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 non-relativistic electrons the de Broglie wavelength (1927) is $\lambda = \frac{h}{p} = \frac{h}{\sqrt{2me\ {}_0}}$.
If V_0 = 5 kV → $v = 4 \cdot 10^7$ m/s and λ≈0.2 Å

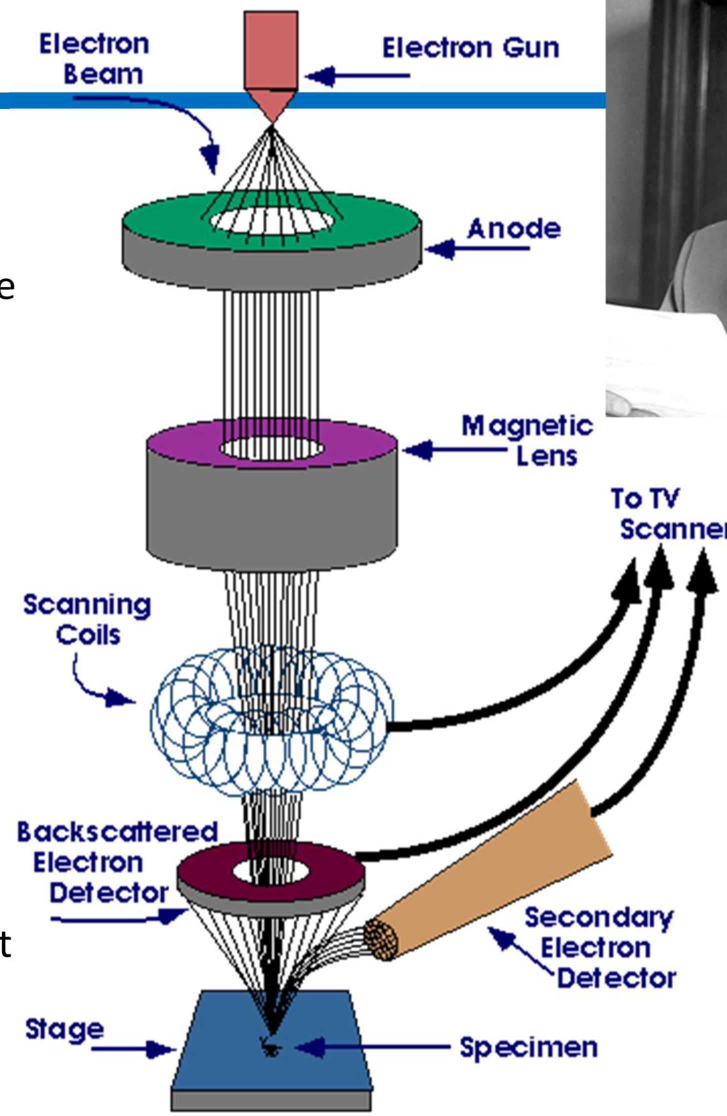
Types

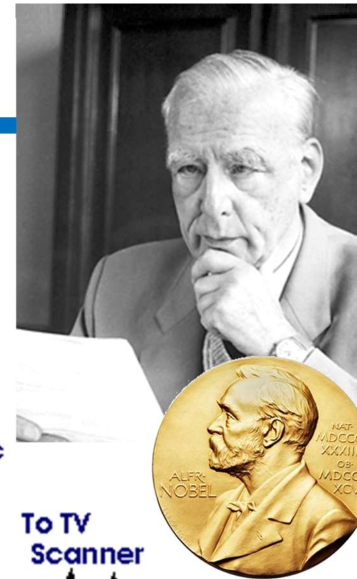
- **Scanning electron microscopy (SEM)** (standard res. 1 nm)
- **Transmission e microscopy (TEM)** (resolution sub 50 pm)

The SEM System

- Creation of e beam: thermionic emission from a filament or field emission from a sharp tip (heated emitter or cold cathode)
- Acceleration to a desired beam energy from 0.2 kV – 300 kV
- Beam passes through magnetic condenser 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 sample surface generate secondary electrons (detected with scintillator/photomultiplier) or backscattered electrons (detector around the electron beam line) → intensity of 1 pixel in the generated image
- Move the beam on the surface in x,y dir. by scanning coils, and the secondary/backscattered electron signal is recorded → full image (intensity vs position)
- works in vacuum (special systems for biology in low vacuum, ~mbar)

Nattelson Section 4.1.3





(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 to and through 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

Electron microscopy

Beam generation

Thermionic emission gun:

Heated filament coated with a low work function material, heated to ~1000 K. Electrons are thermally excited out of the metal. They accelerate away by anode with high positive voltage. Current density (thermionic/Schottky emission):

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

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

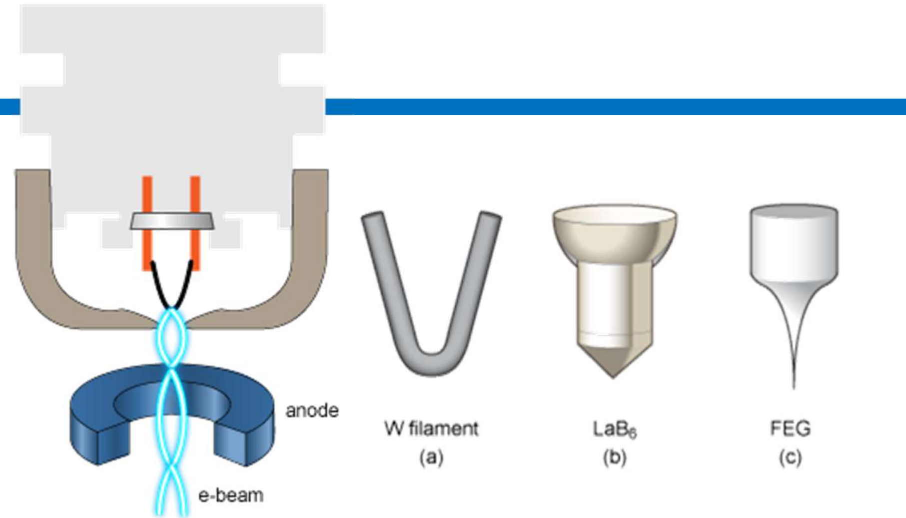
Field emission gun (FEG):

Electrostatic tip effect → use extremely sharp tip with voltage bias (top right, anode not shown) 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 is $F = \frac{1}{4\pi\varepsilon_0}\frac{Q}{a^2} = \frac{V}{a}$

E.g. a=100 nm, V=1 kV → F= 10^{10} V/m. Thus if the work function is ϕ=2 eV (distance from E_F to vacuum level), the F field tilts the vacuum level $E(x)$ so much that the width of tunnel barrier is 2 Å. Electrons could tunnel through the tilted potential barrier. This is the regime of Fowler–Nordheim tunneling: current is

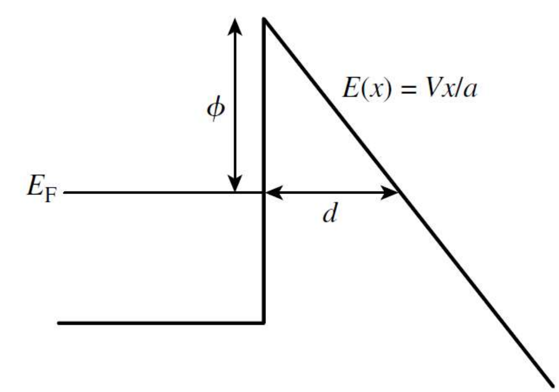
$$J \propto F^2 \exp\left(-\frac{4\sqrt{2m}}{3e\hbar}\frac{\phi^{3/2}}{F}\right)$$

