Electron microscopy

Most widely used nanoscale microscopy. Based on possibility to create bright electron beam with sub-nm spot size.
History: Ernst Ruska (1931), Nobel Prize (1986)
For visible light λ=400-700nm, for electrons de Broglie wavelength (1927) is:

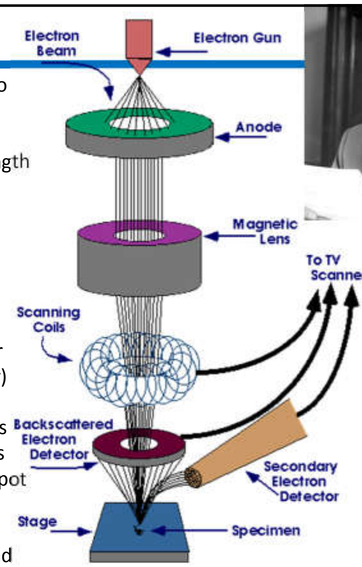
$$\lambda = \frac{h}{\sqrt{meV_0}}$$ if V_0 = 5kV → λ≈0.2Å

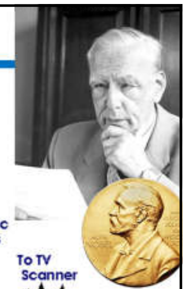
Types

Scanning electron microscopy (SEM) (standard res. 1nm)
Transmission e microscopy (TEM) (resolution sub 50pm)

The SEM System

- Creation of e beam: thermionic emission from a filament or field emission from a sharp tip (cold cathode or heated emitter)
- Acceleration to a desired beam energy from 0.2kV – 300kV
- Beam passes through magnetic condensor lenses, apertures in vacuum system and focused by magnetic objective lenses SEM objectives are close to sample → focusing to sub nm spot
- electrons hitting the surface generate secondary electrons, which are detected with (scintillator/photomultiplier) or backscattered electrons are detected with a detector around the electron beam line.
- scanning the surface in x,y direction by scanning coils the secondary/backscattered electron signal is recorded.
- It workes in vacuum, (Special systems also in low vacuum ~mbar →biology)





(Right) Ernst Ruska, who received the Nobel Prize 1986 for his fundamental work in electron optics, and for the design of the first electron microscope, (Left) Basic setup of a SEM. Electrons are generated by a gun and accelerated via an anode by dc voltage. The e/beam is focused to small spot size by magnetic lenses, its direction is oriented by scanning coils. The electrons hitting the surface either backscatter or generate secondary electrons, which are detected. https://www.purdue.edu/ehps/rem/rs/sem.htm

Nattelson Section 4.1.3

Electron microscopy

Beam generation

Thermionic emission gun:

Heated filament coated with a low workfunction material, heated to ~1000K. Electrons are thermally excited out of the metal. They accelerated away by anode with high positive voltage. Current density:

$$J = \frac{4\pi me}{h^3}(k_B T)^2 \exp\left(-\frac{\phi}{k_B T}\right)$$

Simple, but emitted electrons have a broad energy spectrum.
→Role of chromatic aberration

Field emission gun (FEG):

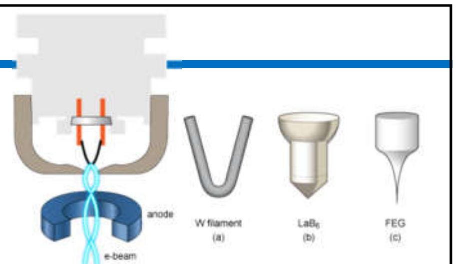
extremely sharp tip to generate very high local electric fields. Assuming the end of the tip as a sphere with radius a, the electric field at the surface:

$$E = \frac{1}{4\pi\varepsilon_0}\frac{Q}{a^2} = \frac{V}{a}$$

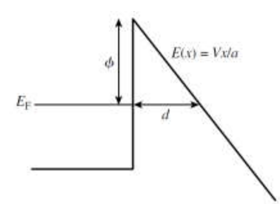
E.g. a=100nm, V=1kV → E= 10^{10}V/m. Thus if ϕ=2eV the width of tunnel barrier tilted by E is 2Å. Electrons could tunnel through the tilted potential barrier. This is the problem of Fowler–Nordheim tunneling:

$$J \propto E^2 \exp -\left(\frac{4\sqrt{2m}}{3e\hbar}\frac{\phi^{3/2}}{E}\right)$$

Large electron current without heating → monoenergetic e beam. There are cold cathodes or thermally assisted Schottky type.



