3. Scanning Electron Microscopy

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Outline

a. SEM principle
b. Detectors
c. Electron probe and resolution
d. Depth of field
e. Stereoscopy
f. Electron-matter interaction volume
g. Secondary and back-scattered electrons
h. Contrasts
i. Examples
j. Charging effects
Outline

This chapter will describe the principle of a scanning electron microscope (SEM). We will start with a description of the detectors allowing signal detection, the formation of an electron probe and its influence on the spatial resolution. Then we will define the depth of field and see how to control it, how to do stereoscopy.

In order to understand the image formation and the contrasts observed on a picture, there will be considerations about the electron-matter interaction volume, and then an explanation of the origin of the secondary and back-scattered electrons (SE and BSE). This will allow us to analyse the different possible contrasts of a SEM picture, including artefacts. We will end with application examples.

a. SEM principle

- Image formed step by step by the sequential scanning of the sample with the electron probe
- Image acquisition as numerical data
- Bulk sample
- Imaging the sample « surface » (from 1 nm to ≈1 μm depth depending on the analysed signal
- Contrast is due to secondary electrons (SE) emission or back scattered electrons (or sometimes to photons, RX, absorbed current)
- Resolution: 1 nm to 10 nm
a. SEM principle

Response to incident electrons:

- Secondary electrons SE
topography, low energy = 0-30 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
- Auger Electrons: not detected in conventional SEM, surface analysis
- Cathodoluminescence: photons UV, IR, vis
- Absorbed current, electron-holes pairs creation, EBIC
- Sample heating (phonons)
- Radiation damages: chemical binding break, atomic displacement out of site (knock-on)

Energy spectrum of electrons leaving the sample
b. Detectors

Everhardt-Thornley detector: for SE and BSE

SE: the positive collector voltage (≈ +200 à +400V) attracts the SE toward the detector, the 10kV post acceleration give them enough energy to create a bunch of photons for each SE.

BSE: a negative collector polarisation (≈-100V) repels the SE and the only BSE emitted in the narrow cone to the scintillator are detected (low collection efficiency = poor S/N ratio).
b. Detectors

BSE detectors

BSE Robinson detector: a large scintillator collects the BSE and guides more or less efficiently the light to a photomultiplier

- large collection angle
- works at TV frequency

BSE semiconductor detector: a silicon diode with a p-n junction close to its surface collects the BSE (3.8eV/e−-hole pair)

- large collection angle
- slow (poor at TV frequency)
- some diodes are split in 2 or 4 quadrants to bring spatial BSE distribution info

c. Electron probe and resolution

A "light" source

a magnification section (lenses, apertures...)

a sample (+ a "goniometer")

an illumination section (lenses, apertures...)
c. Electron probe and resolution

Resolution in probe mode (SEM, STEM)

- Spherical aberration
  \[ d_{sph} = C_s \cdot \alpha^3 \]
- Chromatic aberration
  \[ d_{ch} = C_{ch} \left( \frac{\Delta E}{E} + \frac{2 \Delta E}{l} \right) \alpha \]
- Diffraction (Airy, Rayleigh)
  \[ d_d = 0.61 \frac{\lambda}{n \cdot \sin \alpha} \]
- Brillance \( \beta \) conservation
  \[ d_g = \sqrt{\frac{4I}{\pi^2 \beta} \cdot \frac{1}{\alpha}} \]
- Combination
  \[ d_{ech} = \sqrt{d_g^2 + d_{sph}^2 + d_{ch}^2 + d_d^2} \]

Incoherent source

Probes with coherent source: see Mory C, Cowley J M, Ultramicroscopy 21 1987 171

Resolving power ("resolution"): Rayleigh criterium

\[ V_{acc} = 20 \text{ kV}, \Delta E = 1.5 \text{ eV}, \beta = 1.10^5 \]
\[ C_{sph} = 17 \text{ mm}, C_{ch} = 9 \text{ mm} \]

Intensity

83% énergie totale dans tache principale
c. Electron probe and resolution

SEM: Limiting parameters on resolving power $\rho$ with SE

1. High magnification
   
   The probe size (generation of SE1)
   
   \[ r = d_{\text{probe}} \]

2. The volume of interaction (generation of SE2+SE3 from BSE): energy and atomic number influence

3. Low magnification
   
   The screen (or recording media) pixel size $d_{\text{screen}}$
   
   \[ r = d_{\text{screen}}/\text{magnification} \]

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How to increase resolving power?