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 (black line) 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



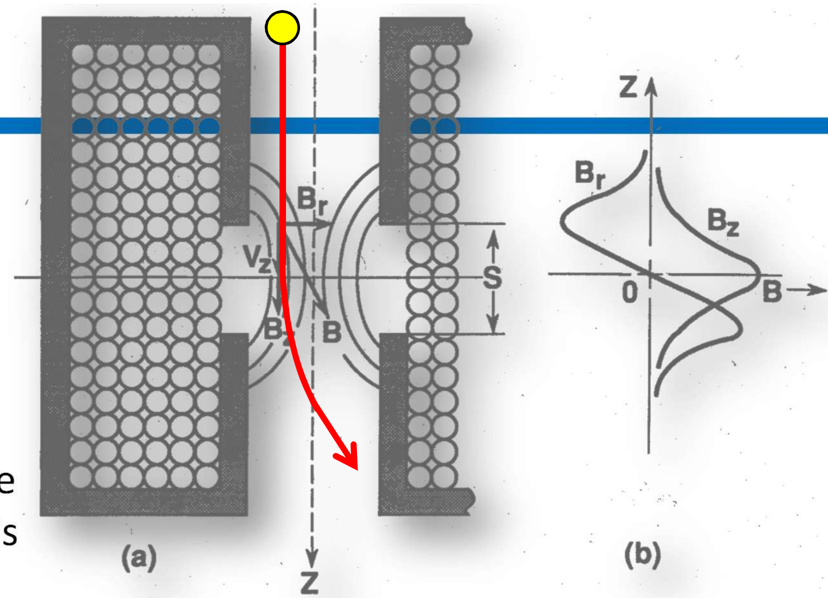
(Up) FEG: the variation of potential with distance perpendicular to the surface of a metal. Inside the metal (left) electrons at the Fermi energy are with energy Φ (the work function) below the energy of free electrons. The application of a large electric field, V/a, makes the tunneling barrier narrow (width d) for electrons at the Fermi energy to escape through. (Consider: $d \to \infty$ if $V \propto F = 0$)

Lindsay Section 4. 3

Electron microscopy

Electron optics

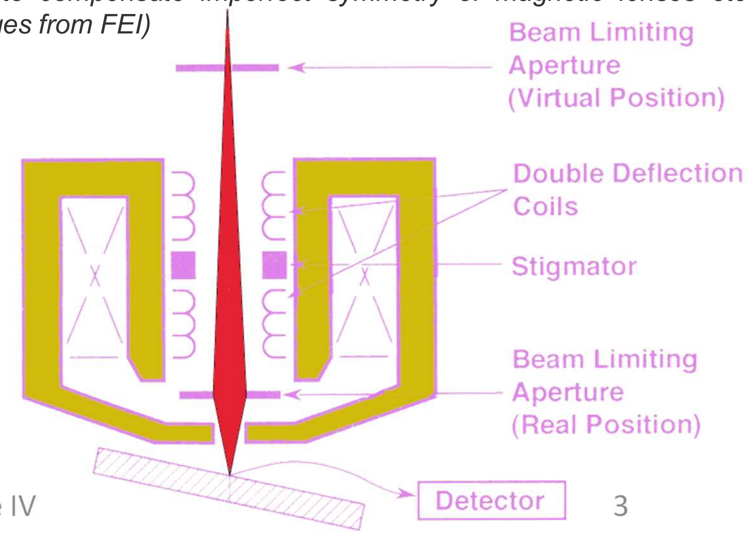
Analogy to geometrical optics: electron trajectory as a *ray* path.
For simplicity, consider a wide, parallel beam of electrons. For a microscope a convex lens is needed to focus the beam to a small spot.



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

(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)



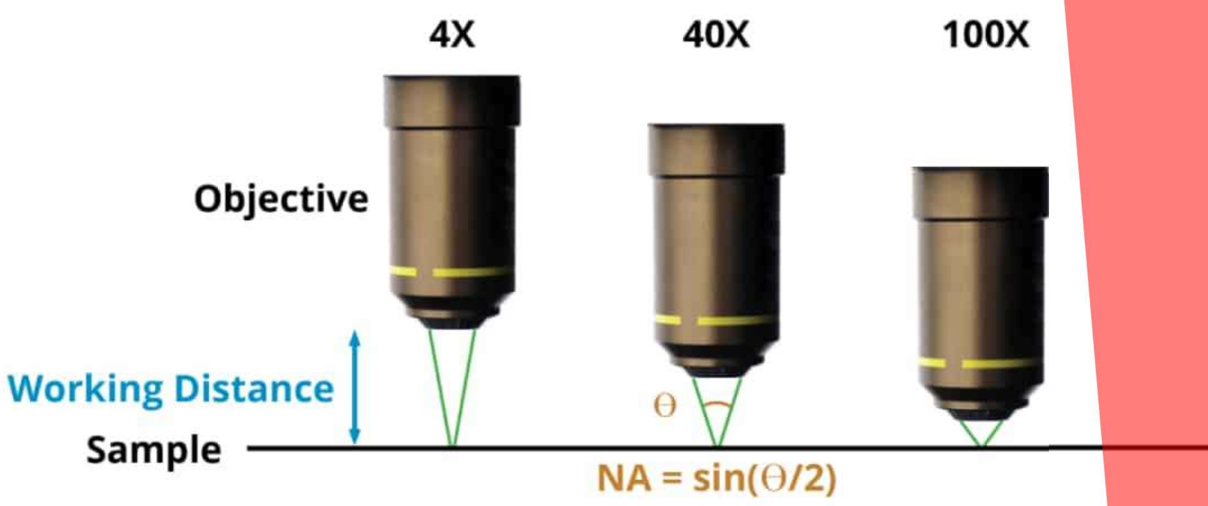
Magnetic lens

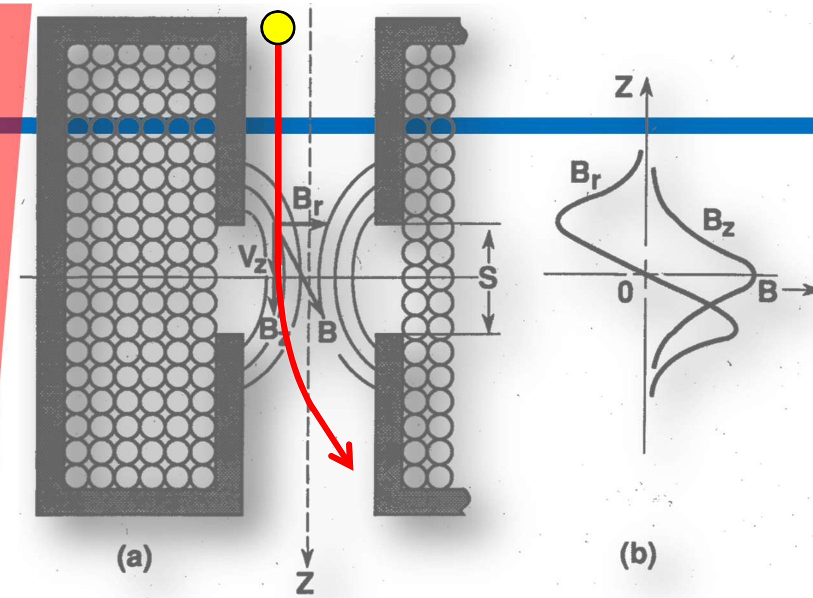
Principle: use B-field-induced **Lorenz force**. The beam is guided along the axis of a current-carrying coil (cross-section on the right: circles), which is mostly enclosed by a high-permeability material such as soft iron (easily magnetized, gray). The iron carries most of the flux lines. However there is a gap without magnetic material → flux lines "escape", they 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, $v_r = 0$ and the dominant Lorenz force (not el.field now) component is tangential, $F_\phi \propto v_z B_r$
→ **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! The non-uniform B field acts similar to convex lens.
It has a focal distance f which can be controlled by coil current.
Typical SEM has 2 condenser lenses (make parallel beam) and an objective lens (focus to spot).