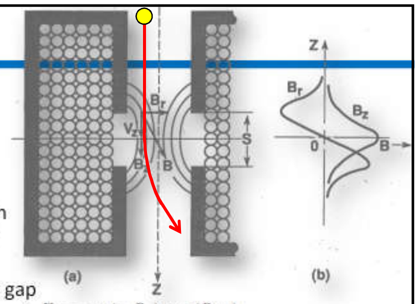
(Up) Structure of the gun. A W filament (thermionic emission) or a sharp tip (for FEG) is surrounded by the Wehnelt cylinder. The tungsten filament is heated by passing current between its ends. Below the cap sits an anode, which, being positive, attracts the electrons away from the filament. http://www.ammrf.org.au/myscope/sem/practice/principles/gun.php



(Up) The variation of potential with distance perpendicular to the surface of a metal. Inside the metal (left) electrons at the Fermi energy are with energy Φ (the work function) below the energy of free electrons. The application of a large electric field, V/a, generates a small tunneling barrier (width d) for electrons at the Fermi energy to escape through.

Lindsay Section 4. 3

Electron microscopy



Electron optics

Analogy to geometrical optics: electron trajectory as a *ray* path
For a microscope a convex lens is needed to focus the beam.

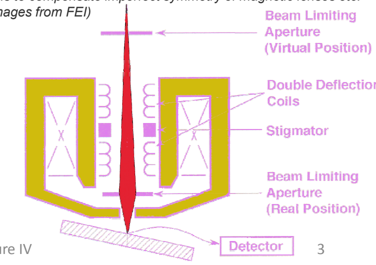
Magnetic lens

Principle: Use B field induced **Lorenz force**.
Electron beam is guided along the axis of a current-carrying coil, which is enclosed by ferromagnetic material such as soft iron (black). Ferro-magnet is required to increase the radial component of B. Due to high permeabilty iron carries most of the flux lines. However there is a gap without magnetic material → flux lines appear within the internal bore of the lens. (see Fig. a) The lens has cylindrical symmetry, with z dependent B_z and B_r components (see Fig. b).
As e approaches from top dominant Lorenz force component is tangential ~$v_z B_r$ → **e starts to spiral** in the field. → v_ϕ increases → **Radial force** F_r is generated → It attracts electron towards z axis.
It is a focusing action, non-uniform B field acts similar to convex lens.
It has a focal distance, f, which can be changed by coil current.

$$F_\phi = -e(v_z B_r) + e(B_z v_r)$$
$$F_r = -e(v_\phi B_z)$$
$$F_z = e(v_\phi B_r)$$

(Up) Structure of magnetic lens (a) and its B field components (b).

(Up) Lorenz force in cylindrical coordinate system.
(Down) Pinhole type objective lens design. This objective focuses the e beam on the surface of the sample. It contains an aperture to reduce beam size. Coils to deflect the beam for scanning. Stigmator coils to compensate imperfect symmetry of magnetic lenses etc. (Images from FEI)



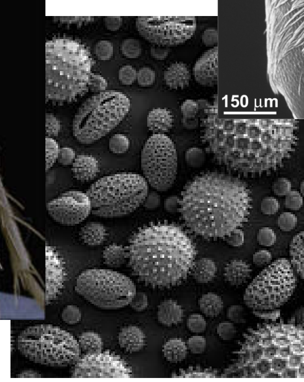
Typical SEM has 2 condenser lenses and an objective lens.
High/low objective strength → short/high working distance, short/high dept-of-field, high/low resolution SEM resolution to ~1nm

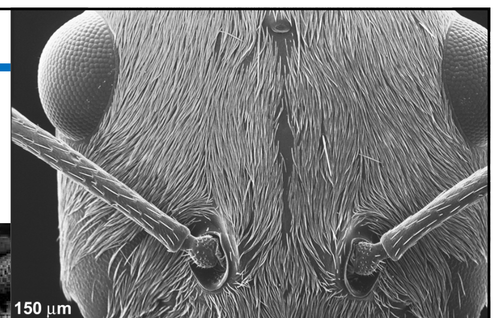
Comparing to high magnification optical microscopes the focal length of objective is much higher → large dept of field in low magnification.

