Electron microscopy and focused ion beam systems

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All unreferenced images and spectra were obtained by Thomas Qureishy with a 200 Quanta FEI FEG SEM on 01.04.2015.
History

- The first electromagnetic lens was created by Hans Busch in 1926 [1]. Until then, magnification was limited by poor resolution in visible light microscopes.

- Ernst Ruska and Max Knoll built the world’s first transmission electron microscope (TEM) in 1931.

- Ruska was given half of the Nobel Prize in Physics in 1986 for his work on electron microscopy.

- Today’s electron microscopes can reach resolutions down to 0.5 Å [2].

Important abbreviations

**TEM:** Transmission electron microscope/microscopy

**SEM:** Scanning electron microscope/microscopy

**STEM:** Scanning transmission electron microscope/microscopy

**FIB:** Focused ion beam
Why electrons?

- For atomic resolution, the waves irradiating the specimen must have wavelengths on the order of distances between atoms in the specimen. Wavelengths of visible light, which is used in visible light microscopes, are too long. Electrons in electron microscopes have much shorter wavelengths than interatomic distances, and therefore provide images with much higher resolution.

- However, resolution in electron microscopes are limited by lens aberrations. The three most common ones are spherical aberration, chromatic aberration and astigmatism. Aberrations may be reduced, compensated for and even used to our advantage!

[Spherical aberration (left) and chromatic aberration (right)](1).

Detectable signals in a TEM

A schematic of detectable signals in a TEM specimen [1].

[1] T. Qureishy (2012), *Synthesis and structural studies of Mg$_2$Si$_{1-x}$Sn$_x*$. Department of Physics, University of Oslo
Diffraction from crystals

- Incident electrons are scattered by the combined electrostatic potential of atomic nuclei and electron clouds in a sample.
- Parallel planes of atoms: Semi-transparent specularly reflecting surfaces. Each plane reflects and transmits some of the incident electron waves.
- Diffraction: constructive interference between waves reflected from adjacent planes. 

**Bragg’s law:**

\[ 2d \sin \theta = n\lambda, \]

where \( d \) is the interplanar spacing, \( \theta \) the semi-angle between incident and reflected waves, \( \lambda \) the wavelength and \( n \) an integer.

Constructive interference occurs when the path difference between waves reflected from adjacent planes, \( 2d\sin\theta \), is equal to an integer multiple of the wavelength, \( n\lambda \) [1].

[1] T. Qureishy (2012), *Synthesis and structural studies of Mg\(_2\)Si\(_{1-x}\)Sn\(_x\)*. Department of Physics, University of Oslo
Reciprocal space

A crystal structure can be represented by a lattice and a motive (also called a basis). Every lattice in real space given by lattice vectors \( \mathbf{a}, \mathbf{b}, \mathbf{c} \) has a reciprocal lattice given by vectors \( \mathbf{a}^*, \mathbf{b}^*, \mathbf{c}^* \).

\[
\begin{align*}
\mathbf{a}^* &= \mathbf{b} \times \mathbf{c} / V \\
\mathbf{b}^* &= \mathbf{c} \times \mathbf{a} / V \\
\mathbf{c}^* &= \mathbf{a} \times \mathbf{b} / V
\end{align*}
\]

where \( V = \mathbf{a} \cdot (\mathbf{b} \times \mathbf{c}) \) is the volume of the unit cell in real space.

Lattice points in reciprocal space are defined by a reciprocal lattice vector:

\[
g = h\mathbf{a}^* + k\mathbf{b}^* + l\mathbf{c}^*,
\]

where \( h, k, l \) are integers called Miller indices.

Coherent waves are scattered by parallel planes into the same point. → A point in reciprocal space corresponds to a set of planes in real space.
Structure factors

Kinematic intensities of the points in a diffraction pattern are proportional to the square of the structure factor of the unit cell. The structure factor, $F$, is found by adding the scattering factors, $f$, of every atom and ion in the unit cell, while taking into account their relative positions.

$$F_{(h,k,l)} = \sum_{j=1}^{n} f_{(j)} \exp[2\pi \cdot i (hx_{(j)} + ky_{(j)} + lz_{(j)})]$$

Where $x$, $y$, $z$ represent the position of atom $j$ with scattering factor $f$, and $h$, $k$, $l$ are the Miller indices of the Bragg reflections [1].

