Transmission Electron Microscopy
I. Introduction

EMA 6518
Spring 2007

01/08/07
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• Time: Mon Wed 5:40-6:55pm

• Location:

• Office hours: Fri 4:00-5:00pm or by appointment

• Prerequisite: EMA 5507
• Reference:
  Transmission Electron Microscopy
  I: Basics
  II: Diffraction
  III: Imaging
  IV: Spectrometry
  by D.B. Williams and C.B. Carter

• Grading:
  ✓ 1 Written exam: 20%
  ✓ Lab: 30%
  ✓ Homework: 20%
  ✓ Report & presentation: 30%
Extremely expensive equipment!

- A typical commercial TEM costs about $2 (up to $4-5) for each electron volt of energy in the beam.
- Beam energy of a TEM: 100,000-40,000 eV

- Why use electrons?
- Why you need TEM to characterize materials?
- Advantages and Drawbacks?
Brief History

• In 1801, Thomas Young passed a beam of light through two parallel slits in an opaque screen, forming a pattern of alternating light and dark bands on a white surface beyond. This led Young to reason that light was composed of waves.

wave theory of light

Thomas Young’s Double Slit Experiment

Thomas Young (1773-1829)
Brief History

• In 1897, J.J. Thomson discovered “corpuscles”, small particles with a charge/mass ratio more than 1000 times greater than that of protons, swarming in a sea of positive charge (“plum pudding model”).

Sir Joseph John Thomson
(1856-1940)
Nobel prize 1906

Thomson’s 2nd Cathode ray experiment
Brief History

• In 1924, Louis de Broglie first theorized that the electron had wave-like characteristics. Application of the idea of particle–wave dualism (only known for photons up to then) for any kind of matter. (first person to receive a Nobel Prize on a PhD thesis)

Louis Victor de Broglie (1892-1987)
Nobel prize 1929

Electron=Particle & Wave

$$\lambda = \frac{h}{p} = \frac{h}{mv}$$
In 1926, Hans Busch discovered that magnetic fields could act as lenses by causing electron beams to converge to a focus (electron lens).
Brief History

• In 1927, Davisson and Germer, Thomson and Reid, independently carried out their classic electron diffraction experiments (demonstration of wave nature of electrons)

Sir George Paget Thomson (1892 – 1975)
Nobel Prize: 1937
(shared with C.J. Davison)

Electron=Wave

GP Thomson Experimental Apparatus and Results

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Brief History

• In 1931, Knoll (inventor of SEM, 1935) and Ruska co-invent electron microscope and demonstrated electron images.

Knoll and Ruska co-invent electron microscope

Max Knoll (1897-1969)

Ernst Ruska (1906-1988)
Nobel Prize 1986
Brief History

• 1938: M. von Ardenne: 1st STEM
• 1936: the Metropolitan Vickers EM1, first commercial TEM, UK
  – 1939z: regular production, Siemens and Halske, Germany
  – After World War II: Hitachi, JEOL, Philips, RCA, etc
• 1945: 1nm resolution
• 1949: Heidenreich first thinned metal foils to electron transparency
• Cambridge group developed the theory of electron diffraction contrast
• Thomas pioneered the practical applications of the TEM for the solution of materials problems (1962)
• ……
Microscope

- Bright-field microscope
- Dark-field microscope
- Phase-contrast microscope
- Fluorescence microscope
- Confocal microscope
  - Electron beam
    - Constant distance
  - Constant current

- Scanning Electron Microscope (SEM)
- Transmission Electron Microscope (TEM)
  - Electron beam

- Scanning Probe Microscope (SPM):
  - Atomic force microscope (AFM)
  - Scanning tunneling microscope (STM)
  - UV, violet, or blue light
  - ≥2 optical lenses
  - Resolution: wavelength of light

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Optical vs. Electron Microscopy
Optical vs. Electron Microscopy

• Easy to use
• Samples in air or water
• Total magnification: ×100-1000
  product of the magnifications of the
  ocular lens and the objective lens
• Image processing by CCD

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http://www.microscopyu.com/articles/optics/components.html
Optical vs. Electron Microscopy

SEM
Optical vs. Electron Microscopy
Optical vs. Electron Microscopy

(a) Optical micrograph of the radiolarian *Trochodiscus longispinus*. (b) SEM micrograph of same radiolarian. (Taken from J.I. Goldstein et al., eds., Scanning Electron Microscopy and X-Ray Microanalysis, (Plenum Press, NY, 1980).)
Optical vs. Electron Microscopy

Why electrons?