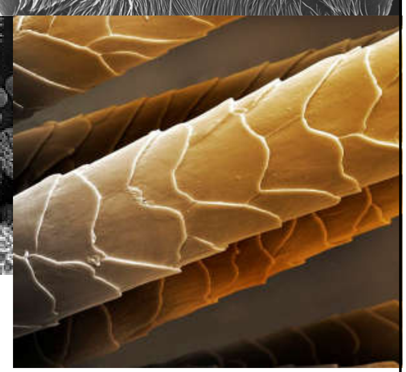
Nattelson Section 4.1.3 also http://www.springer.com/cda/content/document/cda_downloaddocument/9780387258003-c2.pdf?SGWID=0-0-45-280920-p47261066

Electron microscopy

Example of large dept-of-field







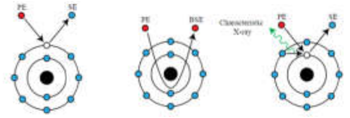
Avoid charging: sample surface should conduct a bit
Surface charges from residual gas could be sufficient.
If not, coating with thin gold layer (~10nm).

Nattelson Section 4.1.3 also http://www.springer.com/cda/content/document/cda_downloaddocument/9780387258003-c2.pdf?SGWID=0-0-45-280920-p47261066

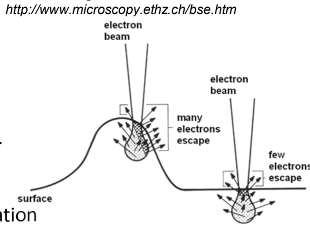
Electron microscopy

SEM

Accelaration voltage (V_{acc}): 1-30keV
Spatial resolution is limited not by diffraction rather on the achieved spot size (non ideal focusing) and interaction volume.
Advantage: large magnification range (from 10 above 500k) is possible with large dept of field, bulk samples also, analytical techniques to study composition



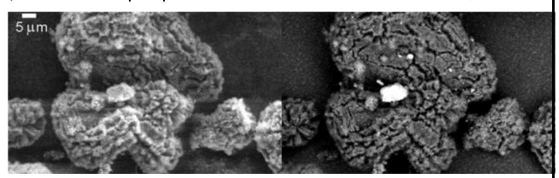
(Up) – From left to right: Mechanism of secondary electron generation, electron backscattering and X-ray emission when electrons relax to empty core state. (Down) Edge effects: more secondary electron can leave the sample at edges leading to increased brightness, which helps to get good contrast in surface topography. Figure also shows the interaction volume, where primary electrons penetrate and backscattered electrons are generated. http://www.microscopy.ethz.ch/bse.htm



Detectors

Secondary electron detector (SE):
As electron beam hits the surface and electrons are kicked out from lower shells and detected.
- Heavy elements are more effective at producing secondary electrons. E.g. large contrast of Au on Si.
- From very close to the specimen surface → high resolution image of the surface. Highest resolution: 0.4nm (2009)
- Nice contrast of edges in surface topography due to many escaping electrons
- Higher V_{acc} -> shorter e travel time → better resolution, but also deeper penetration

Backscattered electron imaging (BSE): Electrons of the beam reflected elastically. Signal from deeper location → lower spatial resolution. It strongly depend on Z. → Contrast between areas with different composition.