- Reduce the probe current at constant dose
- Reduce probe size
- Reduce volume interaction
- Reduce $C_{\text{sph}}$
- Increase brilliance
- Reduce $C_{\text{sph}}$ and increase brilliance
- Increase exposure time $t$
- Decrease spot size
- Increase accelerating voltage
- Reduce accelerating voltage
- Short focus lenses:
  - in-lens, semi in-lens, Snorkel
- Field emission gun:
  - Cold emission, thermal assisted, Schottky effect
- Dedicated columns: Gemini, XL30, ...
c. Electron probe and resolution

SEM: Effect of current, probe diameter and image acquisition time

- 10 pA/10 s: Good resolution, but statistical noise
- 10 pA/160 s: Good resolution, less statistical noise
- 100 pA/160 s: Some loss of resolution, still less statistical noise
- 1 nA/160 s: Very few statistical noise, but high resolution loss!

probe size and resolution (no noise)

- Model 100 nm diam. particles
- Particles 100 nm diam., probe dia 2 nm
- Particles 50 nm diam., probe dia 2 nm
- Particles 25 nm diam., probe dia 2 nm
c. Electron probe and resolution

Probesize and resolution (with noise)

- model 100 nm diam.,
  particles
- Particles 100 nm diam.,
  probe diam 2 nm
- particles 25 nm diam.,
  probe diam 2 nm
- particles 50 nm diam.,
  probe diam 2 nm

Intensive SEM/TEM training: SEM
Aïcha Hessler-Wyser

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c. Electron probe and resolution

Current/probe diameter

- Thermionique source: spherical aberration is the most important
  \[ I_{max} = \frac{3\pi^2}{16} \beta C_{sph}^{-2/3} d^{8/3} \]

- Field emission source:
  gun aberrations and chromatic aberration are more important
  \[ I_{max} = cd^{2/3} \]

Tiré de L.Reimer, SEM

Intensive SEM/TEM training: SEM
Aïcha Hessler-Wyser
c. Electron probe and resolution

How to increase resolving power?

- Reduce the probe current at constant dose
- **Reduce probe size**
- Increase exposure time \( t \)
- Decrease spot size
- Increase accelerating voltage
- Reduce volume interaction
- Reduce \( C_{sph} \)
- Increase brillance
- Decrease spot size
- Increase accelerating voltage
- Reduce \( C_{sph} \) and increase brillance
- Short focus lenses:
  - in-lens, semi in-lens, Snorkel
- Field emission gun:
  - Cold emission, thermal assisted, Schottky effect
- Dedicated columns: Gemini, XL30, ...

Modern SEM short focus length: \( C_{sph}=17 \) mm, \( C_{ch}=9 \) mm, \( \Delta E=1.5 \) eV, \( \beta=1.1.10^5 \) A/cm²sr
c. Electron probe and resolution

How to increase resolving power?

- Reduce the probe current at constant dose
- Reduce probe size
- Increase exposure time $t$
- Decrease spot size
- Increase accelerating voltage
- **Reduce volume interaction**
  - Reduce $C_{sph}$
  - Increase brilliance
  - **Reduce accelerating voltage**
    - Short focus lenses:
      - in-lens, semi in-lens, Snorkel
    - Field emission gun:
      - Cold emission, thermal assisted, Schottky effect
- Reduce $C_{sph}$ and increase brilliance
- Dedicated columns: Gemini, XL30, ...

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**SEM: résolution**

Interaction volume versus $E_0$

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

$$Z = cte$$
c. Electron probe and resolution

How to increase resolving power?