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





$$F_\phi = -e\,(v_z B_r) + e\,(B_z v_r)$$
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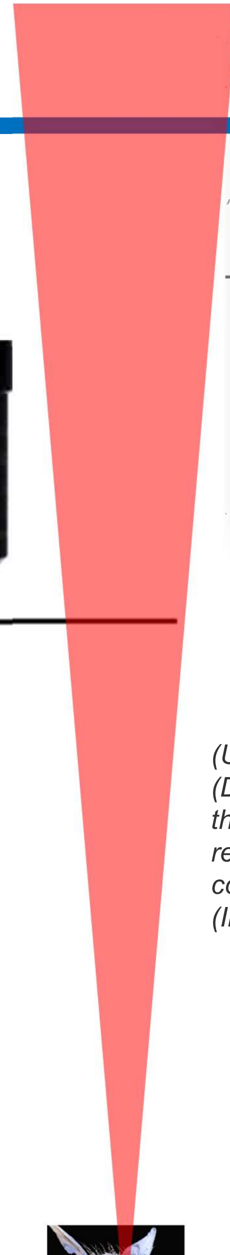
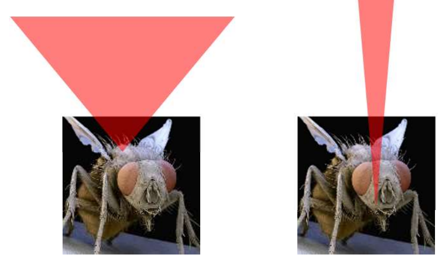
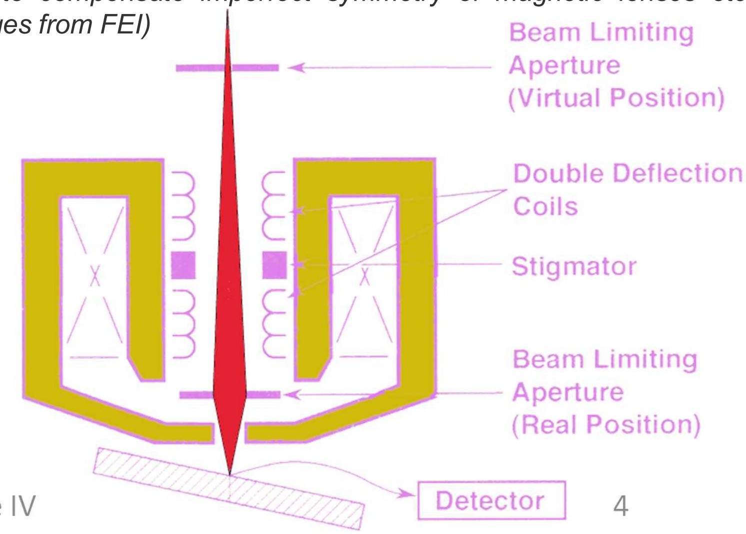
Current-controlled focus length (objective strength):
→ short/high working (focal) distance
→ short/high depth of field.

In optical microscopes the constraints of diffraction require a short working distance objective.
Here the focal length of objective is much higher → large depth of field in low magnification (5–30 mm): narrow electron beam, capable of imaging large objects with little loss of resolution.