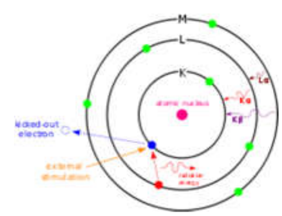
(Right) SEM images of Fe particles in carbon obtained with secondary electrons (left) and back-scattered electrons (right). The BSE image shows the Fe particles with bright contrast. http://www.microscopy.ethz.ch/bse.htm

Nattelson Section 4.1.3 also Wikipedia

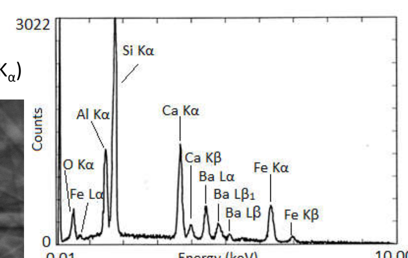
Electron microscopy

SEM

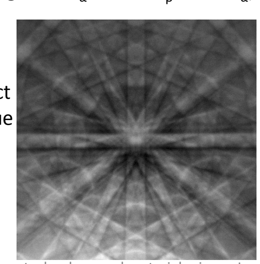
Energy-dispersive X-ray spectroscopy (EDS)
If the incident electron beam has sufficient energy to knock core electrons out of the sample. Higher energy electrons from outer shell fills the hole and the sample then fluoresces in the x-ray, and the resulting radiation is measured by an energy-dispersive spectrometer.
It is very useful for elementary analysis.
Each element has a characteristic set of peaks on its electromagnetic emission spectrum, it can be used for analyzing composition of the specimen.
+ Good for fast check of elementary composition. Qualitative composition can be estimated based on peak-height ratios.
- Elements could have overlapping peaks (e.g. Mn-K_{α} and Cr-K_{β}, or Ti-K_{α})
- Not good for light elements



(Up) Principle of the underlying process of EDS
(Down) EDS spectrum of NIST K309 glass. Silicon, aluminum, barium, calcium, iron, and oxygen are identifiable in the spectrum. J. Goldstein, et. Al, , Scanning Electron Microscopy and X-ray Microanalysis, 3rd, Springer, New York (2003).



Electron backscatter diffraction (EBD)
Backscattered electrons which Bragg diffract from the sample. Characterization technique to study any crystalline or polycrystalline material. Revealing texture, defects, grain morphology and deformation.



(Left) Electron backscatter diffraction pattern of monocrystalline silicon, taken at 20 kV with a field-emission electron source

Nattelson Section 4.1.3 also Wikipedia

Electron microscopy

TEM

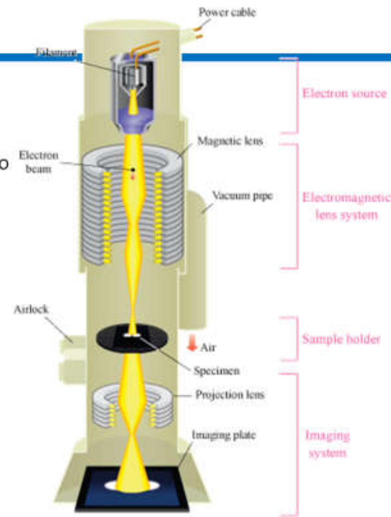
Accelaration voltage (V_{acc}): 100-300keV
Spatial resolution is ≈ 40pm at V_{acc}=200kV in "aberration-corrected" microscopes (spherical aberration is corrected to 5th order). Possible to image lighter atoms like lithium

TEM System

- Thin specimen (~100nm) is placed in the path of the e beam.
- Electrons emitted from the filament are accelerated to ~100keV
- Primary beam formation with condenser lens, then objective lens focus the beam to the sample
- There is interaction with the specimen while electron beam transmit through.
- The projector lens behind the sample expands the beam to the detector (e.g. CCDs).
- Imaging system is e.g. a YAG screen coupled to CCD or phosphor screen.

Specimen stage: Metal grid with a size of ~5 mm, with a thickness and mesh size ranging from a few to 100 µm.

Limitations of TEM: Fabrication of thin samples is challenging (sometimes invasive). Small field of view. Large e flux can damage the sample.



(Up) The main parts of a TEM microscope.
http://www.hk-phy.org/atomic_world/tem/tem02_e.html
(Right) Tipical TEM grid to hold the sample, this one is coated with carbon, which has holes with size in a wide range. It is good to suspend nanoobjects, virus etc.
https://emresolutions.com/tem-products/support-films/

Nattelson Section 4.1.3 and Wikipedia

Electron microscopy

TEM Imaging modes

For small magnification contrast is due to absorption (thickness, composition) for large ones wave nature of electrons (its phase) dominates.

