffraction theory
   4.2. X-ray Diffraction (XRD) & X-ray Reflectometry (XRR)
   4.3. Scanning Electron Microscopy (SEM) & Energy Dispersive X-ray Spectroscopy (EDS)
   4.4. Transmission Electron Microscopy (TEM)
5. Scanning probe techniques (1 lecture)
   5.1. Scanning Tunneling Microscopy (STM)
   5.2. Atomic Force Microscopy (AFM)
6. Summary: Examples of real materials characterization (1 lecture)

* Characterization techniques in blue are available at KARA (KAIST analysis center located in W8-1)
SEM is an excellent technique for obtaining high resolution images.
Typical Instrument View

Many Options are Built Around a Common Core

Key Applications

- When resolution beyond an optical microscope is needed
- First look at a new problem to see if it is a deposit, particles, pits, etc.
- Critical dimension measurements of small features
- Alternative to TEM when considering time and money
Source of Imaged Electrons

- Source of Auger electron signal
- Source of secondary electron (loosely bound outer shell electrons) signal
- Source of Backscattered electrons
- Source of electron-excited characteristic X-rays
- Source of Bremsstrahlung
- Source of secondary fluorescence

Incident electron beam

Sample Surface

X-ray Resolution
Source of Imaged Electrons

- Secondary electron mode: low KE of SE $\rightarrow$ shallow escape (sampling) depth; intensity mainly determined by surface topography

- Backscattered mode: surface topography + large contribution from Z contrast (brighter as Z increases); larger sampling depth ($\sim X100$ of SE)
Electron Beam-Specimen Interaction

- Elastic scattering:
  - electron-nucleus interaction
  - cross-section, $Q$ (cm$^{-2}$)

  $$Q(> \phi_0) = 1.62 \times 10^{-20} \left( \frac{Z^2}{E^2} \right) \cot^2 \left( \frac{\phi_0}{2} \right)$$

- Inelastic scattering:
  - electron-electron interaction

  $$\frac{dE}{ds} \left( \frac{\text{keV}}{\text{cm}} \right) = -7.85 \times 10^4 \left( \frac{Z \rho}{A E_i} \right) \ln \left( \frac{1.166 E_i}{J} \right)$$

  $$J(\text{keV}) = (9.76Z + 58.5Z^{-0.19}) \times 10^{-3}$$

<table>
<thead>
<tr>
<th>Element</th>
<th>$Z$</th>
<th>$A$</th>
<th>$\rho$ (g/cm$^3$)</th>
<th>$J$ (keV)</th>
<th>$dE/ds$ (keV/cm)</th>
<th>$dE/ds$ (eV/nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Carbon</td>
<td>6</td>
<td>12.01</td>
<td>2.1</td>
<td>0.100</td>
<td>$2.24 \times 10^4$</td>
<td>2.24</td>
</tr>
<tr>
<td>Iron</td>
<td>26</td>
<td>55.85</td>
<td>7.87</td>
<td>0.285</td>
<td>$6.32 \times 10^4$</td>
<td>6.32</td>
</tr>
<tr>
<td>Silver</td>
<td>47</td>
<td>107.9</td>
<td>10.5</td>
<td>0.487</td>
<td>$6.94 \times 10^4$</td>
<td>6.94</td>
</tr>
<tr>
<td>Uranium</td>
<td>92</td>
<td>238.03</td>
<td>18.95</td>
<td>0.923</td>
<td>$9.27 \times 10^4$</td>
<td>9.27</td>
</tr>
</tbody>
</table>
Electron Beam-Specimen Interaction

Monte Carlo simulation of trajectories of energetic electron beam

10 keV

- \( Q \sim \frac{1}{E^2} \) and \( \frac{dE}{ds} \sim \frac{1}{E} \)
- Straighter trajectories near the surface with higher \( E \) (reduced \( Q \))
- Larger interaction volume with higher \( E \)

20 keV

- \( Q \sim Z^2 \) and \( \frac{dE}{ds} \sim Z \)
- Increasing backscattering with higher \( Z \) (larger \( Q \))
- Smaller interaction volume with higher \( Z \)

20 keV, Carbon

20 keV, Iron
Backscattered Electrons

\[ \eta = \frac{n_{BSE}}{n_B} = \frac{i_{BSE}}{i_B} \]

- \( \eta \) increases with \( Z \) (stronger dependence at low \( Z \)'s) \( \Rightarrow \) \( Z \) (composition) contrast

- \( \eta \) increases with \( \theta \) (due to dominant tendency of elastic scattering in the forward direction) \( \Rightarrow \) surface topography

Z dependence of \( \eta \)
\( E_0 = 20 \) keV

Tilt angle dependence of \( \eta \)
(angle between the incident beam and the surface normal)
Secondary Electrons

\[ \delta = \frac{n_{SE}}{n_B} = \frac{i_{SE}}{i_B} \]

- \( \delta \) increases with decreasing \( E \) (for \( E > E_1 \)) due to smaller interaction volume \( \rightarrow \) more surface sensitive with lower \( E \)
- Shallow sampling depth & strong dependence of \( \delta \) with \( \theta \) \( \rightarrow \) surface topography
- The secant behavior doesn’t apply at \( E < E_1 \) (a fraction of keV – a few keV)

R: primary electron path within a distance of \( R_0 \) from the surface

At \( \theta = 0^\circ \) (no tilt), SE signal from \( R_0 \)