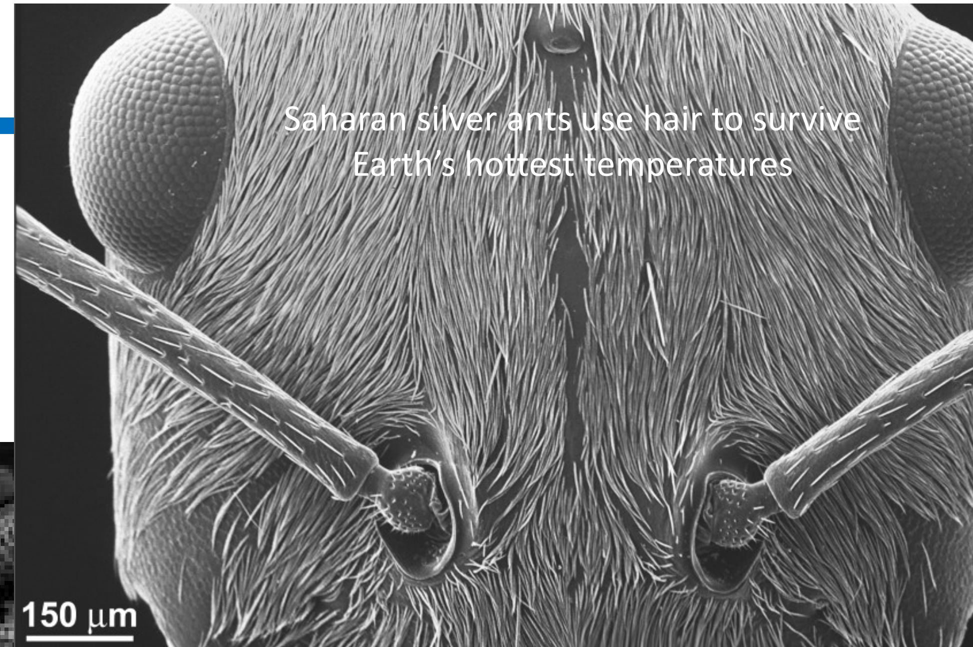


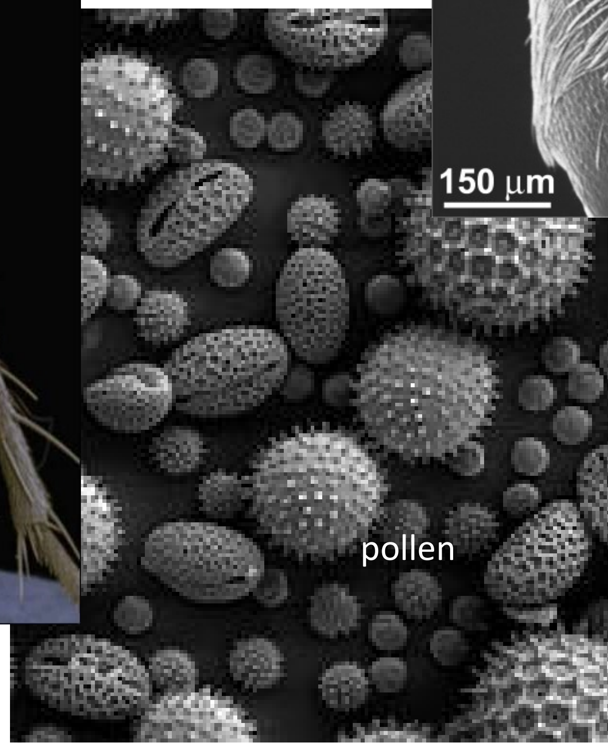


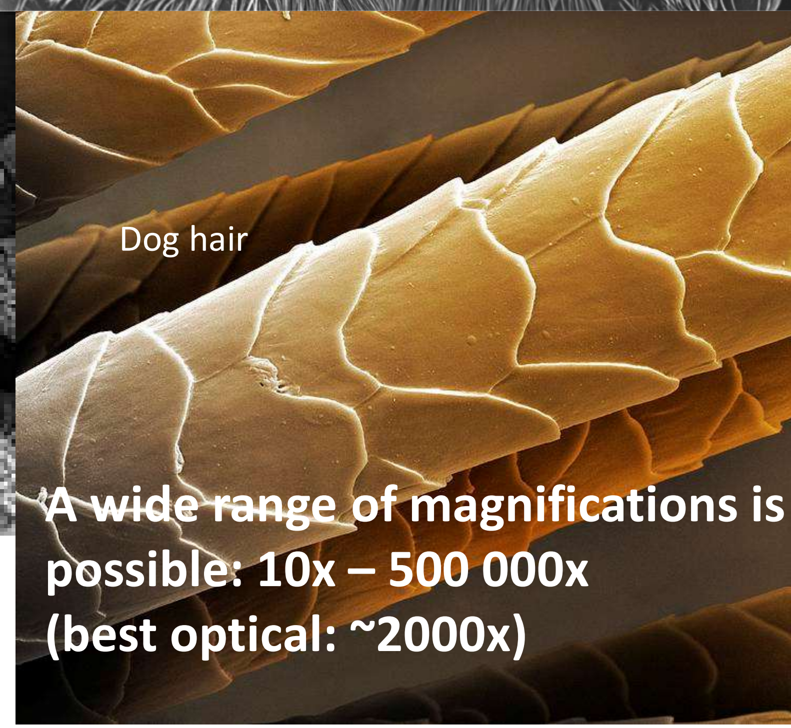
Nattelson Section 4.1.3 also *http://www.springer.com/cda/content/document/cda_downloaddocument/9780387258003-c2.pdf?SGWID=0-0-45-280920-p47261066*

Electron microscopy

Examples of large depth-of-field.
Characteristic 3D appearance





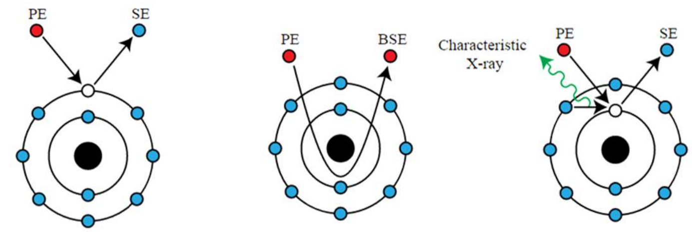


A wide range of magnifications is possible: 10x – 500 000x (best optical: ~2000x)

Potential problem in imaging: electric field from charging (electron accumulation on insulating surface).
Sample surface should conduct a bit. Surface charges from residual gas could be sufficient. If not, coat with thin gold layer (~10 nm).

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

Electron microscopy



(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 electrons 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

SEM overview:

Acceleration voltage (V_{acc}): 1-30 kV

Spatial resolution is not limited not by diffraction; rather the achieved spot size (non-ideal focusing) and interaction volume.

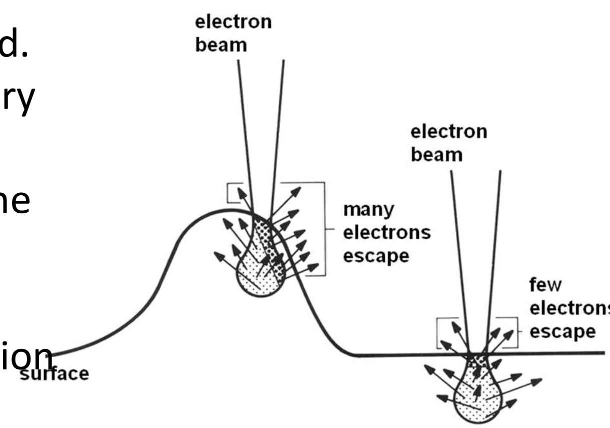
Advantage: large magnification range (from 10 above 500k) is possible with large depth of field, bulk samples also, analytical techniques to study composition

Detectors

Secondary electron detector (SE):

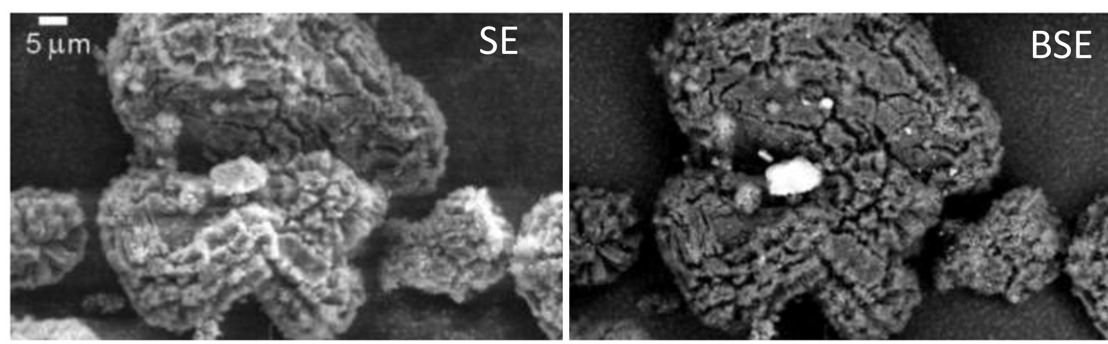
As e-beam hits the surface, electrons are kicked out from lower shells and detected.

- Heavy elements (high atomic number Z) are more effective at producing secondary electrons. E.g. large contrast of Au on Si.
- They come from very close to the specimen surface → high resolution image of the surface. Highest resolution: 0.4 nm (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 depends 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). SE image: edges are brighter. The BSE image shows the Fe particles with bright contrast. http://www.microscopy.ethz.ch/bse.htm