Transmission electron microscopy

Transmission electron microscopes (TEM) are used for:
- Imaging
- Diffraction
- Spectrometry

- Electrons are emitted from an electron gun by thermionic emission or electron tunneling. High acceleration voltages (∼200 kV) are required.
- The electron beam is focused by magnetic lenses and limited by apertures.
- Brightness and shape are controlled by condenser lenses and apertures.
- Electrons travel through the objective lens pre-field.
- Electrons travel through the specimen.
- The objective lens post-field focuses transmitted electrons onto the image plane.
- The image plane or the back focal plane is projected by intermediate lenses and projector lenses onto the viewing screen.

Transmission electron microscopy

- Analysing local nanostructure.
- Defects: impurities, dopants, dislocations, antiphase boundaries, etc.
- Thin specimens are required, preferably < 100 nm thick.
- High vacuums are required.
Ray diagrams showing trajectories of electron beams after transmitting through a specimen in a TEM [1]. The objective aperture and selected area diffraction (SAD) aperture are in the back focal plane and the image plane, respectively. In imaging mode, the image plane is projected onto the viewing screen. In diffraction mode, the diffraction plane is projected onto the viewing screen.

[1] T. Qureishy (2012), *Synthesis and structural studies of Mg$_2$Si$_{1-x}$Sn$_x*$. Department of Physics, University of Oslo
Diffraction techniques

Left: Selected area electron diffraction (SAD or SAED) uses a parallel incident electron beam to form spots in the diffraction plane.

Right: Convergent beam electron diffraction (CBED) uses a converged electron beam to form disks in the diffraction plane [1].

[1] T. Qureishy (2012), *Synthesis and structural studies of Mg$_2$Si$_{1-x}$Sn$_x$.* Department of Physics, University of Oslo
Ewald sphere construction

Radius: \( k = 1/\lambda \)

Bragg condition:
\[ \Delta k = k_D - k_I = g \]

A 2D projection of an Ewald sphere construction. Bragg’s condition is satisfied when the difference between a diffracted wave vector, \( k_D \), and the incident wave vector, \( k_I \), is equal to a reciprocal lattice vector, \( g \) [1].

[1] T. Qureishy (2012), *Synthesis and structural studies of Mg\(_2\)Si\(_{1-x}\)Sn\(_x\).* Department of Physics, University of Oslo
SAD patterns

A [112] diffraction pattern of Mg$_2$Si$_{1-x}$Sn$_x$, with possible superstructure reflexes indexed in yellow [1].

A diffraction pattern of N-containing Mg$_2$Si [1].

[1] T. Qureishy (2012), *Synthesis and structural studies of Mg$_2$Si$_{1-x}$Sn$_x*.* Department of Physics, University of Oslo*
SAD patterns

(a) A bright field image and (b) a SAD pattern of a nanograined ZnO thin film deposited on a sapphire substrate [1].

A ring pattern of polycrystalline Pt [2].

A SAD pattern from a single quasicrystal [3]

Selected area diffraction

\[ Rd = L\lambda \]

\( R \): distance between the direct beam and a diffracted beam

\( d \): distance between crystallographic planes

\( L \): camera length

\( \lambda \): wavelength of electron beam

One can measure interplanar distances in a crystal. The geometry above shows the relation between the camera length, \( L \), and the measured distance between the direct beam and a diffracted beam, \( R \). Since \( R \ll L \), \( Rd = L\lambda \) [1].

[1] T. Qureishy (2012), *Synthesis and structural studies of Mg\(_2\)Si\(_{1-x}\)Sn\(_x\)*. Department of Physics, University of Oslo
A tilt series can be performed to determine the space group of a crystal. The figure shows a tilt series map of SAD patterns of $\text{Mg}_2\text{Si}_{1-x}\text{Sn}_x$ [1].

[1] T. Qureishy (2012), *Synthesis and structural studies of $\text{Mg}_2\text{Si}_{1-x}\text{Sn}_x$*. Department of Physics, University of Oslo
Imaging with diffraction contrast in TEM

The objective aperture in the diffraction plane limits electrons to those that have been diffracted in a certain direction. The result is a bright field image if only the direct beam contributes to the image, whereas a dark field image is formed if one of the diffracted beams passes through the aperture.

