Chapter 2
Instrumentation for Analytical Electron Microscopy
Lecture 4
Outline

**Electron Sources (Electron Guns)**
- Thermionic: LaB$_6$ or W
- Field emission gun: cold or Schottky

**Lenses**
- Focusing
- Aberration
- Probe size

**Imaging**
- Diffraction
- Bright field imaging (BF)
- Dark field imaging (DF)
- Phase contrast imaging
- Scanning transmission electron microscopy (STEM) mode
- Holography mode
- etc.

**Detectors**
- electron detectors
- X-ray detectors
- energy loss spectrometers
Transmission Electron Microscope
Electron Microscopy

Specimen

Electron Beam

Scanning Electron Microscope (SEM) Dealing mainly with Surface

Backscattered Secondary Electron to Image Specimen Topography

Transmission Electron Microscope (TEM) Dealing mainly with Internal Structure

Transmitted and Diffracted Electron to Image Specimen Internal Structure
Electron / Sample Interaction

Auger electrons
E ≤ 10 eV

backscattered electrons
E_0 - E

Electron-hole pairs

Bremsstrahlung X-rays
0 < \hbar \nu < 10 eV.

visible light, cathodoluminescence
0 < \hbar \nu < E_n.

Electron diffraction

Imaging + electron energy loss spectroscopy (EELS)

Characteristic X-rays for EDX

Electron beam
E_0

secondary electrons
E ≤ 20 - 50 eV

positive ions

thin sample

uncattered electrons
E_n

elastically scattered electrons
E_n - E

Absorbed electrons

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Electron-specimen Scattering

- Electron scattering: without interactions we observe nothing
- Interaction cross-section: the probability of an event occurring (unit: barns)
- Elastic scattering: *diffraction*
- Inelastic scattering:
  1. x-ray emission: characteristic, Bremstrahlung
  2. Secondary electrons
  3. Auger electrons
  4. Plasmons
  5. Phonons
- Beam damage: radiolysis and knock-on damage
• Diffuse zone in electron diffraction pattern
• Every information of interacted electron beam is valuable for analysis of specimen structure. Don’t skip it
Typical ED patterns in SiO grains at 500, 750 and 1000 °C respectively. Diffuse rings became sharper in spite of the high temperature. The diffraction rings can be identified as those of Si and cristobalite.
• Diffuse zone in electron diffraction pattern
• Every information of interacted electron beam is valuable for analysis of specimen structure. Don’t skip it
Illumination system: SEM and TEM are similar

Objective lens and stage

Observation /imaging system
Scanning Electron Microscope
# Family of Electron Microscopes

<table>
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<tr>
<th>Gun</th>
<th>Condenser Lens</th>
<th>Scan Coils</th>
<th>Detectors</th>
<th>Specimen</th>
<th>Objective /projective lens</th>
<th>Screen Counter</th>
<th>EELS</th>
<th>CCD Camera</th>
<th>Microscope</th>
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<td>Analytical STEM</td>
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History

- 1897 J.J. Thomson announces the existence of negatively charged particles (later termed electrons).
- 1924 Louis de Broglie proposes that moving electrons have wavelike properties (received Noble Prize in 1929).
- 1926 Hans Busch proves that it is possible to focus electron beams with cylindrical magnetic lenses.
- 1931 Ernst Ruska and Max Knoll build the first transmission electron microscope.
  - Publication: M. Knoll, E. Ruska: Das Elektronenmikroskop. Z. Physik 78, 318-339 (1932)
- 1935 Max Knoll demonstrates the feasibility of scanning electron microscopy (SEM).
- 1939 Siemens Co. produces the first commercial transmission electron microscope in Berlin, Germany.
History TEM

First experimental proof (1931) that specimens can be imaged and magnified by electron (Busch) lenses. (U= 50-75 kV), resolution limit of $\approx 5$-10 Å

Sketch by E. Ruska (1931) of the cathode ray-tube for testing one-stage and two-stage electron-optical imaging
History TEM

Ernst Ruska, 1939

1931 Max Knoll and Ernst Ruska built the first TEM

1939 First Commercial Electron Microscope by Siemens Co.
The newest Cs-Corrected Transmission Electron Microscope

Model: FEI Titan 80-300
Purpose: HRTEM, STEM, EELS, Energy Filtered TEM, Tomography, Electron Holography, Lorentz microscopy
Description: up to 300kV accelerating voltage, HRTEM point-to-point resolution 0.08nm with Cs-corrector of objective lens, 0.13nm STEM resolution
Additions: Cs-corrector, Lorentz-lens system & Bi-prism (Holography), Gatan Tridiem (EELS), Tomography holder
Illumination system:
SEM and TEM are similar

Objective lens and stage

Observation/ imaging system
Electron Illumination Sources

Major electron beam parameters

- Electron probe diameter, $d_p$
- Elect on probe current, $i_p$
- Electron probe convergence, $\alpha_p$
- Accelerating voltage, $V_0$

For AEM we require:
- A stable source of electron
- A large current in a small spot

There are three source types:
- A tungsten thermionic source
- A LaB6 thermionic source
- A field emission source
A. Thermionic Emission

Electron escapes from metal when it acquires enough thermal energy to overcome the work function (\(\Phi\)) barrier.