- Reduce the probe current at constant dose
- Reduce probe size
- Reduce volume interaction
- Reduce $C_{sph}$
- Increase brillance
- Reduce $C_{sph}$ and increase brillance
- Increase exposure time $t$
- Decrease spot size
- Increase accelerating voltage
- Reduce accelerating voltage
- Short focus lenses: in-lens, semi in-lens, Snorkel
- Field emission gun: Cold emission, thermal assisted, Schottky effect
- Dedicated columns: Gemini, XL30, ...

Short focus length… or… FEG?

Snorkel

$C_{sph}=1.7$ mm, $C_{ch}=1.9$ mm

Regular focus length

$C_{sph}=17$ mm, $C_{ch}=9$ mm

FEG

$C_{sph}=17$ mm, $C_{ch}=9$ mm

---

$\beta=1.10^7A/cm^2sr, \Delta E=1.5$ eV

$\beta=1.10^7A/cm^2sr, \Delta E=0.4$ eV
c. Electron probe and resolution

Resolution loss at low voltage

- Observation de la surface réelle
- Échantillons non-métallisés
- Faible endommagement dû au faisceau

Haute tension/haute résolution:
- Effets de bord
- Détails fins non-résolus
- Fort endommagement dû au faisceau

Shorter objective lens focal length and $C_s$

Short questions

1. What is a condensor lens for?
   a) To create the image of the sample
   b) To reduce the size of the electron source

2. Which parameters influence the resolution in SEM?
   a) The size of the probe
   b) The electron current
   c) The electron energy
   d) The acquisition device
   e) The wavelength
   f) The lens aberration

3. How to reduce the probe size?
   a) By reducing the electron energy
   b) By reducing the apperture size
   c) By increasing the Working Distance
   d) By removing the spherical aberration

4. How to reduce the interaction volume?
   a) By reducing the electron current
   b) By reducing the electron energy
d. Depth of field

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

The depth of field is the depth for which the image is focussed. The depth of field increases when $\alpha$ decreases.

- Increase the working distance
- Reduce objective aperture size

$$h_{\text{prof. champ}} = \max \left\{ \frac{2d_{\text{sonde}}}{\alpha}, \frac{1}{\frac{1}{\text{pixel}} \frac{1}{\text{image}}} \frac{1}{\alpha} \right\}$$

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

Diaphragme aperture

Working Distance

Out of focus

Resolution

Focused
d. Depth of field

Light bulb filament

Depth of field

Résolution \( \rho \)

<table>
<thead>
<tr>
<th>10µm</th>
<th>1µm</th>
<th>100nm</th>
<th>10nm</th>
<th>1nm</th>
</tr>
</thead>
<tbody>
<tr>
<td>10mm</td>
<td>1mm</td>
<td>100µm</td>
<td>10µm</td>
<td>1µm</td>
</tr>
<tr>
<td>0.1µm</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Grandissement (grossissement) \( G \)

\( \lambda = 500 \) nm

LM

SEM

Other examples
d. Depth of field

Effect of the objective aperture diameter

100 µm

50 µm

30 µm

Measuring depth of field: stereoscopy
e. Stereoscopy

The 3rd dimension: stereoscopic vision, anaglyphs
e. Stereoscopy

3-D reconstruction (anaglyph)

3-D reconstruction (pseudo-perspective)
e. Stereoscopy

3-D reconstruction (grey levels)

3-D reconstruction (false colors)
e. Stereoscopy
f. Electron-matter interaction volume

Elastic interaction

Total kinetic energy and momentum are constant  \( E_{el} + E_{at} = \text{cte} \)

The light electron interacts with the electrical field in the heavy atom: **Rutherford scattering**.

Only little energy is transferred, the electron speed does not change significantly in amplitude but only in direction (**elastic scattering**).