A sketch showing how a bright field (left, [1]) and a dark field (right, [2]) image is formed.

a) A bright field image, b) dark field image and c) diffraction pattern from polyethylene glycol coated ultrasmall magnetite nanoparticles [1].

Phase contrast imaging in TEM

Phase contrast is used for imaging at atomic resolution in high resolution transmission electron microscopy (HRTEM).

Parallel incident electron beam, large apertures in the back focal plane, and the image is underfocused.

Intensity is proportional to the square of a wave function, so the phase information is lost. Fortunately, lenses are not perfect.

Through-focus imaging: Phase contrast is converted into amplitude contrast when defocusing is combined with lens aberrations [1].

a) A TEM image and b,c,d) HRTEM images of bimetallic PdAg nanoparticles [1].

Energy dispersive X-ray spectrometry (EDS)

- An incident electron knocks out an electron from a low energy atomic orbital. Another electron from an orbital of higher energy in the same atom falls down to replace it. As a result, an X-ray photon or an Auger electron is emitted.
- The energy of an X-ray photon is equal to the energy difference between the two states of the atom. Each element has its characteristic set of X-ray emission energies.
- Up to single atom resolution [1].

An electron from the K-shell was knocked out by an incident electron. An electron from the L-shell replaces it, thereby lowering the energy of the atom. As a result, an X-ray is emitted [2].

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Element | Wt % | At %
---|---|---
O K | 14.68 | 35.14
SiK | 31.21 | 42.56
NbL | 54.11 | 22.30

[2] [http://www3.nd.edu/~kamatlab/facilities_physchar.html](http://www3.nd.edu/~kamatlab/facilities_physchar.html), downloaded on 05.04.2015
Some other interesting characterization methods in TEM

- Electron holography: The creation of holographic images in TEM.

- Kikuchi lines: Lines in diffraction patterns formed from electrons that have scattered inelastically and then elastically before exiting a TEM specimen.

- Environmental transmission electron microscopy (ETEM): TEM with weak gaseous pressures.

- Energy filtered transmission electron microscopy (EFTEM): TEM where only transmitted electrons within a certain energy range contribute to an image or a diffraction pattern.

- Tomography: 3D imaging.

- Imaging of magnetic vortices by off-axis holography or by Fresnel imaging.
**Single magnetic vortices in TEM**

*(not curriculum)*

**Method number 1:**
Off-axis holography. This method is more sensitive to phase shifts in the electron beam.

*Left:* The experimental setup in off-axis holography. The sample is tilted at an angle $\alpha = 45^\circ$ and the image is out of focus. *Right:* A TEM image with an applied magnetic field of 29.2 G, showing individual magnetic vortices [1].

Single magnetic vortices in TEM
(not curriculum)

**Method number 2:**
Fresnel imaging (also referred to as out of focus imaging). Defocused images are obtained. Vortices appear as black and white features, as in method number 1. This method uses a better conversion of noise. Experimental results can be compared with simulations.

TEM images with defocus **a)** $\Delta f = 1.10 \pm 0.02$ cm and **b)** $\Delta f = -1.10 \pm 0.02$ cm [1].

Scanning electron microscopy
In a scanning electron microscope (SEM), an electron beam is focused onto the surface of the specimen and scanned along the surface in a raster pattern.

A sketch of a SEM column (left, [1]) and a simplified sketch showing electron beam paths through a part of the column (right, [2]).

[2] T. Qureishy (2012), *Synthesis and structural studies of Mg$_2$Si$_{1-x}$Sn$_x$*. Department of Physics, University of Oslo
Scanning electron microscopy

- Lower acceleration voltages used in SEM (≈ 10 kV) compared to those in TEM (≈ 200kV).

- High vacuums are required.

- Samples should have conductive surfaces to avoid electron charging on the surface. Nonconductive surfaces can be covered by a thin metallic layer. Alternatively, low vacuum mode may be used, where there is a weak gaseous pressure of water in the SEM.

- Bulk ceramic and intermetallic specimens should be ground and polished.
Advantages with SEM

- High resolution.
- High depth of field and depth of focus.
- Non-destructive method.
- Easy to prepare samples.
- Allows analysis of large samples.

