Characterization with TEM

- Introduction to electron microscopy, diffraction and nano-imaging

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Introduction

• TEM microscope
• Aspects of electron interaction
• Diffraction
• Characterization in TEM
Resolution

• Wavelength limit
• VLM: about 300 nm (half green light)
• Electrons: 4 pm (100 keV)

\[ d = \frac{0.61\lambda}{\mu \sin(\beta)} \]
TEM cross section

[Image of TEM cross section diagram]

The electron source

• Two types of emission sources
  • Thermionic emission
    • W or LaB6

  • Field emission
    • W ZnO/W

Cold FEG  Schottky FEG
Field emission

• Current density:

\[ j_c = \frac{k_1 E^2}{\phi} \exp\left(\frac{k_2 \phi^{3/2}}{E}\right) \]

Fowler-Norheim

Maxwell-Boltzmann energy distribution for all sources
Electron lenses

Any axially symmetrical electric or magnetic field has the properties of an ideal lens for paraxial rays of charged particles.

- **Electrostatic**
  - Require high voltage - insulation problems
  - Not used as imaging lenses, but are used in modern monochromators or deflectors

- **Magnetic**
  - Can be made more accurately
  - Shorter focal length

\[ \vec{F} = -e \vec{E} \]

\[ \vec{F} = -e(\vec{v} \times \vec{B}) \]
General features of magnetic lenses

- Focuses near-axis electron rays with the same accuracy as a glass lens focuses near axis light rays.
- Same aberrations as glass lenses.
- Converging lenses.
- The bore of the pole pieces in an objective lens is about 4 mm or less.
- A single magnetic lens rotates the image relative to the object.
- Focal length can be varied by changing the field between the pole pieces (changing magnification).

http://www.matter.org.uk/tem/lenses/electromagnetic_lenses.htm
Electron Interaction
Electron scattering

• **Elastic**
  - The kinetic energy is unchanged
  - Change in direction relative to incident electron beam

• **Inelastic**
  - The kinetic energy is changed (loss of energy)
  - Energy form the incident electron is transferred to the electrons and atoms in the specimen

• **Coherent**
  - Elastically scattering electrons are usually coherent

• **Incoherent**
  - Inelastic electrons are usually incoherent (low angles (<1°))
  - Elastic scattering to higher angles (>~10°)
Scattering form the specimen

Total scattering cross section/The number of scattering events per unit distance that the electrons travels through the specimen:

$$\sigma_{\text{total}} = N \sigma_{\text{atom}} = N_o \sigma_{\text{atom}} \rho / A$$

$N =$ atoms/unit volume
$N_o$: Avogadros number, $\rho$: density of specimen,
$A$: atomic weight of the scattering atoms

If the specimen has a thickness $t$ the probability of scattering through the specimen is:

$$t \sigma_{\text{total}} = N_o \sigma_{\text{atom}} \rho t / A$$
Mean free path $\lambda$

The mean free path for a scattering process is the average distance travelled by the primary particle between scattering events.

$$\lambda = \frac{1}{\sigma_{\text{total}}} = \frac{A}{N_o \rho \sigma_{\text{atom}}}$$

<table>
<thead>
<tr>
<th>Material</th>
<th>10kV</th>
<th>20kV</th>
<th>30kV</th>
<th>40kV</th>
<th>50kV</th>
<th>100kV</th>
<th>200kV</th>
<th>1000kV</th>
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<tr>
<td>C (6)</td>
<td>5.5</td>
<td>22</td>
<td>49</td>
<td>89</td>
<td>140</td>
<td>550</td>
<td>2200</td>
<td>55000</td>
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<tr>
<td>Al (13)</td>
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<td>7.4</td>
<td>17</td>
<td>29</td>
<td>46</td>
<td>180</td>
<td>740</td>
<td>18000</td>
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<tr>
<td>Fe (26)</td>
<td>0.15</td>
<td>0.6</td>
<td>2.9</td>
<td>5.2</td>
<td>8.2</td>
<td>30</td>
<td>130</td>
<td>3000</td>
</tr>
<tr>
<td>Ag (47)</td>
<td>0.15</td>
<td>0.6</td>
<td>1.3</td>
<td>2.3</td>
<td>3.6</td>
<td>15</td>
<td>60</td>
<td>1500</td>
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<tr>
<td>Pb (82)</td>
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<td>0.34</td>
<td>0.76</td>
<td>1.4</td>
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<td>8</td>
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<tr>
<td>U (92)</td>
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<td>0.19</td>
<td>0.42</td>
<td>0.75</td>
<td>1.2</td>
<td>5</td>
<td>19</td>
<td>500</td>
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</tbody>
</table>

(Mean free path (nm) as a function of acceleration voltage for elastic electron for scattering more than 2° events.)
Lattice properties of crystals

• The crystal structure is described by specifying a repeating element and its translational periodicity
  • The repeating element (usually consisting of many atoms) is replaced by a lattice point and all lattice points have the same atomic environments.

Crystals have a periodic internal structure
Atoms and lattice points situated on corners, faces and edges are shared with neighbouring cells.
Bravais lattice

The point lattices can be described by 14 different Bravais lattices

Hermann and Mauguin symbols:
- P (primitive)
- F (face centred)
- I (body centred)
- A, B, C (base or end centred)
- R (rhombohedral)
Electron diffraction

- Bragg’s Law \( n\lambda = 2d \sin(\Theta) \)
- Since \( \lambda \) is very small, Bragg angles are also small, so the Bragg Law can be simplified to:
  \( \lambda = 2d\Theta_B \)

Si [111] diffraction. (Futz and Howie)
Diffraction

• Thin sample
• Transmitted
• Coulomb interaction
• Diffraction pattern

High res TEM overview.mp4

http://nanohub.org/resources/4640
Real or Reciprocal Space?

• Comparing images is equally good
• Convert images with fast Fourier transformation (FFT)
• Real space: «scattering intensites», some resolution lost with FFT
• Realspace is more intuitive, easy to interprate.
Determination of an unknown crystalline phase

Tilting series around a dense row of reflections in the reciprocal space

Positions of the reflections in the reciprocal space

50 nm
Diffraction of small volumes

- The thin-foil effect
- In reciprocal space: “small becomes large”
Characterization methods

- XEDS
- EELS

From Williams and Carter (2009)
• TEM vs SEM
• High resolution of a small area
• Real or reciprocal images
• Comprehensive characterization by diffraction and complimentary methods in TEM machine.

• Thin, small sample (or else electrons are backscattered more than transmitted)
• Trade in. More spatial resolution, for less spectroscopy resolution (XRD/BSE)
• Price
• Size of the machine
References


• [2] Fultz and Howe, TEM and Diffractometry of Materials

Web Pages:
• http://nanohub.org/members/23993/usage
• http://www.matter.org.uk/diffract ion/
Thank You!