<table>
<thead>
<tr>
<th>angle de diffusion</th>
<th>100 kV</th>
<th>1000 kV</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>C</td>
<td>Au</td>
</tr>
<tr>
<td>0.5°</td>
<td>0.5 meV</td>
<td>0.03 meV</td>
</tr>
<tr>
<td>10°</td>
<td>0.15 eV</td>
<td>9 meV</td>
</tr>
<tr>
<td>90°</td>
<td>10 eV</td>
<td>0.6 eV</td>
</tr>
<tr>
<td>180°</td>
<td>20 eV</td>
<td>1.2 eV</td>
</tr>
</tbody>
</table>

Elastic interaction:

Energy transfer from the electron to the target

<table>
<thead>
<tr>
<th>100 kV</th>
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<tr>
<td>10 eV</td>
<td>0.6 eV</td>
</tr>
<tr>
<td>20 eV</td>
<td>1.2 eV</td>
</tr>
</tbody>
</table>

f. Electron-matter interaction volume

Inelastic interaction

part of the total kinetic energy is dissipated (energy loss)

- vibration in molecules or crystals (**phonons** =meV-100meV)
- collective oscillations of electrons (**plasmons** ≈10 eV)
- intra- et interband **transitions**  (≈meV≈1 eV)
- inner shell atom **ionisation** (≈50 to150 keV < eV<sub>0</sub>)
- bond breaking ≈ eV, atom displacement ≈ 10-30 eV (requires \( V_{acc} \ 100\text{kV}...1\text{MV, no longer SEM!} \))
f. Electron-matter interaction volume

Mean free path

Elastic cross-sections $\sigma_{el}$ and mean free path $\Lambda_{el}$; total (elastic+inelastic) mean free path $\Lambda_t$ and electron range $R$

The mean free path is the average path that an electron does before having interaction with an atom.

<table>
<thead>
<tr>
<th>$E$ [keV]</th>
<th>1</th>
<th>5</th>
<th>10</th>
<th>20</th>
<th>30</th>
<th>50</th>
</tr>
</thead>
<tbody>
<tr>
<td>C, Z=6</td>
<td>0.65</td>
<td>0.11</td>
<td>0.055</td>
<td>0.027</td>
<td>0.018</td>
<td>0.012 $\times 10^{-18}$ cm$^2$</td>
</tr>
<tr>
<td>$\rho$ = 2 g cm$^{-3}$</td>
<td>1.5</td>
<td>9</td>
<td>18</td>
<td>37</td>
<td>55</td>
<td>83 nm</td>
</tr>
<tr>
<td>$r = 3$</td>
<td>0.4</td>
<td>2.3</td>
<td>4.5</td>
<td>9</td>
<td>14</td>
<td>20 nm</td>
</tr>
<tr>
<td>$\Lambda_{el}$</td>
<td>0.033</td>
<td>0.49</td>
<td>1.55</td>
<td>4.9</td>
<td>9.7</td>
<td>22.6 nm</td>
</tr>
</tbody>
</table>

Al, Z=13

<table>
<thead>
<tr>
<th>$E$ [keV]</th>
<th>1</th>
<th>5</th>
<th>10</th>
<th>20</th>
<th>30</th>
<th>50</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cu, Z=29</td>
<td>1.26</td>
<td>0.31</td>
<td>0.16</td>
<td>0.08</td>
<td>0.053</td>
<td>0.034 $\times 10^{-18}$ cm$^2$</td>
</tr>
<tr>
<td>$\rho$ = 2.7 g cm$^{-3}$</td>
<td>1.3</td>
<td>5</td>
<td>10</td>
<td>21</td>
<td>31</td>
<td>49 nm</td>
</tr>
<tr>
<td>$r = 1.5$</td>
<td>0.5</td>
<td>2</td>
<td>4</td>
<td>8</td>
<td>12</td>
<td>20 nm</td>
</tr>
<tr>
<td>$\Lambda_{el}$</td>
<td>0.025</td>
<td>0.36</td>
<td>1.14</td>
<td>3.6</td>
<td>7.1</td>
<td>16.7 nm</td>
</tr>
<tr>
<td>$\Lambda_t$</td>
<td>0.025</td>
<td>0.36</td>
<td>1.14</td>
<td>3.6</td>
<td>7.1</td>
<td>16.7 nm</td>
</tr>
<tr>
<td>$R$</td>
<td>0.025</td>
<td>0.36</td>
<td>1.14</td>
<td>3.6</td>
<td>7.1</td>
<td>16.7 nm</td>
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Ag, Z=47