Richardson’s law:

\[
J_0 = AT^2 e^{-\frac{\Phi}{kT}}
\]

A/cm\(^2\)

J\(_0\): Current density
T: operating temperature
K: Boltzmann’s constant, 8.5x10\(^{-5}\) eV/K
A: Richardson’s constant
Tungsten and LaB6 are used electron sources.

In the thermionic electron gun, electrons are emitted from a heated filament and then accelerated towards an anode. A divergent beam of electrons emerges from the anode hole.
a) Schematic of a thermionic electron gun; b) photograph of the cross-sectioned Wehnelt assembly of the JEOL 120CX microscope.
Electron Illumination Sources

B. Field Emission

\[ E = \frac{V}{r} \]

- **E**: Electrical field strength
- **V**: voltage
- **r**: radius of tungsten tip

In the field emission gun, a very strong electric field \((10^9 \text{ Vm}^{-1})\) is used to extract electrons from a metal filament. Temperatures are lower than that needed for thermionic emission.

This gives a much higher source brightness than in thermionic guns, but requires a very good vacuum.

In order to get high field strengths with low voltages, the **field emitting tip** has a strong curvature. This is done by etching a single crystal tungsten wire to a needle point. This is welded on to a hairpin filament.

The emitting region can be less then 10 nm.

The **anode** is a positively charged metal plate, i.e. at earth potential, that has a hole in it.

Its function is to accelerate the electron beam to the HT potential.
Schematic illustration of a Schottky electron gun.
Electron Source Brightness

\[ \beta = \frac{i_p}{\pi \left( \frac{d_p}{2} \right)^2 \pi (\alpha_p)^2} = \frac{4i_p}{(\pi d_p \alpha_p)^2} \]

where: \( \alpha_p \) is the convergence angle of the beam
\( i_p \) is the probe current
\( d_p \) is the probe diameter

The unit of brightness is \( \text{A/cm}^2/\text{steradian} \)

Brightness is constant throughout the electron column if lens aberration are ignored

Brightness is proportional to the accelerating voltage (not entirely true for field emission guns, FEGs)
Thermionic sources: turning up the current

Emission (cathode) current, \( i_e \)

At saturation, an increase in current does not increase emission current. Operating at higher current shortens the life of the filament.
## Comparison of Electron Sources

<table>
<thead>
<tr>
<th>Source</th>
<th>Brightness $\beta$ (A/m$^2$/rad)</th>
<th>Source size $\mu$m</th>
<th>Energy spread $\Delta E$ (eV)</th>
<th>Lifetime hr</th>
</tr>
</thead>
<tbody>
<tr>
<td>w</td>
<td>$10^9$</td>
<td>10</td>
<td>1-3</td>
<td>40-100</td>
</tr>
<tr>
<td>LaB6</td>
<td>$5 \times 10^{10}$</td>
<td>5</td>
<td>1-2</td>
<td>500-1000</td>
</tr>
<tr>
<td>Cold FEG</td>
<td>$10^{13}$</td>
<td>0.005</td>
<td>0.3</td>
<td>&gt;1000</td>
</tr>
<tr>
<td>Schottky</td>
<td>$10^{13}$</td>
<td>0.015</td>
<td>1.0</td>
<td>1000</td>
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</tbody>
</table>

Best source? It depends on the applications and how much money you have
Calculating the beam diameter (or called probe size in convergent beam)

- The beam diameter is defined as the full width at half maximum (FWHM) of the Gaussian distribution.

- The actual beam diameter results from:
  (a). the diameter of the original beam leaving the electron gun, \( d_g \),
  (b). broadened by the effect of spherical and chromatic aberration in the lenses \( d_s, d_c \),
  (c). diffraction at the aperture, \( d_d \).

All effects depend on the current density, \( j_c \), convergence angle (\( \alpha_p \)), brightness, \( \beta \), spherical/chromatic aberration coefficient \( C_s/C_c \), and wavelength, \( \lambda \).
Gaussian Probe size (or spot size)

The equation for brightness:

\[ \beta = \frac{4i_p}{(\pi d_p \alpha_p)^2} \]

Can be re-arranged to obtain:

\[ d_g = \left( \frac{4i_p}{\beta \pi^2 \alpha_p^2} \right)^{1/2} = \frac{C_0}{\alpha_p} \]

where \( C_0 = \left( \frac{4i_p}{\beta \pi^2} \right)^{1/2} \)

Note: The brightness is important: the higher brightness, the smaller spot size.