With a tilt, SE signal from \( R = R_0 \sec \theta \)
BSE vs SE images

Back Scattered Electron image

Silica-coated Au nanoparticles

Secondary Electron image at 20 kV

Secondary Electron image at 5 kV
SEM instrumentation

Objective Lens

Condenser Lens

$V_0$: accel. voltage

$d_p$: probe size

$\alpha_p$: convergence angle

$l_p$: probe current
Electron Gun: Thermal vs. Field Emission

W Thermal  LaB₆ Thermal  W Field Emitter

Advantages of FE sources
- Smaller spot size: small virtual source size and high numerical aperture = small spot size and high depth of field
- Excellent low voltage performance: low energy spread leads to less image distortion from chromatic aberration
- Long lifetime

<table>
<thead>
<tr>
<th>Source</th>
<th>Brightness (A/cm² sr)</th>
<th>Lifetime (h)</th>
<th>Source size</th>
<th>Energy spread ΔE (eV)</th>
<th>Beam current stability (%/h)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tungsten hairpin</td>
<td>10⁵</td>
<td>40–100</td>
<td>30–100 μm</td>
<td>1–3</td>
<td>1</td>
</tr>
<tr>
<td>LaB₆</td>
<td>10⁶</td>
<td>200–1000</td>
<td>5–50 μm</td>
<td>1–2</td>
<td>1</td>
</tr>
<tr>
<td>Field emission</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Cold</td>
<td>10⁸</td>
<td>&gt;1000</td>
<td>&lt;5 nm</td>
<td>0.3</td>
<td>5</td>
</tr>
<tr>
<td>Thermal</td>
<td>10⁸</td>
<td>&gt;1000</td>
<td>&lt;5 nm</td>
<td>1</td>
<td>5</td>
</tr>
<tr>
<td>Schottky</td>
<td>10⁸</td>
<td>&gt;1000</td>
<td>15–30 nm</td>
<td>0.3–1.0</td>
<td>~1</td>
</tr>
</tbody>
</table>
SEM lenses

Magnetic Lens
• Strongest magnetic field near the side of the polepieces (soft magnetic materials such as iron) → stronger deflection further off-axis

Objective Aperture Size
• Optimal aperture angle minimizing aberration
• Convergence angle → image depth of focus
• Probe current

Electron Gun
Condenser Lens
Aperture
Objective Lens
Sample

α₂: convergence angle
α₃: aperture angle
α₀: aperture angle
q₁, f₁
q₂, f₂
p₁, p₂

Effect of Working Distance

Working Distance

Increasing $W$ (while keeping the focus):
- Larger spot size (degraded image resolution)
- Probe current approximately the same
- Improved depth of focus
Effect of Condenser Lens Strength

Weaker condenser lens:
- Larger spot size (degraded image resolution)
- Higher probe current
Lens Aberrations

Spherical Aberration

\[ d_s = \frac{1}{2} C_s \alpha^3 \]

spherical aberration coefficient

- Limiting \( \alpha \) reduces spherical aberration but reduces the probe current

Aperture Diffraction

\[ d_d = \frac{0.61\lambda}{\alpha} \]

- Dependence on \( \alpha \) opposite to spherical aberration
Chromatic Aberration

\[ d_c = C_c \alpha \left( \frac{\Delta E}{E_0} \right) \]

chromatic aberration coefficient

- Worse chromatic aberration for smaller acceleration voltage \((E_0)\)

Astigmatism

From not perfectly cylindrical lens

- Fixed by a stigmator (device applying a weak supplement magnetic field)
- Repeat: X-stigmator control \(\rightarrow\) focus \(\rightarrow\) y-stigmator control \(\rightarrow\) focus
Resolution of SEM

Resolution, \( d^2 = d_s^2 + d_c^2 + d_{as}^2 + d_d^2 = f(\alpha) \)

At 10 keV, \( \lambda \approx 0.012 \) nm with \( C_s \approx 20 \) nm, \( C_c \approx 10 \) nm, \( \Delta E \approx 2 \) eV

To find \( \alpha \) that minimizes \( d \), set \( d(d^2)/d\alpha = 0 \) and solve for \( \alpha \)

\( \Rightarrow \alpha_{opt} \approx 3 \times 10^{-3} \) rads, \( d_R \approx 5 \) nm

(For best SEM’s, \( d_R \approx 1 \) nm)
Strengths and Limitations

• **Strengths**
  – Relatively simple to operate, can provide image resolution on the order of 1-2 nm.
  – Can be coupled to several analytical techniques
  – Relatively fast imaging
  – Wide range of sample types

• **Limitations**
  – Vacuum compatibility typically required
  – May need to etch for contrast
  – Imaging may spoil subsequent analyses
  – Size restrictions may require cutting
  – Resolution is a strong function of sample and preparation.
EDS is a fast method for elemental identification (all elements with Z > B).
Primary & Secondary Processes

Electron → Excited Ion

Relaxation Process 1

Excited Ion → Relaxation Process 2

Fluorescent X-ray

Auger electron emission
Strengths and Limitations

• **Strengths**
  – Fast elemental ID
  – Coupled with an SEM or STEM can provide mapping
  – <1 um spot size for low kV
  – Readily available

• **Limitations**
  – Quantification requires standards
  – Insulators can be more difficult
  – Spectral interferences
  – Relatively low sensitivity (1%-0.1 at%), worse for low Z