<table>
<thead>
<tr>
<th>$E$ [keV]</th>
<th>1</th>
<th>5</th>
<th>10</th>
<th>20</th>
<th>30</th>
<th>50</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cu, Z=29</td>
<td>1.84</td>
<td>0.64</td>
<td>0.37</td>
<td>0.21</td>
<td>0.15</td>
<td>0.11 $\times 10^{-18}$ cm$^2$</td>
</tr>
<tr>
<td>$\rho$ = 10.5 g cm$^{-3}$</td>
<td>0.64</td>
<td>1.8</td>
<td>3.2</td>
<td>5.6</td>
<td>7.8</td>
<td>10.7 nm</td>
</tr>
<tr>
<td>$r = 0.6$</td>
<td>0.4</td>
<td>1.1</td>
<td>2.0</td>
<td>3.5</td>
<td>4.9</td>
<td>6.7 nm</td>
</tr>
<tr>
<td>$\Lambda_{el}$</td>
<td>0.007</td>
<td>0.11</td>
<td>0.35</td>
<td>1.10</td>
<td>2.26</td>
<td>5.1 nm</td>
</tr>
<tr>
<td>$\Lambda_t$</td>
<td>0.007</td>
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<td>$R$</td>
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<td>0.11</td>
<td>0.35</td>
<td>1.10</td>
<td>2.26</td>
<td>5.1 nm</td>
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Au, Z=79

<table>
<thead>
<tr>
<th>$E$ [keV]</th>
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<th>5</th>
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<th>20</th>
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<th>50</th>
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<tr>
<td>Cu, Z=29</td>
<td>3.93</td>
<td>1.60</td>
<td>1.05</td>
<td>0.67</td>
<td>0.52</td>
<td>0.37 $\times 10^{-18}$ cm$^2$</td>
</tr>
<tr>
<td>$\rho$ = 19.3 g cm$^{-3}$</td>
<td>0.43</td>
<td>1.0</td>
<td>1.6</td>
<td>2.5</td>
<td>3.3</td>
<td>4.6 nm</td>
</tr>
<tr>
<td>$r = 0.2$</td>
<td>0.36</td>
<td>0.9</td>
<td>1.3</td>
<td>2.1</td>
<td>2.7</td>
<td>3.8 nm</td>
</tr>
<tr>
<td>$\Lambda_{el}$</td>
<td>0.003</td>
<td>0.05</td>
<td>0.17</td>
<td>0.51</td>
<td>1.0</td>
<td>2.3 nm</td>
</tr>
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f. Electron-matter interaction volume

Monte-Carlo simulations

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

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

CASINO (Freeware)
- " monte CARlo Simulation of electron trajectory in sOlids "
- by P. Hovongton and D. Drouin
f. Electron-matter interaction volume

Number/Energy of backscattered electrons by Monte-Carlo simulations

1 kV
3 kV
30 kV

Penetration and back-scattering vs elements (Z)

\[ V_{\text{acc}} = 20 \text{keV} = \text{cte} \]

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

- U 20 keV
- Cu 20 keV
- C 20 keV
f. Electron-matter interaction volume

Penetration and back-scattering vs elements (Z)

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

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

- **C:** 5 keV, BSE = 33%
- **Cu:** 5 keV, BSE = 47%
- **U:** 5 keV, BSE = 47%
f. Electron-matter interaction volume

Penetration and backscattering vs energy \( E \)

\[ Z = cte \]

Depth of electron penetration in Cu vs energy \( E_0 \) and yield of electron backscattering BSE (Monte-Carlo simulation):

\[ \text{BSE} = 33\% \]

Cu 20 keV

Cu 1 keV

1 μm

1 μm

Cu 1 keV

Cu 5 keV

Cu 5 keV

g. SE and BSE

"true" secondary electrons \( SE_1 \) and "converted BSE" secondaries \( SE_2 + SE_3 \)

Various SE types from

- \( SE_1 \): incident probe
- \( SE_2 \): BSE leaving the sample
- \( SE_3 \): 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

\[ X_0, Y_0 \]
g. SE and BSE

"true" secondary electrons \textbf{SE1} and "converted BSE" secondaries \textbf{SE2+SE3}

