Introduction: Basics of Transmission Electron Microscopy (TEM)

TEM Doctoral Course MS-637
April 10th-12th, 2017

Outline

1. What is microscopy?
2. Why do we use electrons as a probe?
3. Electron-matter interactions in a thin sample
4. Components of the TEM
5. TEM imaging modes
1) What is Microscopy?

Wikipedia defines it: Microscopy is the technical field of using microscopes to view objects and areas of objects that cannot be seen with the naked eye (objects that are not within the resolution range of the normal eye).

Light (optical) Microscope  
Scanning Electron Microscopes (SEM)  
Transmission Electron Microscopes (TEM)

1) What is electron microscopy?

- The electron microscopy is the use of specialized microscopes that interact a high energy electron beam with samples as a means to probe a material’s structure.
- Electron microscopes have a greater resolving power than light microscopes, allowing it to see much smaller objects in finer detail (sub nanometer resolution).
- EMs are large, expensive pieces of equipment, generally stand-alone instruments in a small, specially designed room and requiring trained personnel to operate them. *(quoted from the internet site of ASU)*
2) Why do we use electrons as a probe?

1. Easy to produce high brightness electron beams
2. Easily manipulated
3. High energy electrons have a short wavelength
4. Interact strongly with matter

- Electrons are accelerated to high energies *(which gives high spatial resolution!)*
- Electron beams are monochromatic *(having the same energy means less chromatic aberrations!)*
- Electrons beams are shaped (e.g., focused, collimated etc.) and directed onto the samples using electron static and magnetic lens and deflector coils
- The interaction of the high energy electrons produces secondary signals that have intrinsic information, specific to the sample’s properties

I will discuss more in the next lecture about electron guns, optics and detectors
Why do we use electrons as a probe? Resolution!

High energy electrons have short wavelengths that allow us to observe nanoscale features in samples.

2) Why do we use electrons as a probe?
High energy = short wavelengths = high spatial resolution

Electromagnetic radiation: $E = \frac{hc}{\lambda}$ so if $\lambda < 5$ nm, $E > 1$ keV

Electron wavelength according to de Broglie equation: $\lambda = \frac{h}{p}$

with $p = m_e v = 2m_e eV$

Electrons

$\gamma$ X UV visible IR radio

$10^{-32}$ $10^{-9}$ $10^{-6}$ $10^{-3}$ $10^3$ $10^{10}$
2) Why do we use electrons as a probe?

Electron charge (e) \(-1.602 \times 10^{-19} \text{ C}\)
1 eV \(1.602 \times 10^{-19} \text{ J}\)
Electron rest mass \((m_0)\) \(9.109 \times 10^{-31} \text{ kg}\)
Electron rest energy \((m_0c^2)\) \(511 \text{ keV}\)
Kinetic energy (charge \times\) tension \(1.602 \times 10^{-19} \text{ Nm (per 1 volt)}\)
Plank's constant \((h)\) \(6.626 \times 10^{-34} \text{ Nm-s}\)
1 amp \(1 \text{ C/sec}\)
Light speed in vacuum \((c)\) \(2.998 \times 10^8 \text{ m/sec}\)

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Green light : \(\lambda \approx 532 \text{ nm}\), 
\(\beta\) (objective collection angle) \~1 \text{ rad}
\(n=1.7\) for oil immersion lens
*** \(d=200\text{nm}\)
Electrons 200 keV : \(\lambda \approx 0.00251\text{nm}\)
\(n=1\) for vacuum 
max \(\beta=0.1\ \text{rad given TEM geometry}
***\(d=0.015\ \text{nm}\)

- **Lens aberrations limit spatial resolution**
- Spherical and Chromatic Aberration corrections allow for 0.05 nm resolution
2) Why do we use electrons as probe?

Without Cs correction, optimal convergence angle and resolution are given by:

- $2.5 \times 10^{-12} \text{ m} @ 200 \text{ kV}$
- 1-10 mrad
- $d$ (spatial resolution) is 0.2-3 nm

Higher convergence angle produces more aberrations which lower spatial resolution.