- Resolution
- Depth of Focus

Resolution:

- Our eyes: 0.1-0.2 mm
- Optical microscope: 400-700 nm, resolution?
- Electron microscope: 100-1000 keV, resolution?
Diffraction

- Image formed by a small circular aperture (Airy disk) as an example
- Image by a point source forms a circle with diameter $1.22 \lambda f/d$ surrounded by diffraction rings (airy pattern)
- Diffraction is usually described in terms of two limiting cases:
  - Fresnel diffraction - near field.
  - Fraunhofer diffraction - far field.
Rayleigh resolution

- Rayleigh suggested that a reasonable criterion for resolution ($R = \text{distance between A and B}$) is that the central maximum of one point source lies at the first minimum of the Airy pattern of the other point ($R = \text{diameter of circle}$).
Rayleigh resolution

- The numerical aperture (NA) of a lens represents the ability of the lens to collect diffracted light and is given by $NA = n \sin \alpha$ in this expression. $n$ is the index of refraction of the medium surrounding the lens, and $\alpha$ is the acceptance angle of the lens ($n = 1$ for air). 

$$R = \frac{1.22 \lambda f}{d} = \frac{1.22 \lambda f}{n(2f \sin \alpha)} = \frac{0.61 \lambda}{n \sin \alpha} = \frac{0.61 \lambda}{NA}$$

Rayleigh resolution: $R = \frac{0.61 \lambda}{NA}$ 

Practical resolution: $R = k_1 \frac{\lambda}{NA}$ where $0.6 < k_1 < 0.8$
Why electrons?

- The ability to “resolve” tiny objects improves as the wavelength decreases. Consider the microscope objective:

\[ \alpha_c = 1.22 \frac{\lambda}{D} \]

The minimum \( d \) for which we can still resolve two objects is \( \alpha_c \) times the focal length:

\[ d_{\text{min}} \approx f\alpha_c = 1.22\lambda \frac{f}{D} \]

A good microscope objective has \( f/D \approx 2 \), so with \( \lambda \approx 500 \) nm the optical microscope has a resolution of \( d_{\text{min}} \approx 1 \mu m \).
Why electrons?

- **Resolution of Electron microscope:** ?

- **Wave Behaviors**
  - images and diffraction patterns
  - wavelength can be tuned by energies

- **Charged Particle Behaviors**
  - strong electron-specimen interactions
  - chemical analysis is possible

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**Light**
- \( p = \frac{h}{\lambda} \) (matter also)
- \( p = \frac{E}{c} \)
- \( E = hf = \frac{hc}{\lambda} \)

**Matter**
- \( p = \frac{h}{\lambda} \) (light also)
- \( p = \sqrt{2mE} \)
- \( E = \frac{h^2}{2m\lambda^2} \)

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**Planck's constant**
- \( h = 6.626 \, 0693(11) \times 10^{-34} \) J \cdot s
- \( h = 4.135 \, 667 \, 43(35) \times 10^{-15} \) eV \cdot s

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Wavelength of an Electron

• The DeBroglie wavelength of an electron:

\[ \lambda = \frac{h}{p} \]

• the relation between the electron’s wavelength and its kinetic energy \( E \).

\( p \) and \( E \) are related through the classical formula:

\[
E = \frac{p^2}{2m} \quad \text{m}_e = 9.11 \times 10^{-31} \text{kg}
\]
\[
E = \frac{h^2}{2m\lambda^2} \quad h = 4.14 \times 10^{-15} \text{eV} \cdot \text{s}
\]

For \( m = m_e \):

(electrons)

Don’t confuse with for a photon!

\[
E = \frac{1.505 \text{eV} \cdot \text{nm}^2}{\lambda^2}
\]

\[
E_{\text{photon}} = \frac{1240 \text{eV} \cdot \text{nm}}{\lambda}
\]
Why electrons?

- For a 100 keV electron:
  \[ \lambda \sim 0.004 \text{nm} \ (4 \text{pm}) \]

- BUT nowhere near building TEMs that approach this wavelength limit of resolution, because we can’t make perfect electron lenses.

- HRTEM

- HVEM: 1-3 MV (1960s)
  300-400 kV (1980s), very high resolution close to that achieved at 1 MV

Figure 1.2. A twin boundary in spinel stepping from one \{111\} plane to another parallel plane. The white dots are columns of atoms. The change in atomic orientation across the twin boundary can be readily seen, even if we do not know what causes the white dots or why, indeed, they are white.
You wish to observe a virus with a diameter of 20 nm, which is much too small to observe with an optical microscope. **Calculate the voltage required** to produce an electron DeBroglie wavelength suitable for studying this virus with a resolution of $d_{\text{min}} = 2$ nm. The “f-number” for an electron microscope is quite large: $f/D \approx 100$. 
To accelerate an electron to an energy of 5.6 keV requires 5.6 kilovolts.
Depth of Focus & Depth of Field

• Depth of focus is a lens optics concept that measures the tolerance of placement of the image plane (e.g. film plane in a camera) in relation to the lens.

At f/32, background is distracting

DOF = \( k_2 \lambda / (NA)^2 \)

Shallow DOF at f/5 isolates flowers from the background.