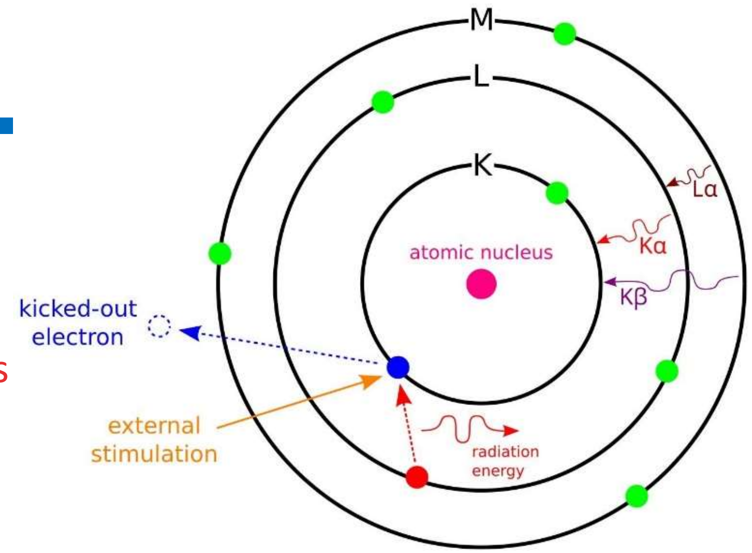
Nattelson Section 4.1.3 also Wikipedia

Electron microscopy

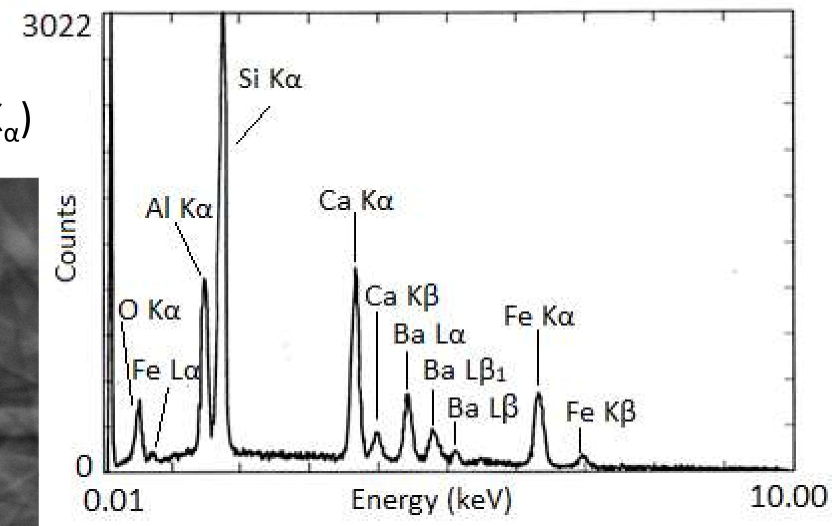
SEM

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

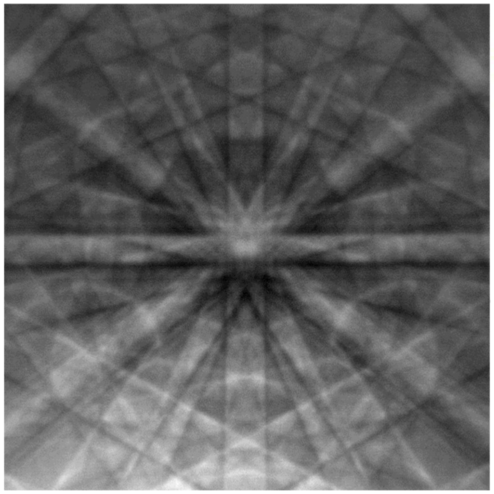


(Up) Principle of the underlying process of EDS
(Down) EDS spectrum of NIST K309 glass. Silicon, aluminum, barium, calcium, iron, and oxygen are identifiable in the spectrum. J. Goldstein, et. Al, , Scanning Electron Microscopy and X-ray Microanalysis, 3rd, Springer, New York (2003).



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

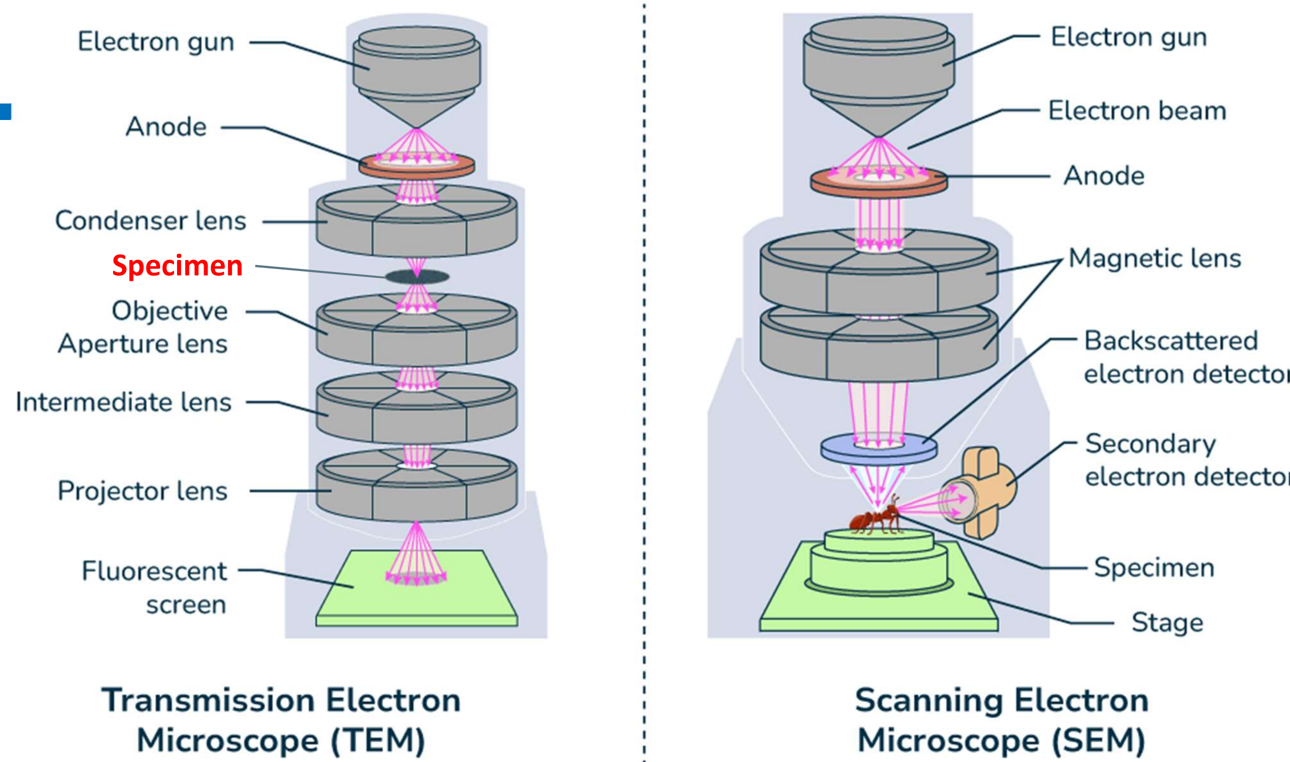
TEM

Acceleration voltage (V_{acc}): 100-300 kV

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

TEM System

- Thin specimen (~100 nm) is placed in the path of the e beam.
- Electrons emitted from the filament are accelerated to ~100 keV
- Condenser lenses for beam formation. Objective lenses for focusing.
- 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.



(Up) The main parts of a TEM microscope. https://www.geeksforgeeks.org/biology/electron-microscope-diagram/
(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/

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.

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, the 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. At 200 kV range the velocity v of electron is 70% of speed of light, thus relativistic expression of energy: $\lambda = \frac{h}{\sqrt{2meV_0}} \frac{1}{\sqrt{1+\frac{eV_0}{2mc^2}}}$.

For 200 kV, λ≈2.5 pm. Short wavelength → it is powerful for analyzing the **crystal structures of solids**. Intensity of the diffracted beam:

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

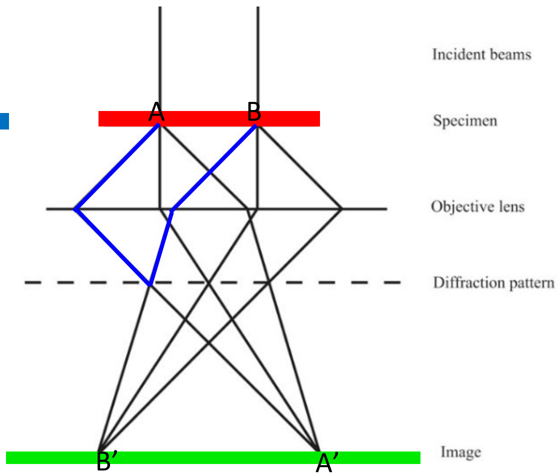
where F_g is the structure factor and **g** is the scattering vector.