Bright field imaging Dark regions indicating occlusion (scattering or absorption) of the incident beam. Direct transmitted electrons.

Dark field image Simple transmitted electrons are not detected, but diffracted incident beam is measured. It is powerful to analyze the **crystal structures of solids**.

At 200kV range v of electron 70% of speed of light, thus relativistic expression of energy:

$$\lambda = \frac{h}{\sqrt{2m_0 eU}} \frac{1}{\sqrt{1 + \frac{eU}{2m_0 c^2}}}$$

for 200kV λ≈2.5pm.

Intensity of the diffracted beam:

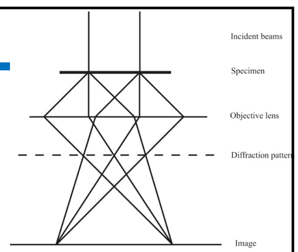
$$I_{\mathbf{g}} = |\psi_{\mathbf{g}}|^2 \propto |F_{\mathbf{g}}|^2 \qquad F_{\mathbf{g}} = \sum f_i e^{-2\pi i \mathbf{g}\cdot\mathbf{r}_i}$$

where F_g is the structure factor and ***g*** is the scattering vector.

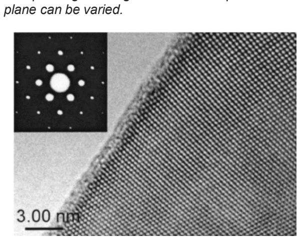
Advantages of electron diffraction

- Wave length is smaller than for X-ray (100pm).
- Geometry of diffraction experiment can be varied by electron lenses.
- Diffraction on nanoscale single crystal is possible
- Can be combined with direct image of the crystal, EDS, EELS or electron holography.

Good for symmetry determination, not so accurate to get lattice parameters

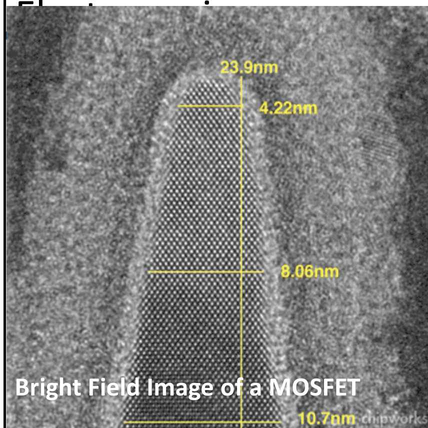


(Up) The objective lens acts to collect all electrons scattered from one point of the sample in one point on the fluorescent screen, resulting an image of the sample.. Note that at the dashed line in the figure, electrons scattered in the same direction by the sample are collected into a single point. This is the back focal plane., and is where the diffraction pattern is formed. By manipulating the magnetic lenses the position of focal plane can be varied.

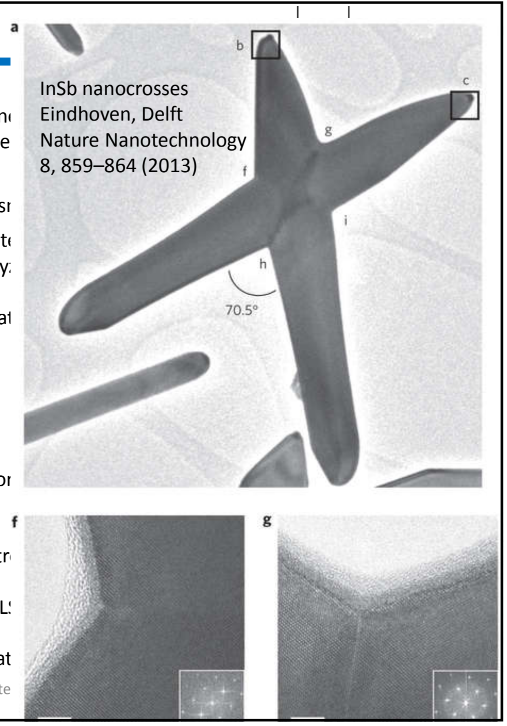


(Up) High-resolution TEM image of a 22 nm single crystal Si nanowire. The inset is a selected area electron diffraction pattern of the nanowire.
Applied Physics Letters 83, 2934 (2003)