• The very small angular aperture of the electron probe forming system permits a large depth of field all in focus at once.
Depth of Focus & Depth of Field

- We have to use very small limiting apertures in the lenses, narrowing the beam down to a thin “pencil” of electrons (few micronmeters across).
  - In SEM, to produce 3D-like images
  - In TEM, usually in focus at the same time, independent of the specimen topography (as long as it’s electron transparent)

“depth of field” refers to the specimen
“depth of focus” refers to the image
Interaction of high energy (~kV) electrons with (solid) materials
Electron Beam-Specimen Interactions

-------------------Visualizing the interaction volume

- The interaction volume can be observed in certain plastic materials such as PMMA
- Undergo Molecular bonding damage during electron bombardment that renders the material sensitive to etching in a suitable solvent
- Polymethylmethacrylate (PMMA)
- e-beam: 20 keV, ~ 0.5µm

This phenomenon is the basis for EB lithography

(Everhart et al., Proc. 6th Intl. Conf. on X-ray Optics and Microanalysis)
Scanning Electron Microscope

• SEM permits the observation and characterization of heterogeneous organic and inorganic materials on a nm to µm scale.
  » Imaging capabilities
  » elemental analysis

• In the SEM, the area to be examined or the microvolume to be analyzed is irradiated with a fine focused electron beam, which may be swept in a raster across the surface of the specimen to form images or maybe static to obtain an analysis at one position.

• The types of signals produced from the interaction of electron beam with the sample include secondary electrons, backscattered electrons, characteristic x-rays, and other photons of various energies.
Interaction of high energy (~kV) electrons with (solid) materials

Figure 1.3. Signals generated when a high-energy beam of electrons interacts with a thin specimen. Most of these signals can be detected in different types of TEM. The directions shown for each signal do not always represent the physical direction of the signal but indicate, in a relative manner, where the signal is strongest or where it is detected.
Interaction of Electrons with Matter

• Electrons are one type of “ionizing radiation”—capable of removing one of the tightly bound inner-shell electrons from the attractive field of the nucleus.
• “Ionizing radiation” produces many of the secondary signals from the specimen are used in “analytical electron microscopy” (AEM)
Abbreviations

• HEED: high energy electron diffraction
• LEEM: low energy electron microscope (many variations with special names)
• EELS: electron energy loss spectroscopy
• EDXS: energy dispersive X-ray spectroscopy
• SEM: scanning electron microscope (electrons do NOT normally transmit the sample)
Interaction of Electrons with Matter

• Modern TEMs are very good signal-generating instruments.
• Electron beam: typically <10 nm and at best <1 nm
• Combining TEM and SEM — STEM
Diffraction

• Electron diffraction is an indispensable part of TEM and is the most useful aspect of TEM for materials scientists.
  • Crystal structure, lattice repeat distance, specimen shape, point-group and space-group determination, etc.

Figure 1.6. TEM diffraction pattern from a thin foil of Al-Li-Cu containing various precipitate phases, shown in the inset image. The central spot (X) contains electrons that come directly through the foil and the other spots and lines are diffracted electrons which are scattered from different crystal planes.
Limitations of the TEM

- Sampling---0.3mm$^3$ of materials
- Interpreting transmission images---2D images of 3D specimens, viewed in transmission, no depth-sensitivity.
- Electron beam damage and safety---particularly in polymer and ceramics
- Specimen preparation---”thin” below 100nm
Different Kinds of TEMs

A wide variety of types:

- HRTEMs
- HVEMs
- IVEMs
- STEMs
- AEMs
Some Fundamental Properties of Electrons

• Typical electron beam current in a TEM is 0.1-1μA, which corresponds to $10^{12}$ electrons passing through the specimen plane.

• With 100-keV energy, these electrons travel at about 0.5c ($1.6\times10^8$ m/s), so they are separated by 0.16 cm and this means that there is never more than one electron in the specimen at any one time.

• Electron diffraction and interference occur, both of which are wave phenomena, and imply interaction between the different electron beams.
# Some Fundamental Properties of Electrons

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Charge ( e )</td>
<td>(-1).602 \times 10^{-19} \text{ C}</td>
</tr>
<tr>
<td>1 eV</td>
<td>1.602 \times 10^{-19} \text{ J}</td>
</tr>
<tr>
<td>Rest mass ( m_0 )</td>
<td>9.109 \times 10^{-31} \text{ kg}</td>
</tr>
<tr>
<td>Rest energy ( m_0 c^2 )</td>
<td>511 \text{ keV}</td>
</tr>
<tr>
<td>Kinetic energy ( (\text{charge} \times \text{voltage)} )</td>
<td>1.602 \times 10^{-19} \text{ N m} (for 1 volt potential)</td>
</tr>
<tr>
<td>Planck’s constant ( h )</td>
<td>6.626 \times 10^{-34} \text{ N m s}</td>
</tr>
<tr>
<td>1 ampere</td>
<td>1 \text{ C/sec}</td>
</tr>
<tr>
<td>Speed of light in vacuum ( c )</td>
<td>2.998 \times 10^{8} \text{ m/sec}</td>
</tr>
</tbody>
</table>
Some Fundamental Properties of Electrons

<table>
<thead>
<tr>
<th>Accelerating voltage (kV)</th>
<th>Nonrelativistic wavelength (nm)</th>
<th>Relativistic wavelength (nm)</th>
<th>Mass ($\times m_0$)</th>
<th>Velocity ($\times 10^8$ m/s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>100</td>
<td>0.00386</td>
<td>0.00370</td>
<td>1.196</td>
<td>1.644</td>
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<td>2.823</td>
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