Advantages of electron diffraction

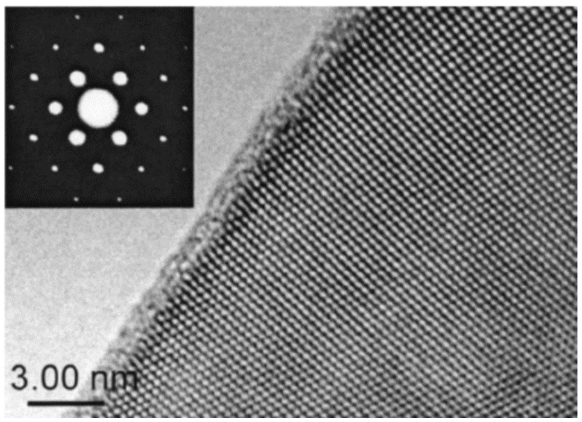
- Wavelength is smaller than for X-ray (X-ray: ~ 10 pm – 10 nm).
- Geometry of diffraction experiment can be varied by electron lenses.
- Diffraction on nanoscale single crystal is possible (compare: focusing X-rays??)
- Can be combined with direct image of the crystal, EDS, EELS or electron holography.

Good for symmetry determination (wurtzite or zinc blende, etc., even detect stacking faults), not so accurate to get lattice parameters

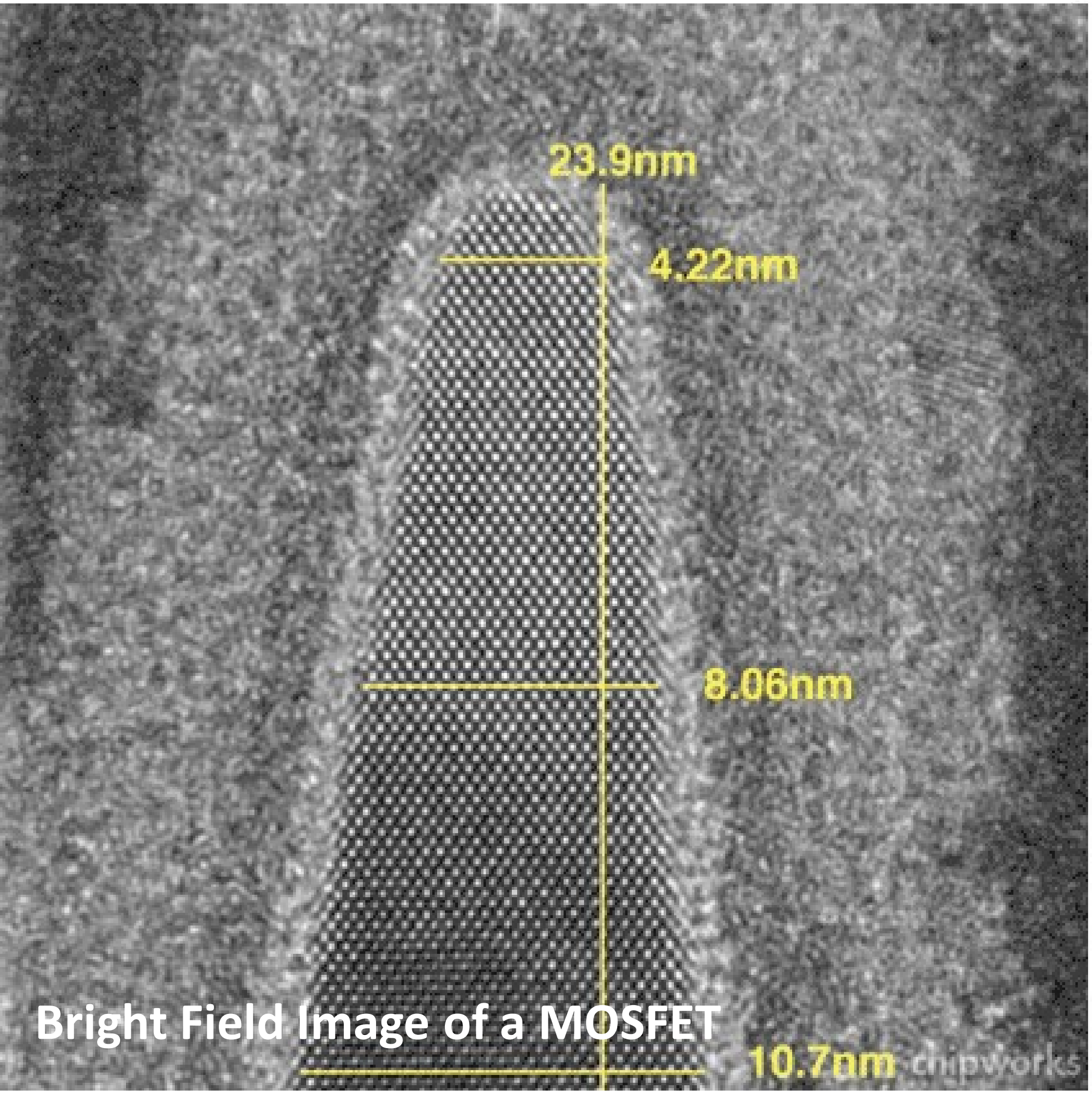
Nattelson Section 4.1.3 also Wikipedia



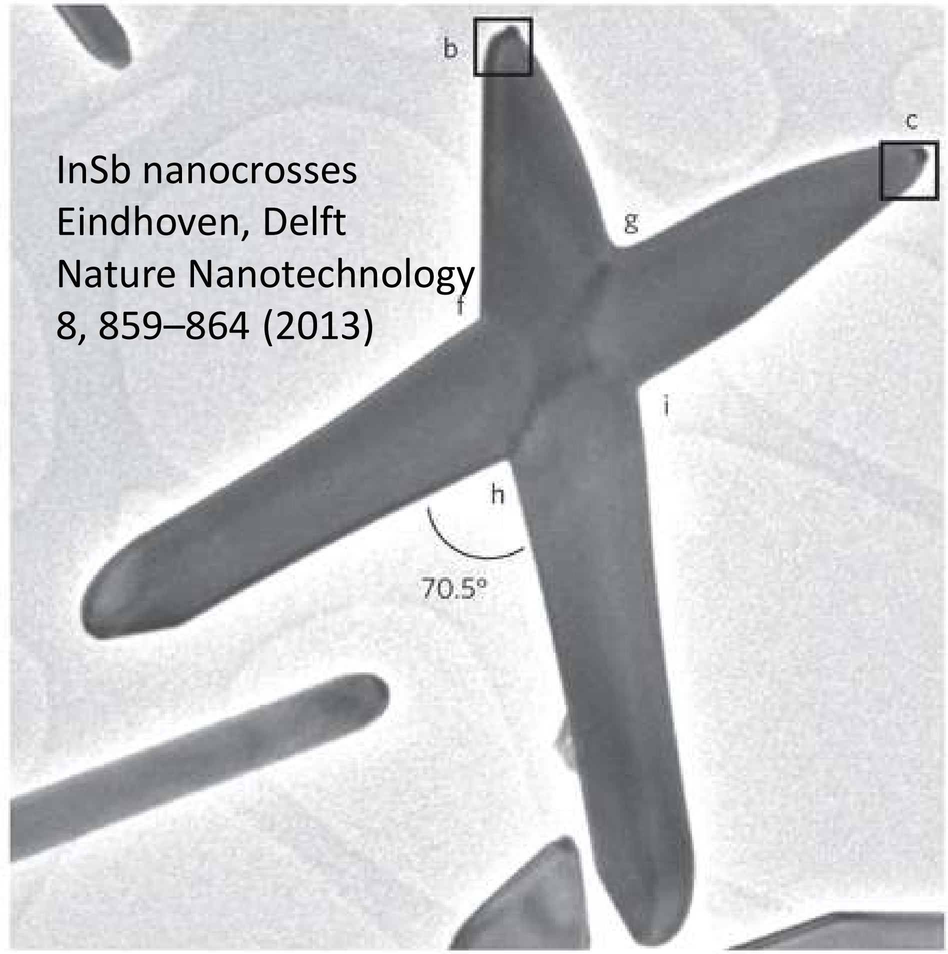
(Up) The objective lens acts to collect all electrons scattered from one point (A or B) of the sample to one point on the fluorescent screen, resulting an image of the sample. Note that at the dashed line in the figure, electrons scattered in the same ***direction*** *by the sample are collected into 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 → detect image plane (BF bright field) or diffraction plane (DF dark field). See also:*
https://www.globalsino.com/EM/page3710.html
https://en.wikipedia.org/wiki/Selected_area_diffraction
Other DF imaging:
https://myscope.training/TEM_Dark_field_imaging