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

![Diagram showing SE and BSE processes](image)

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

![Graph showing relative secondary electron yield vs radius from axis](image)

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, 1959)
**g. SE and BSE**

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

Sample *surface polished* (no topography) and perpendicular to the incident beam direction (intermediate energy \( E_0 = 15 \) keV)

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

\[
I_{\text{BSE}} = I_{\text{pe}} \eta \quad \text{with } I_{\text{pe}} \text{ the intensity of the primary beam, } \eta \text{ the BSE yield}
\]

**SE**: low or no chemical contrast but for light elements

*the topographical contrast will dominate on rough surfaces*

\[
I_{\text{SE}} = I_{\text{pe}} \left( \delta_{\text{pe}} + \delta_{\text{pe}} \eta + \delta_{\text{sur}} \eta \right)
\]

with \( \delta \) the total SE yield, \( \delta_{\text{pe}} \) the yield for SE1 and \( \delta_{\text{sur}} \) the SE3 yield for materials surrounding the sample (pole-pieces...)

---

**g. SE and BSE**

Dust on WC  *(different Z materials)*

- flat material
- rough material low Z material
- low Z material

**SE 25 kVBSE**
g. SE and BSE

Contaminated area around a soldering spot

Toner particle (penetration in light material)
g. SE and BSE

Topographical contrast in SE mode

Effet de l’inclinaison de la surface

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

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

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

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

h. Contrast

SE and BSE topography contrast

For one position (x, y) of the electron probe:

- BSE escape from a "pear" volume around the probe position
- SE1 escape from a thin layer under the entrance surface of the probe
- SE2 escape from a thin layer under the escape surface of BSE

\[ \text{contrast} = 2(I_1-I_2)/(I_1+I_2) \]

- \( I_{SE}^{(0^\circ)} = I_{PE} \cdot \frac{1}{\cos 40^\circ} = I_{PE} \cdot 13\% \)
- \( I_{BSE}^{(0^\circ)} = I_{PE} \cdot 31\% \)
- \( I_{SE}^{(40^\circ)} = I_{PE} \cdot 37\% \)
- \( I_{BSE}^{(40^\circ)} = I_{PE} \cdot 37\% \)

SE1 contrast = 26%
BSE contrast = 18%

BSE topographical contrast is not negligible! Chemical contrast is well observed only on polished samples.
h. Contrast

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)

h. Contrast

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

intensity profile on image
h. Contrast

Size and edge effects

(From L. Reimer, Image Formation in Low-Voltage Scanning Electron Microscopy, (1993))
h. Contrast

Comparison of SE and BSE contrast modes

The observator looks down to the column and the "light" seems to come from the Everhardt-Thornley detector.

What does it suggest?
Which objective information?
h. Contrast

What does it suggest?
Which objective information?
h. Contrast

Detector ?

pyramid?

Detector ?

etch-pit?

Change in SE contrast with the voltage

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

Contrast enhancement at low voltage: less delocalization by SE2.

An example: a fracture in Ni-Cr alloy

Intensive SEM/TEM training: SEM
Aïcha Hessler-Wyser

Contrast

SEM: Effect of the accelerating voltage on penetration and SE signal
(from D.C. Joy
Hitachi News 16 1989)

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.
i. Examples

Physical limit to the imaging in secondary electron mode

Tin grains on a thin carbon film (TEM supporting grid)
HRSEM 25 kV 1 nm nominal resolution
left: SE
right: scanning transmitted electrons (STEM)

The average grain size looks larger in SE (12.3 nm) than in STEM (9.1 nm)

'Delocalisation': the elastic scattering in STEM (Rutherford) occurs at a much closer distance from the atom nucleus than the inelastic coulombian e⁻/e⁻ interaction required to eject a SE
### i. Examples

**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 (SE$_1$=cte)

(by courtesy of Dr. K. Leifer, IPEQ/EPFL)

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**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)
j. Charging effects

Fiberglass on epoxy

(by courtesy of B. Senior CIME/EPFL)

j. Charging effects

Improving SE contrast at low voltage

fiberglass on epoxy

Which polarity ??????
j. Charging effects

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

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

when $eV_{\text{acc}} = E_2$ charging-up disappears!