Nattelson Section 4.1.3 also Wikipedia



rption (thickn…
ase) dominate
occlusion
. Direct transı
ns are not det…
verful to analy…
ight, thus rela…



InSb nanocrosses
Eindhoven, Delft
Nature Nanotechnology
8, 859–864 (2013)

where Fg is the structure factor and g is the scattering vector

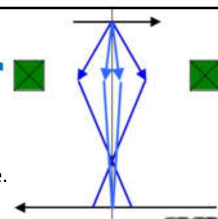
Advantages of electron diffraction

- Wave length is smaller than for X-ray (100pm).
- Geometry of diffraction experiment can be varied by electr…
- Diffraction on nanoscale single crystal is possible
- Can be combined with direct image of the crystal, EDS, EEL… holography.

Good for symmetry determination, not so accurate to get lat…

Nattelson Section 4.1.3 also Wikipedia

Electron microscopy



(Left) Spherical aberration: The focal point of the ray depends on diffraction angle of the beam.
(Down) Example how the interference process effect the HRTEM images as defocus is changed for Si3N4 (0001) sample.
https://nanohub.org/resources/4020/download/2008.02.06-mse582-l11.pdf

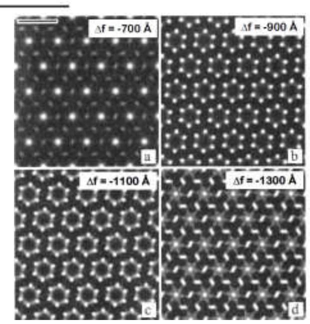
TEM Imaging modes

Phase contrast image (High Resolution TEM)

It is based on interference between electrons transmitted and diffracted by the sample. → sub-A level of resolution is possible.
Interference generates a complex pattern → Extensive image simulations is required.
Complications:

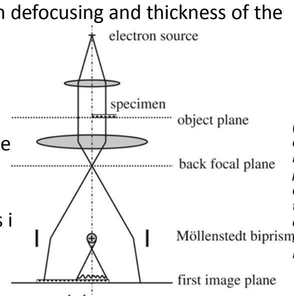
- Phase shift via optical lens changes with distance from optical axis.
- Non-ideal lenses: E.g. spherical aberration adds extra phase shift to diffracted waves. It also has wavelength dependence (chromatic aberration). … →

Phase contrast transfer function of the setup describes the relation between wave exiting from the sample and wave arriving to the image plane. Highly non-linear function. → Complicated image interpretation.
Interference pattern strongly depends on defocusing and thickness of the sample.



Electron holography

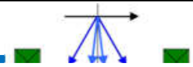
Advantages: it allows the reconstruction of the complete electron wave, i.e. phase and amplitude as well.
- Their separate analysis is possible.
- Posterior correction e.g. for aberrations i… is possible.



(Left) Setup for recording image-plane off-axis holograms. The object covering only half the object plane and the adjacent empty area are illuminated coherently. The object plane is imaged in the first image plane. By means of the electron biprism, the image wave and the adjacent empty wave are superimposed at an angle ('off-axis'). In the image plane, an interference pattern ('hologram') arises, consisting of parallel fringes modulated in contrast and fringe spacing by image amplitude and phase, respectively. H. Lichte et al. DOI: 10.1098/rsta.2009.0126