With Cs Correction:

$d$ is 0.06 to 0.07 nm


Why do we use electrons as probe?
Electrons interact strongly with matter
3) Electron-matter interactions in a thin sample

Interaction of electrons with the sample:

- Backscattered electrons (BSE)
- Secondary electrons (SE)
- Visible light
- Characteristic X-rays
- Electrons directly absorbed in the specimen
- Electron-hole pairs
- Bremsstrahlung X-rays
- Inelastically scattered electrons
- Elastically scattered electrons

1-100 nm

Two Categories of electrons:

1. Elastically scattered
   - Coherent
     - Bragg diffracted electrons (selected area electron diffraction, bright-field, dark-field, weak beam)
     - Phase Contrast imaging (HRTEM)
   - Incoherent
     - Mass-thickness contrast imaging
     - Z-Contrast imaging (HAADF STEM)
     - Backscattered electrons

2. Inelastically scattered
   - Secondary signals
     - Characteristic X-rays and Bremsstrahlung
     - Visible light (CL)
     - Auger electrons
   - Incoherent
     - Secondary Electrons
     - Electron Energy Loss Spectroscopy (EELS)
3) Electron-matter interactions in a thin sample: Elastic scattering

Elastic scattering

No energy transfer
- Low angle scattering: Coulomb interaction with the electron cloud.
- High angle scattering (or back scattering): Coulomb interaction with nucleus.
- Atoms are not ionized.

Example: Bragg diffraction

Bragg angle

High angle scattered electron

Example: Rutherford scattering, so-called Z-contrast

θ

The Bragg's law

Bragg diffraction occurs when radiation, with a wavelength comparable to the atomic spacing, is scattered by the atom centers and undergoes constructive interference. The path difference, $d_{hkl}$, associated to the scattering angle, $\theta$, is given by

$$d_{hkl} = n \lambda / 2 \sin \theta$$

Elastic diffraction

$$| \mathbf{k} | = | \mathbf{k'} |$$

Periodic arrangement of atoms in the real space:
$\mathbf{g}$ : vector in the reciprocal space

Bragg's Law

$\ell \lambda = 2d \sin \theta$

Constructive interference when

$\theta = \frac{\pi}{2}$

$k$

$k'$

$\mathbf{g} = \mathbf{k} - \mathbf{k'}$

**Dr. Duncan Alexander will discuss this topic in detail in his lectures this afternoon.**

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3) Electron-matter interactions in a thin sample: Diffraction Contrast imaging

Using Bragg diffracted electrons, you can generate
1. Diffraction patterns, which have crystallographic information, i.e., crystal symmetry, lattice parameters, atom postions, etc…
2. Bright-field images that hold information about morphology, grain size, defect structures, etc…
3. Dark-field images, which can be used to correlate crystalline phase and orientation to grain morphologies, distribution with the microstructure, defect structures, etc.

Phase contrast for crystalline specimen

Sounds simple:

Electron beam

Crystal structure properly oriented

Objective lens

Projected image

Si [110] orientation

**HRTEM will be discussed in more detail in lectures on Tuesday**
3) Electron-matter interactions in a thin sample: Phase Contrast and HRTEM

Unfortunately things are much more complicated

- Electron beam
- Crystal thickness with a given orientation affects phase and amplitude of the beam in a complicated way
- Direct and Diffracted beams (Phase shift!)
- Objective lens (with aberrations affects phase shifts)
- Mixing of information from the sample and microscope via INTERFERENCES!
- Projected amplitude after interference!

3) Electron-matter interactions in a thin sample: Phase Contrast and HRTEM

Thickness-defocus map in Fe₃Al intermetallics

Next nearest neighbour antiphase boundary in the Fe₃Al. The inset shows simulation of the atomic configuration of the defect.
3) Electron-matter interactions in a thin sample: Incoherent elastic scattering – Mass thickness

Mass Thickness Contrast:
- Incoherent elastic scattering that results from the difference in the atomic number (Z) and/or thickness (t)
- Scattering is proportional to $Z^2 t$
- Higher-Z or thicker areas will appear darker in bright-field TEM mode
- Being incoherent, this contrast mechanism applies to both crystalline and amorphous materials

Mass thickness Contrast examples: Hollow polymer spheres with different wall thicknesses

3) Electron-matter interactions in a thin sample: Incoherent elastic scattering - STEM

Focused e-probe scanned on sample; disc and annular detectors in back focal (diffraction) plane

High-angle annular dark-field => compositional contrast:
intensity $\propto Z^2 t$
(thickness t, atomic number Z)

EELS spectrometer

Z-contrast examples:
- Pt catalyst on Al$_2$O$_3$
- Si-Ge/Si multilayer
- Cs-corrected - graphene with dopant atoms (Krivanek et al., Nion)
3) Electron-matter interactions in a thin sample: Inelastic Scattering

- An incident electron ejects a bound electron and scatters with an energy lowered by the electron bound energy.
- The ejected electrons having low energies (5-50 eV) are called secondary electrons (SE) and carry information about the surface topography.
- The incident electron can be scattered by Coulomb interaction with the nucleus.
- In the case of inelastic interaction, there is energy transfer, and the target atom can be ionized.