An image of a radiolarian obtained in a visible light microscope (left) and a SEM (right) [1]. The whole SEM image is in focus, because of the large depth of focus.

Emission of detectable signals

A schematic of emission volume for detectable signals in SEM [1]. The shape depends on the acceleration voltage of the incident beam and the atomic number of the atoms in the sample.

Emission of detectable signals

The higher the acceleration voltage and the lower the average atomic number, the deeper the emission of signals.

Simulations of volume interaction vs accelerating voltage. The blue tracks represent electron trajectories that finish in the specimen, while the red tracks represent electrons that have escaped as backscattered electrons [1].

Secondary electrons (SE)

- Created by inelastic scattering events: An incident electron knocks out an electron from an outer orbital of an atom in the sample. If the electron that has been knocked out exits the sample and reaches the SE detector, it contributes to the SE image.

- There are more SEs than incident electrons.

- SEs are generated near the surface, so they provide information about the sample’s topography and morphology.

- SEs are created in small areas. Therefore, they provide images with high resolution in SEM.

- If the SE was knocked out by an electron in the incident beam, it’s called an SE1. If it was knocked by a backscattered electron that has returned to the surface, it’s an SE2.
Secondary electrons (SE)

The image shows that electrons entering edges have a higher chance of exiting the sample and hence reaching the detector [1]. Therefore, edges appear bright and flat areas appear dark, providing us with information about the surface’s topography and morphology.

SE images

Metal organic framework crystals [1].

ZnO nanoneedles [2].

SE images of a wasp: a) A low magnification image of the head and thorax, b) an eye, and higher magnification images of a part of c) an eye, d) the abdomen, e) a wing, and f) a leg.
SE images of diatoms
Backscattered electrons (BSE)

- Created by elastic scattering: Incident electrons that have been scattered by electrostatic interactions with atomic nuclei in the specimen.

- If a BSE is scattered by almost 180 °, exits the sample and reaches the BSE detector, it contributes to the BSE image.

- If a BSE was scattered by one atomic nuclei, it’s a BSE1. If it was scattered by several nuclei, it’s a BSE2.

- BSE mainly provide information about average atomic composition. Z-contrast: The higher the average atomic number, the heavier the average atomic nuclei, the stronger the scattering, and hence the brighter the part of the image appears.
A BSE image of a specimen containing Mg, Si and Sn [1]. There are at least seven different phases with different compositions, numbered from 1 to 7.

[1] T. Qureishy (2012), Synthesis and structural studies of $\text{Mg}_2\text{Si}_{1-x}\text{Sn}_x$. Department of Physics, University of Oslo
BSE images of an electronic circuit
Energy dispersive spectrometry

- EDS is used for analysing the chemistry in specimens.

- Same principal as in TEM.

- TEM specimens are thinner than SEM specimens, so SEM samples have larger X-ray emission volumes. As a result, EDS in SEM has higher X-ray counts, but lower spatial resolution.

- X-ray emission volume is much larger than SE and BSE emissions in SEM. Therefore, the spatial resolution of emitted X-rays is poor compared to the resolution of SE and BSE emission.

- In SEM, one can quantitatively analyse spots and lines (line scans) and qualitatively characterize large areas (EDS mapping).
EDS maps

Elemental maps of a sample containing Mg, Si and Sn [1]. The greyscale image is a BSE image.

[1] T. Qureishy (2012), *Synthesis and structural studies of Mg$_2$Si$_{1-x}$Sn$_x$*. Department of Physics, University of Oslo
Layered samples

- If two areas of the same phase with equal brightness in BSE images have different atomic compositions according to EDS analyses, one of the areas could be a thin layer covering another phase. X-rays are created deeper into the sample than BSEs.
- One can perform EDS at different incident electron beam strengths (acceleration voltages) to investigate layered samples.

EDS spot analyses indicate that the area in the red circle in the image of the electronic circuit above contains Si, O and Nb. The composition varies with the acceleration voltage of the electron beam.
Some other interesting characterization methods in SEM

- Environmental scanning electron microscopy (ESEM): SEM with gases with low pressure in the specimen chamber.

- Electron backscatter diffraction (EBSD): Diffraction patterns are collected from BSEs over the scanned area, resulting in an overview of crystallographic orientations of crystals in the specimen.