If there were no aberrations, a simple increase in convergence angle \( \alpha_p \) would decrease the probe diameter. But aberrations complicate matters.
Spherical aberration, Cs

Focus for marginal rays is nearer to lens than the focus for paraxial rays.

\[ d_s = 0.5 C_s \alpha_p^3 \]

\( d_s \): beam broadened by sphere aberration

Cs: sphere aberration coefficient

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Chromatic aberration, $C_c$

$$d_c = \frac{\Delta E}{E} C_c \alpha_p$$

- $d_c$: beam broadened by chromatic aberration
- $C_c$: chromatic aberration coefficient
- $\Delta E/E$: fractional variation in electron beam voltage

Disc of least confusion $d_c$

- Shorter wavelength to a focus near to lens
- Longer wavelength to a focus further to lens
Aperture Diffraction

$d_d$: beam broadened by diffraction

$\lambda$: electron wavelength

$$d_d = \frac{0.61 \lambda}{\alpha_p}$$

The finite size of lens and aperture causes a point to be imaged as a disc (Airy disc) rather than a point in the image plane.
Astigmatism

- Magnetic in-homogeneities in the lens materials
- Machining errors
- Dirty apertures or lenses
- Astigmatism is fully correctable using an octupole stigmator
- So ignore astigmatism in calculating probe size
A general expression for the beam size, $d_p$:

$$d_p^2 = d_g^2 + d_s^2 + d_c^2 + d_d^2$$

For best resolution in many applications we need to use the smallest beam diameter. For STEM mode, the smallest probe size determines the best resolution. Since $d_p$ includes all the disks of least confusion from all lens aberrations, image resolution can be obtained from above equation.
Final Probe Size (cont’)

\[ d_p^2 = d_g^2 + d_s^2 + d_c^2 + d_d^2 \]

\[ C_0 = \left( \frac{4i_p}{\beta \pi^2} \right)^{1/2} \]

For a thermionic gun, \( C_0 \gg \lambda \), the contribution of \( d_d \) and \( d_c \) can be neglected.

\[ \alpha_{optimum} = \left( \frac{4}{3} \right)^{1/8} \left( \frac{C_0}{C_s} \right)^{1/4} \]

\[ d_{\text{min}} = \left( \frac{3}{4} \right)^{1/8} C_0^{\frac{3}{4}} C_s^{\frac{1}{4}} \approx 0.96 C_0^{\frac{3}{4}} C_s^{\frac{1}{4}} \ldots \ (a) \]
Final Probe Size (cont’)

\[ d_{p}^{2} = d_{g}^{2} + d_{s}^{2} + d_{c}^{2} + d_{d}^{2} \]

\[ C_{0} = \left( \frac{4i_{p}}{\beta \pi^{2}} \right)^{1/2} \]

For a FEG gun, \( C_{0} \ll \lambda \), the contribution of \( d_{g} \) and \( d_{c} \) can be neglected.

\[ \alpha_{\text{optimum}} = 0.9 \left( \frac{\lambda}{C_{s}} \right)^{1/4} \]

\[ d_{\text{min}} = 0.8 \lambda^{4} C_{s}^{1/4} \]

These expressions can be used to estimate the optimum aperture angle and the resolution limit of a high resolution TEM/STEM. Equation (b) is especially important for evaluating the capabilities of different TEM instruments.

Note that for resolution depends more strongly \( \lambda \) than \( C_{s} \). This encourages the use of high accelerating voltage (small \( \lambda \)). Cs can be corrected by current technology.
Probe size @ V0=200 KV and $\lambda=2.5$ pm

**Diagram:**

- **dg**
- **ds**
- **dd**

**Labels:**

- $\alpha_p$
- FEG
- LaB6
- W

**Legend:**

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Summary of Probe Formation

• Small electron probe generation is limited by
  1. spherical aberration
  2. Chromatic aberration
  3. diffraction error

• Careful choice of convergence angle (final aperture size) is critical to forming the smallest probe with highest current

• Too large an aperture will result in spherical aberration

• Too small an aperture results in low probe current and a larger aberrated probe due to diffraction effects
HW# 7, due day: 09/17/08

(a) Derive the equation for optimum aperture angle $\alpha_{opt}$ and the minimum size of the focused beam $d_{min}$ for a field emission gun.

(b). Using the results obtained in part (a), plot $\alpha_{opt}$ (rad) and $d_{min}$ (nm) as the accelerating voltage at 100, 200, 400 Kv, for Cs=1 and 3mm. What can you conclude from these graphs?

Next lecture
- Magnetic lens in TEM
- TEM imaging mode
- Sample preparation

Demo time: 4:00-4:30 PM, Tuesday, 09/16/08.
I will meet you at 170H, HRC