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

Caution: $E_1$ and $E_2$ are specific to the material, but also change with the incidence angle $\theta$!

Charging-up on a mask for microelectronic

$(\text{SiO}_2 \text{ substrate, photoresist, SE mode})$

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

Charging-up on spherical silica particles

charges at the particle surface lead to anomalous contrast as a flying saucer

at 1.5 kV, close to the neutrality point, particles recover their sphere contrast

Charging-up effects

Observation of insulating samples

Charging-up is reduced or even cancelled when working at $E_2$

Charging-up may be cancelled under partial atmosphere in a "low vacuum" or "low pressure" SEM, ESEM

- Caution the "skirt" (incident electrons from the probe are scattered out of it by the atmosphere
- reduced resolution and contrast
- delocalized microanalysis (may attain mm!)
j. Charging effects

Contrast reversal in SE mode close to the neutrality point

\( \text{SiO}_2-\text{Cr} \) mask for TEG-FET transistors production

\( \text{SiO}_2 \) (\( E_2 \approx 3.0 \text{keV} \))

\( \text{Cr} \) (\( E_2 \approx 1.8 \text{keV} \))

3.0 kV

1.8 kV

Some values of the neutrality \( E_2 \) energy

<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>
</tr>
<tr>
<td>Be–Cu bronze</td>
<td>2.20–4.0</td>
<td>0.3–0.4</td>
<td>2.23–3.0</td>
</tr>
<tr>
<td>Molybdenum</td>
<td>1.0–1.24</td>
<td>0.40–0.65</td>
<td>2.62</td>
</tr>
<tr>
<td>Silver</td>
<td>1.0–1.4</td>
<td>0.70–0.80</td>
<td>3.2–5.8</td>
</tr>
<tr>
<td>Gold</td>
<td>1.31–1.45</td>
<td>0.7–0.8</td>
<td>7.8–8.27</td>
</tr>
<tr>
<td>( \text{Al}_2\text{O}_3 )</td>
<td>2.60–3.0</td>
<td>0.30–0.4</td>
<td>3.0</td>
</tr>
<tr>
<td>( \text{SiO}_2 )</td>
<td>2.5</td>
<td>0.42–0.5</td>
<td>6.5</td>
</tr>
<tr>
<td>Glass passivation</td>
<td>2.3</td>
<td>0.3–0.42</td>
<td>2.0</td>
</tr>
<tr>
<td>Ni silicide</td>
<td>1.97</td>
<td>0.8</td>
<td>6.5</td>
</tr>
<tr>
<td>GaAs</td>
<td>1.20</td>
<td>0.6</td>
<td>2.6</td>
</tr>
<tr>
<td>PVC</td>
<td>1.65</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Teflon–FEP</td>
<td>2.21–3.0</td>
<td>0.3–0.4</td>
<td>1.82–1.9</td>
</tr>
<tr>
<td>Kapton (polyimide)</td>
<td>2.10</td>
<td>0.15</td>
<td></td>
</tr>
<tr>
<td>HPR resist</td>
<td>1.09</td>
<td>0.37</td>
<td>0.55</td>
</tr>
<tr>
<td>PBS resist</td>
<td></td>
<td>0.70</td>
<td></td>
</tr>
<tr>
<td>AZ1470 resist</td>
<td></td>
<td>0.9–1.10</td>
<td></td>
</tr>
</tbody>
</table>

\( E_2 \): 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
**j. Charging effects**

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)

**j. Charging effects**

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

Surface potential (voltage) contrast

![Image of a micrograph showing charging effects in a semiconductor circuit]

**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 0</td>
<td>0</td>
</tr>
<tr>
<td>b</td>
<td>0 1</td>
<td>1</td>
</tr>
<tr>
<td>c</td>
<td>1 0</td>
<td>1</td>
</tr>
<tr>
<td>d</td>
<td>1 1</td>
<td>0</td>
</tr>
</tbody>
</table>

(from Golstein et al. Practical SEM (1975))