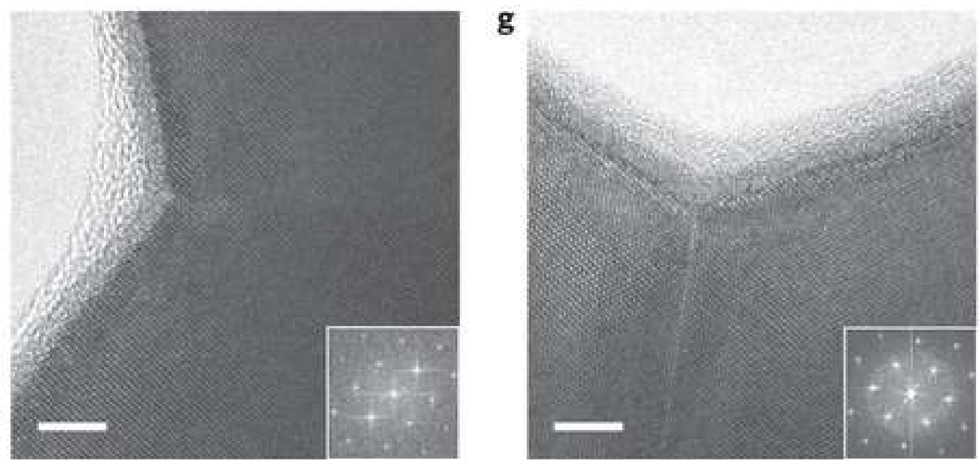
(Up) High-resolution TEM image of a 22 nm single crystal Si nanowire. The inset is a selected area electron diffraction pattern of the nanowire. Applied Phys. Letters 83, 2934 (2003)



Bright Field Image of a MOSFET



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



Important compared to other diffraction (neutron and X-ray): the transmitting electrons have a different interaction with specimen (Coulomb interaction).

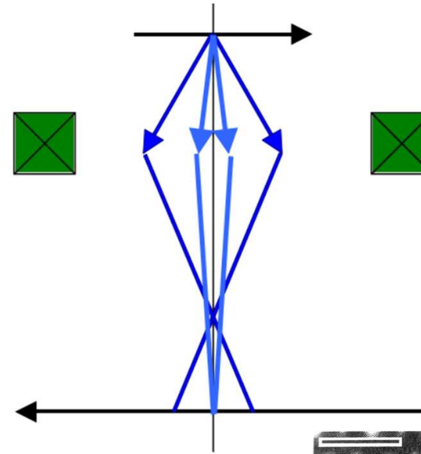
Nattelson Section 4.1.3 also Wikipedia

Electron microscopy

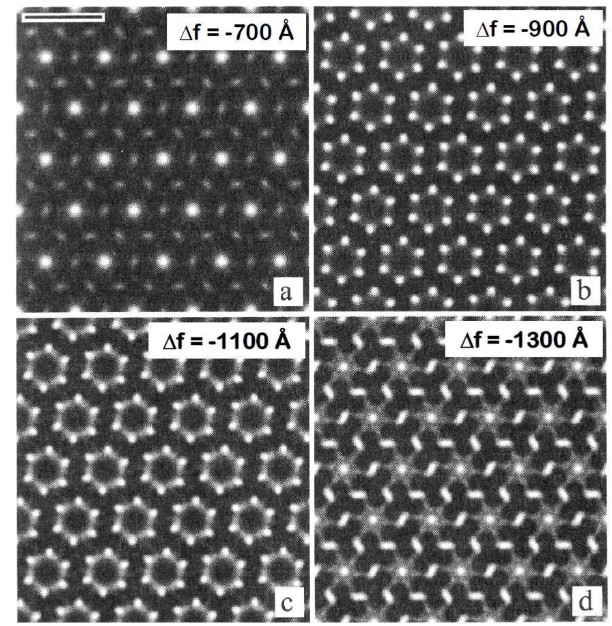
TEM Imaging modes

Phase contrast image (High Resolution TEM)

It is based on interference between electrons transmitted and diffracted by the sample. → sub-Å level of resolution is possible. Interference generates a complex pattern → Extensive image simulations are required.
Complications:
- Lensing: path length changes with distance from optical axis → phase shift
- 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.

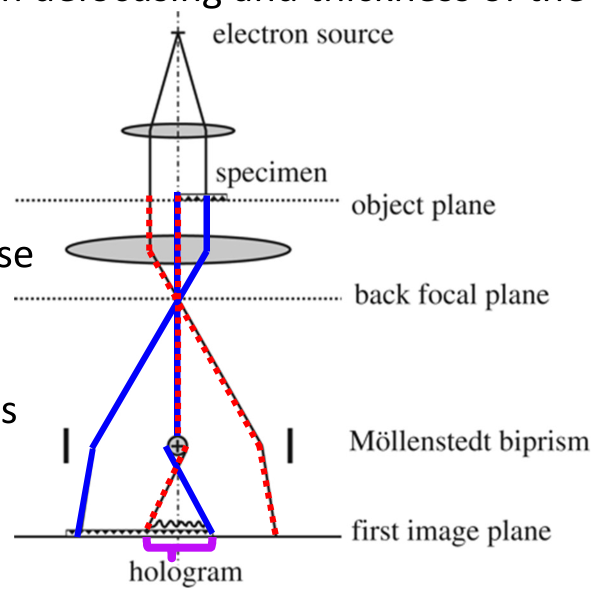


(Left) Spherical aberration: The focal point of a ray depends on its diffraction angle.
(Down) Example how the interference process affects the HRTEM images as defocus is changed for Si3N4 (0001) sample.
https://nanohub.org/resources/4020/download/2008.02.06-mse582-l11.pdf



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 is possible.