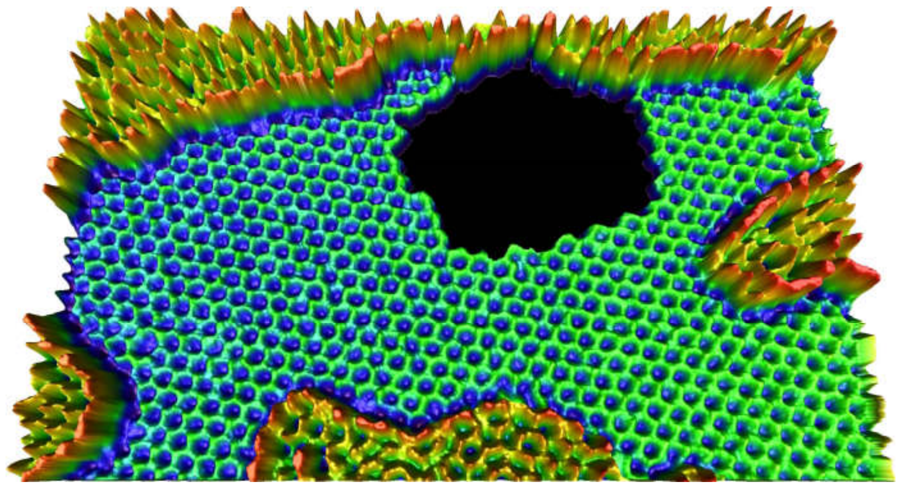
Nattelson Section 4.1.3 also Wikipedia

Electron microscopy

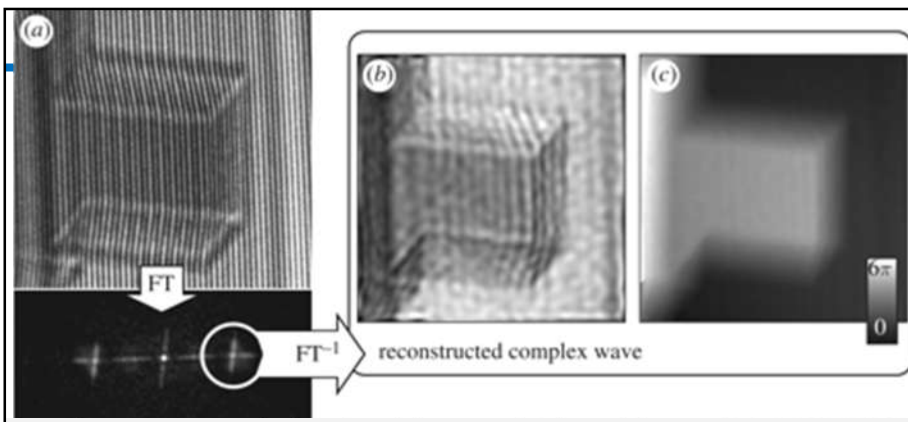


(Left) Spherical aberration: The focal point of the ray depends on diffraction angle of the beam

https://www.youtube.com/watch?v=EogdalfXF4c

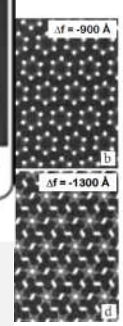


Transmission Electron Aberration-corrected Microscope (TEAM). It has 0.5A resolution
3D rendering of a graphene hole imaged on TEAM 0.5 shows that the carbon atoms along the edge assume either a zigzag or an armchair configuration. The zigzag is the more stable configuration and shows promise for future spintronic technologies.



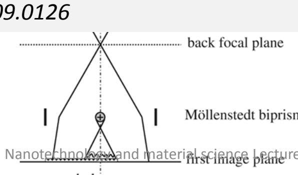
Hologram of an MgO crystal and reconstructed object (*b*) amplitude and (*c*) phase. The complex object wave is retrieved by cutting out one sideband of the hologram spectrum and applying an inverse Fourier transform (FT^{-1}). Afterwards, the reconstructed complex wave can be evaluated in amplitude and phase separately and quantitatively.
H. Lichte et al. DOI: 10.1098/rsta.2009.0126

berration: The focal
epends on diffraction
n.
how teh
ess effect the
s defocus is
4 (0001) sample.
rg/resources/4020/do
5-mse582-l11.pdf



is holograms. The objec
djacent empty area are
naged in the first image
image wave and the
adjacent empty wave are superimposed at an angle ('off-axis'). In the image plane, an interference pattern ('hologram') arises, consisting of parallel fringes modulated in contrast and fringe spacing by image amplitude and phase, respectively. H. Lichte et al. DOI: 10.1098/rsta.2009.0126

and amplitude as well.
Aberration corrected TEM helps in its performance.