Electron absorption

- atom displacement ("knock on")
  - Radiation damage
- chemical bound breaking
  - Radiolysis
- lattice atom vibrations (phonons)
  - Sample heating
- charge collective oscillation excitation (plasmons)
- excitation of surface electronic level (transition valence/conduction,...)
- core atomic level excitation (ionization)
- Bremsstrahlung radiation
- the relative impact of these various interaction mechanisms varies with the type of material (no simple modelling of absorption)
3) Electron-matter interactions in a thin sample: Inelastic Scattering

Relaxation processes of the excited state

- **X-ray generation**
  - X-ray energy characteristic interorbital electron transitions and thus of the element

- **Fluorescence**
  - Low transition energy, visible or UV photon is emitted

- **Emission Auger**
  - The relaxing process interacts with an electron with a characteristic energy

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3) Electron-matter interactions in a thin sample: Inelastic Scattering

- **Ejected electron**
  - $E = E_{\text{vac}} - E_{\text{in}}$
- **Vacuum**
- **Conduction Band**
- **E$_F$**
- **Valence Band**
- **Atomic energy levels**
- **Energy loss electron**
  - $E = E_{\text{in}} - \Delta E$ ($\Delta E > E_{\text{in}}$)
- **Characteristic X-rays**
  - $E_X = E_{\text{K}} - E_L$
- **Auger electrons**
  - $E_{\text{K}} - E_{\text{L1}} - E_{\text{L2}}$

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3) Electron matter interactions in a thin sample: Inelastic Scattering

Characteristic X-rays

Energy Dispersive Spectroscopy

Scanning TEM EDS mapping

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3) Electron-matter interactions in a thin sample: Inelastic Scattering

Interaction with plasmons and core losses

- Plasmons are collective oscillations of weakly bound electrons – plasmon losses dominate in materials with “free electron” bonding (can be used to calculate thickness)
- Core losses (atom ionization) depends on atomic species and thus carries some chemical information about the sample as well as bonding, valance states, density of states (pre-edge ELNES), near neighbour coordination (post edge, EXEFS), etc.

Energy filtered TEM with core loss electrons
3) Electron-matter interactions in a thin sample: Inelastic Scattering

Energy filtered TEM with core loss electrons

Example taken from KIT - LEM - Research - Solid oxide fuel cells

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3) Electron-matter interactions in a thin sample: Catholuminescence

Electron-holes creation:
An incident electron onto a semiconductor can excite a valence electron to the conduction band, creating an electron-hole pair.

Cathodoluminescence:
The excited electron recombines with its hole, emitting a photon with energy equal to the band gap, that is usually in visible range. This technique can be used to understand how defects modify the band gap in nanostructures.

Electron incident
Summary
Why do we use electrons as probe?

1. Easy to produce high brightness electron beams
   ➢ High coherence beams allow us to generate diffraction patterns and high spatial resolution images

2. Easily manipulated
   ➢ Electron lenses and deflectors can be used to easily change focal lengths and beam directions which is a necessary operating condition for flexible imaging devices

3. High energy electrons have a short wavelength
   ➢ Shorter wavelengths mean higher spatial resolution (Raleigh Criterion)

4. Electrons interact strongly with matter
   ➢ Secondary signals have information specific to the material
   ➢ Bragg diffracted electrons — structure, orientation, phase distribution, defect content and structures, etc.

4) Components of the TEM

1. Electron Source

2. Sample Illumination (Condenser lenses)

3. Imaging lens (Objective)

4. Magnification and projection (intermediate and projector lenses)

5. Detectors
4) Components of the TEM

1. Electron Source

**Electron gun**: generates and accelerates the electrons to the desired energy (velocity)

**Filament**: emits the electrons

**Accelerator stage**: accelerates them to high energies >60keV

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A typical TEM: Jeol 200cx

2. Illumination or Condenser system

1. Demagnifies gun crossover (electron “point” source) on sample
2. First condenser lens (C1) defines probe size
3. Second Condenser (C2) lens controls the illumination area (intensity) on the sample
4. Third condenser (or condenser mini) lens controls convergence angle
5. Apertures control signal, convergence angle and coherence