- Electron beam induced current (EBIC): Used for viewing junctions and defects under a layer in semiconductors, and for analysing minority charge carrier properties [1].

SEM lab demonstrations

- We will visit the Centre for Materials Science and Nanotechnology.

- I will demonstrate how the 200 Quanta FEI FEG SEM works. Then you will all get a chance to use it yourselves.

- Bring your own samples if there is anything you want to analyse.

- At the end, I will show some other instruments in the area.
Images obtained by the students in 2015

Butterfly wing

Titania crystals

Superconducting microchip (above) and MgB$_2$-Ni alloy (below)
Images obtained by last year’s students

Upper-left image: TiO$_2$ with Pd.
All other images: Parts from a flower. The upper image in the middle was edited by Pavlo Mikheenko.
Scanning transmission electron microscopy (STEM)

- Combines TEM and SEM.
- Bright field (BF) imaging.
- Annular dark field (ADF) imaging: Elastically scattered electrons.
- High angle annular dark field imaging (HAADF): Electrons that are elastically scattered through large angles. Z-contrast similar to BSE images in SEM.
- EDS: Sub-Ångström resolution in TEM-based STEM [1].
- Wavelength dispersive spectrometry (WDS).
- Electron energy loss spectrometry (EELS).


A schematic showing the positions of BF, ADF and HAADF detectors [2].
Scanning transmission electron microscopy (STEM)

A bright field (left) and a dark field (right) STEM image of multiwalled carbon nanotubes containing Ag nanoparticles [1].

EELS

- Used in TEM and STEM.
- Measures the energy loss of transmitted electrons. Transmitted electrons enter a magnetic prism at the bottom of the STEM. The higher the energy of the electrons, the faster they travel and the more their path will be deviated by the magnetic field. They are subsequently sorted into a spectrum.
- Gives information about elemental identity, chemical bonds, electronic properties of the valence and conduction band, surface properties and pair distance distributions of specific elements [1].

From Ref. [2].

The formation of an EELS spectrum and an energy filtered TEM image [3].

Focused ion beam systems

- A focused ion beam (FIB) microscope is similar to a SEM, but irradiates the specimen with ions (usually Ga\(^{+}\)) instead of electrons.
- The source is a liquid metal ion source (LMIS).
- A FIB can be used for imaging, analysis and specimen modification.
- Incident ions remove atoms, ions and molecules from the specimen. Secondary ions and secondary electrons may be used for imaging. Secondary ions may be used for characterising the composition.
- Sputtering: The ion beam is used for milling the surface of the specimen.
- Gases can be used to aid material deposition.
- Generally, a high current to spot size ratio is desired. Small currents and spot sizes are used for high resolution imaging. Higher currents are used for analysis. A range of currents are used for sample modification.

A sketch of a FIB microscope [1].

A schematic of a dual beam system. An ion beam and an electron beam intersect at 52 ° relative to each other near the surface of the specimen. This enables immediate SEM imaging of milled surfaces [1].

Further reading

FEI Company, *Introduction to electron microscopy*, 2010


Questions

- Why are electron beams used instead of visible light as in visible light microscopes?
- What are the main principles behind TEM, and what is TEM used for?
- Draw electron beam paths exiting a TEM sample, then going through the objective lens, and finally reaching the first intermediate or projection lens. Where is the diffraction plane, and where is the image plane? Which apertures are used in those planes?
- What are diffraction patterns used for?
- What are the main limitations for resolution in TEM images?
- Describe different kinds of lens aberrations.
- What are the main principles behind SEM, and what is SEM used for?
- How do incident electrons interact with the sample in SEM, and what kind of signals are detected? What do the signals tell us about the specimen?
- Two spots within the same phase have equal brightness in BSE images, but may have different atomic compositions according to EDS analyses. Why may this be the case?
- What are the main principles behind STEM, and kind of signals can be detected in STEM?
- Describe EDS. What are the main differences in quality of EDS results in TEM and SEM?
- Describe how EELS is carried out, and what it is used for.
- What are the main principles behind FIB, and what is FIB used for?
- What is the advantage of a dual beam system?
- If your Master’s or PhD project involves characterization of materials, how could you use electron microscopy or FIB in your particular project?