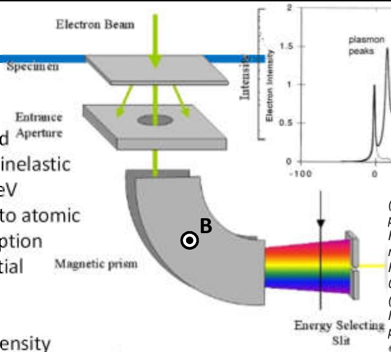


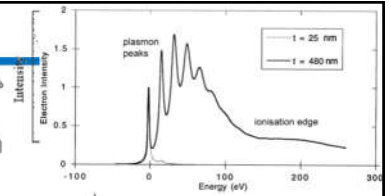
Nattelson Section 4.1.3 also Wikipedia

Electron microscopy

TEM Imaging modes

Electron energy loss spectroscopy (EELS)
precisely measuring the energies of transmitted electrons, it is possible to identify excitations (inelastic processes) even with energy resolution of 0.1eV (typical 1eV) and with spatial resolution down to atomic level. (Clear advantage compare to x-ray absorption spectroscopy (XAS), where using cyclotron spatial resolution is ~30nm).
Good for local properties: band gap, chemical composition, thickness, plasmons, electronic density states.

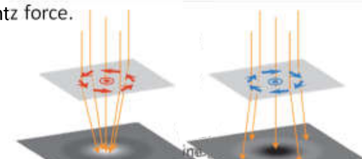


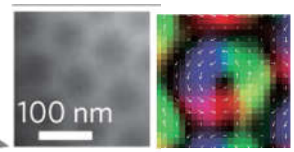


(Up) EELS spectrum of plasmon peak in Si. For tow thicknesses (t). For thicker sample peak appears at multiples of plasmon energy. RF Egeerton Rep. Prog. Phys. 72 016502 (2009)
(Left) EELS setup attached to TEM. Magnetic prism is used with perpedicular B field to separate electrons with different energy. http://www-hrem.msm.cam.ac.uk/research/EFTEM/EFTEM.html

Lorentz TEM
Principle: electron beam passing through an area with a component in magnetic induction perpendicular to its trajectory will be deflected by the Lorentz force.
Induction either from magnetization in the sample itself or stray field exterior of the sample. It allows to detect local magnetic property of the sample.
E.g. magnetic domains, skyrmions.

(Left) Ordering of spins in one class of skyrmions. (Left down) How skyrmions influence the electron beam of TEM for different helicity.





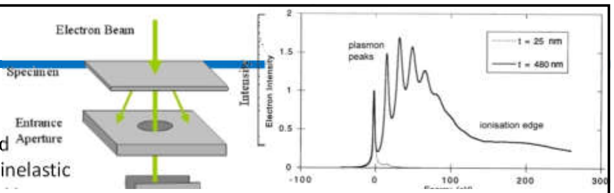
(Up Left) Overfocused Lorentz images of skyrmions in FeGe (Up Right) Corresponding lateral-magnetization distribution map zoomed on one skyrmion
Nature Materials 10, 106–109 (2011) Nature Nanotechnology 8, 723–728

Nattelson Section 4.1.3 also Wikipedia

Electron microscopy

TEM Imaging modes

Electron energy loss spectroscopy (EELS)
precisely measuring the energies of transmitted electrons, it is possible to identify excitations (inelastic



Rep. Prog. Phys. **72** (2009) 016502 R F Egerton

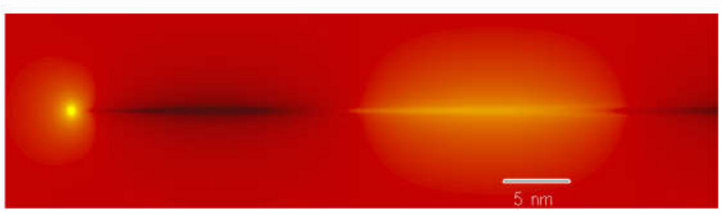


Figure 5. Plasmon wake of a 100 keV electron travelling through aluminium, calculated from the dielectric properties (P E Batson, personal communication). The electron is represented by the bright dot on the left; alternate dark and bright bands represent positive and negative regions of space charge that trail behind the electron.

distribution map zoomed on one skyrmion
Nature Materials 10, 106–109 (2011) Nature Nanotechnology 8, 723–728

Nattelson Section 4.1.3 also Wikipedia