A typical TEM: Jeol 200cx
4) Components of the TEM

2. Illumination or Condenser system

3. Objective lens (forms the image!)
4) Components of the TEM

3. Objective lens (forms the image!)

- Eucentric Focus is the “standard” or optimal objective lens current (focus)
- Eucentric height/plane is the optimal specimen position at the objective lens standard focus
- Image will appear over or under focus if specimen is not at the eucentric plane

4. Intermediate and Projector lenses

1. Intermediate lenses are used to switch between imaging & diffraction mode
2. Intermediate lenses are used to change magnification and camera length, i.e., objective lens current.
3. Three intermediate lenses are necessary to compensate for the spiraling of electrons and resulting image rotation
4. Projector lens used to greatly magnify the last intermediate lens image plane onto the detector
4) Components of the TEM

4. Intermediate and Projector lenses

Apertures

Small, 10-200 \( \mu \text{m} \) holes (generally 4 to 8 sizes to choose from) limiting the beam diameter in specific imaging planes and lens crossovers to change properties of illuminating electron beam or specimen scattered electrons

1. Condenser (2): used to change probe size, convergence angle and electron beam coherence
2. Objective: used to form diffraction (and phase) contrast images and other type images by omitting scattered beams
3. Selected Area: used to limit or select the region which contributes to the electron diffraction pattern
4) Components of the TEM

4. Detectors

1. Phosphor screen
2. Film
3. Image plates
4. CCD or CMOS cameras
5. YAG scintillator – multichannel plates
6. Phosphor – photomultiplier tube
7. X-ray detectors
8. BSE, SE, STEM detectors

I will discuss more in the next lecture about electron guns, optics and detectors

Summary
Components of a TEM

1. Electron Source
   - Produces high energy, large current, and high coherence electron beams necessary for generating diffraction patterns and high spatial resolution images
2. Condenser lenses
   - Controls spot size and illumination area on sample (beam intensity)
3. Objective lens
   - Images sample and is strongest lens in the system
4. Intermediate and projector lenses
   - Changes modes from diffraction to imaging
   - Controls magnification
5. Detectors
   - Various different configurations designed to collect secondary signals produced by the high-energy electron beam
5) TEM imaging modes: Mass-thickness contrast

- Areas of higher mass thickness scatter electrons more than others
- Electrons are captured by the aperture and lost from the beam path
- Areas of higher mass thickness will therefore appear dark in the image
- This is known as
  - mass thickness contrast,
  - scattering contrast,
  - aperture contrast or
  - amplitude contrast!
- Applies to both Crystalline and Amorphous materials

5) TEM imaging modes: Fresnel contrast – phase contrast

Fresnel fringes

- Fringes arise due to the high coherence of the electron beam and interface of two electron waves, one which is not scattered (vacuum) and one that scatters off the edges of sample features (the hole in this example), resulting in a path length difference and modulation with that has a fringe contrast.
- Light or dark fringe thickness depends on focus!

1-2) Under-focused, uniform white fringes
3) Focussed, min of contrast, no fringes
4-5) Over-focused, uniform dark fringes
5) TEM imaging modes: Fresnel contrast

Fresnel fringes can also be used to observe and correct the astigmatism of the objective lens.

1) Good Astigmatism, uniform fringe
2) Bad Astigmatism, non-uniform fringe

5) TEM imaging modes: Fresnel contrast

a) Under-focused, dark core
b) Over-focused, 2 dark fringes and white core
c) Measured wire width ZZ at under-focused condition
d) Measured wire width ZZ at over-focused condition

Note!! ZZ ≠ ZZ**

Width of Fresnel fringes depends on the thickness, defocus, beam properties and lens aberrations. Do not use them to assist in measuring particles sizes unless you have calculated how they relate to the true particle size!
5) TEM imaging modes

- **Diffraction mode**
  The intermediate and projector lens magnify and project the **back focal plane** (first diffraction pattern formed in the microscope) of the objective lens to the detector system.

- **Imaging mode**
  The intermediate and projector lens magnify and project the **image plane** of the objective lens to the detector system.

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5) TEM imaging modes: Selected Area Electron Diffraction

- **Diffraction pattern** is produced in the **Back focal plane** of the objective lens.
- **Selected area aperture** that is inserted in the “**image plane**” defines the area from which diffraction beams are transmitted to intermediate and projector lenses.
- The intermediate lenses are adjusted to either focus on the objective image plane (TEM image mode) or back focal plane (TEM diffraction mode). They also define magnification and camera length.
5) TEM imaging modes: Selected Area Electron Diffraction

Bragg Diffraction: Camera Length

Selected Area Electron Diffraction (SAED)

Diffraction spots converge at infinity (far-field)

The intermediate and projective lenses allow us to get this focal plane into our microscope:

The magnification of the diffraction pattern is represented by the camera length $CL$.

$$\tan(2\theta_{hkl}) = R/CL$$

For small angles, $\theta = \sin \theta = \tan \theta = R/CL$

and with the Bragg's law $2d_{hkl} \sin \theta_{hkl} = n \lambda$

we have: $d_{hkl} R = \lambda CL (=\text{constant})$

Right image is diffraction pattern from a fine grained material (Polycrystalline TiCl fcc calibration sample) that produces "rings" if the selected area is large

Scattering from angles which undergo constructive interference or "reflections" (i.e. all atomic planes with non-zero intensity in the structure factor) are present

Powder patterns are also called "ring or powder pattern"

Angular relations between the atomic planes are lost in powder patterns

Large grains or single crystals produce spot patterns
5) TEM imaging modes: Selected Area Electron Diffraction

Diffraction pattern indexing
- Indexing is used to determine phase and orientation
- Cubic system can be done by hand but more complex crystal structures require simulations and software
- Simulations: Software JEMS (P. Stadelmann), e.g., calculates orientation of a known structure and compares with experimental diffraction pattern.

Phase identification

FIB lamella of ≈ 50 nm thickness, GJS600 treated
Bright Field micrograph, 2750x (Philips CM20)
Simulated diffraction on JEMS software
5) TEM imaging modes: Diffraction Contrast

No aperture  Objective aperture
5) TEM imaging modes: Diffraction Contrast

- **Bright field (BF):** the image is formed with the transmitted beam only.
- **Dark field (DF):** the image is formed with a selected diffracted beam which gives information on regions associated with that diffracted beam, e.g., phase.
- **Two ways to setup a dark-field image**
  1. **Off-axis:** Shift aperture to diffracted beam
  2. **Beam tilt:** Use electromagnetic deflectors to tilt diffracted beam on to optical axis.

![Diagram of TEM imaging modes: Diffraction Contrast](image)

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5) TEM imaging modes: Diffraction Contrast

Structural and Morphological Analysis using Dark-field Images

Which method produces fewer distortions, i.e., has spatial resolution, off-axis or beam tilt method?
5) TEM imaging modes: Diffraction Contrast

Stacking Fault defects (striped contrast) look different. Why??

5) TEM imaging modes: Weak Beam Imaging

- Strong beam Bright-field or Two beam condition: the sample is first tilted such that g image is excited (Ewald sphere intersects $g$) and strong in intensity

- Strong beam Dark field: the image is formed using the excited, high intensity $g$ spot

- Weak Beam Dark-field (WBDF): sample is tilted such that the $3g$ and $0$ spots are both excited and the objective aperture is centered on the WEAK $g$ spot.

- WBDF is useful for imaging defects since the excitation error is small and defects such as dislocations are better resolved
5) TEM imaging modes: Weak Beam Imaging

Stripes spacing in the Stacking Fault correlate to partial dislocation separation distances which can only be seen by weak beam dark-field imaging.
Summary

TEM imaging modes

1. Mass-Thickness contrast
   - Applies for both crystalline and amorphous phase and scales with Z^t

2. Fresnel contrast
   - Developed by the interference of two waves, reference vacuum wave and one which scatters off the edges in the sample causing a path length difference and phase shift, resulting in fringe patterns that depend on the focus setting of the microscope
   - Can be used to correct objective astigmatism

3. Diffraction patterns
   - Can be used to identify phase, orientation and crystal structure and these data can be correlated to the nanostructure features in the TEM image
   - Most calculate the camera constant (camera length for the TEM) in order to precisely index patterns
   - Simulations are needed to index lower symmetry crystals (non-cubic)

4. Diffraction contrast
   - Brightfield: objective aperture centered on "unscattered" beam (spot)
   - Dark-field: objective aperture centered on "diffracted beams (spot)
   - Weak Beam Dark-field: sample tilted to excited higher order "3g" reflections to higher intensity and objective aperture centered on low intensity "1g" spot

5. HR-TEM or Phase contrast
   - Developed by the interference of two waves, one which undergoes a phase shift due to the interactions with the sample crystal structure of a given thickness and is altered by the microscope aberrations
   - Simulations are needed to interpret phase contrast images properly