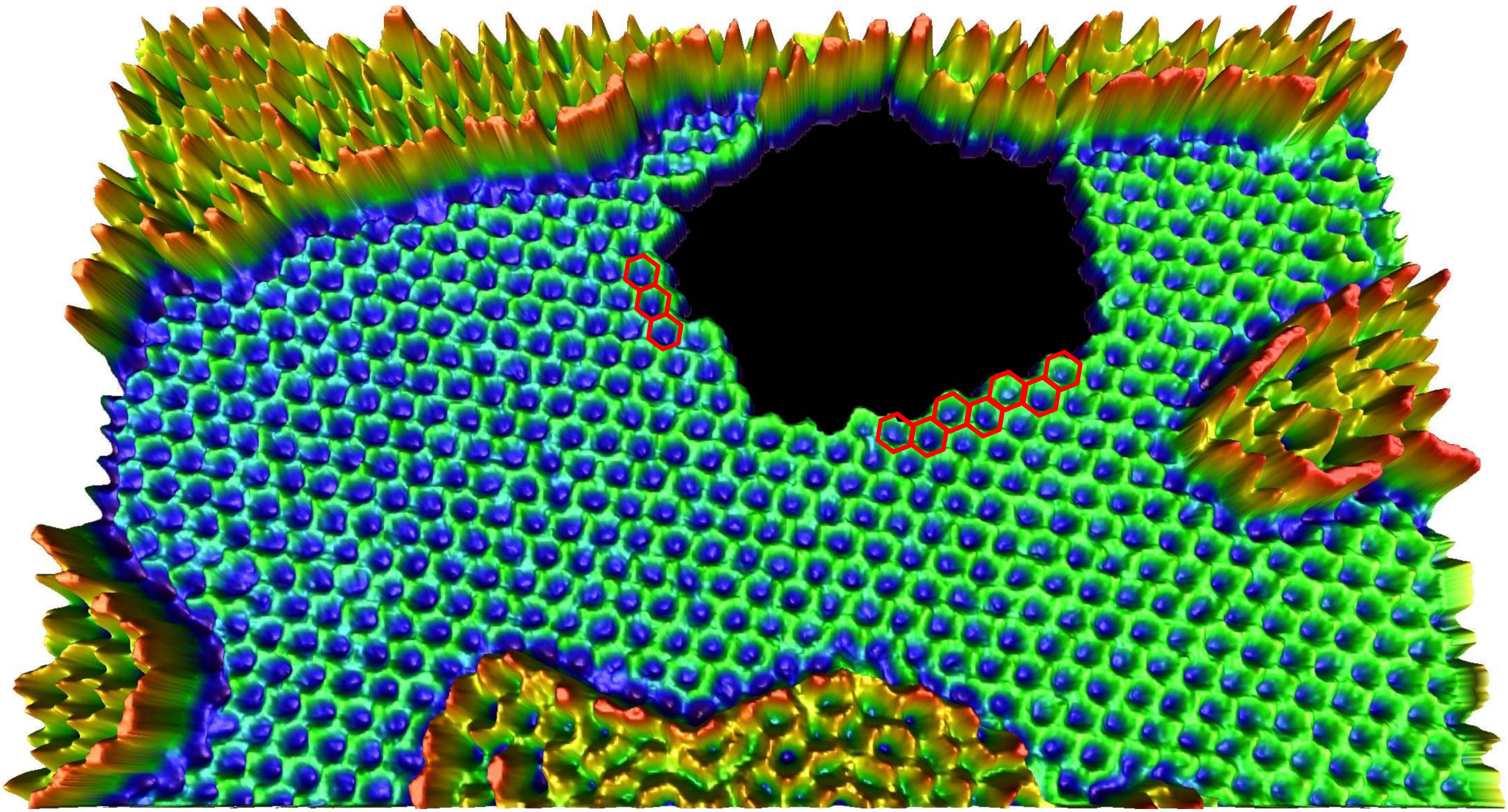


(Left) Setup for recording image-plane off-axis holograms. The object covering only half the object plane and the adjacent empty area are illuminated coherently. The object plane is imaged in the first image plane (between the blue ray paths). By means of the electron biprism (positively charge wire in the middle, grounded side plates), 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

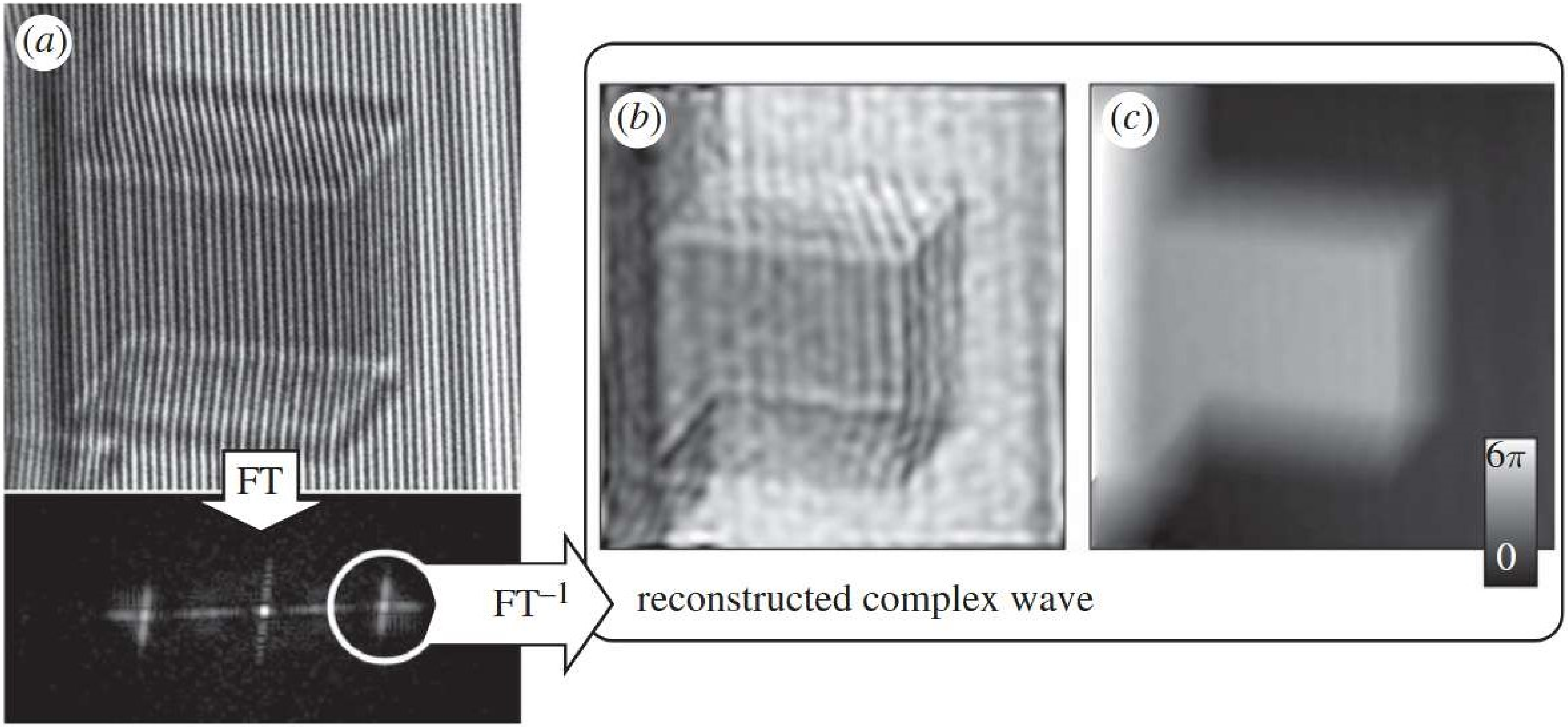
Nattelson Section 4.1.3 also Wikipedia

Electron microscopy

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



Transmission Electron Aberration-corrected Microscopy (TEAM). It has 0.5 Å 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.
http://newscenter.lbl.gov/2009/03/26/atoms-in-action/ Berkeley



(a) Hologram of an MgO crystal and reconstructed object (*b*) amplitude and (*c*) phase …. retrieved by [selecting] one sideband of the hologram **spectrum** [FT] 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

Nattelson Section 4.1.3 also Wikipedia

Electron microscopy

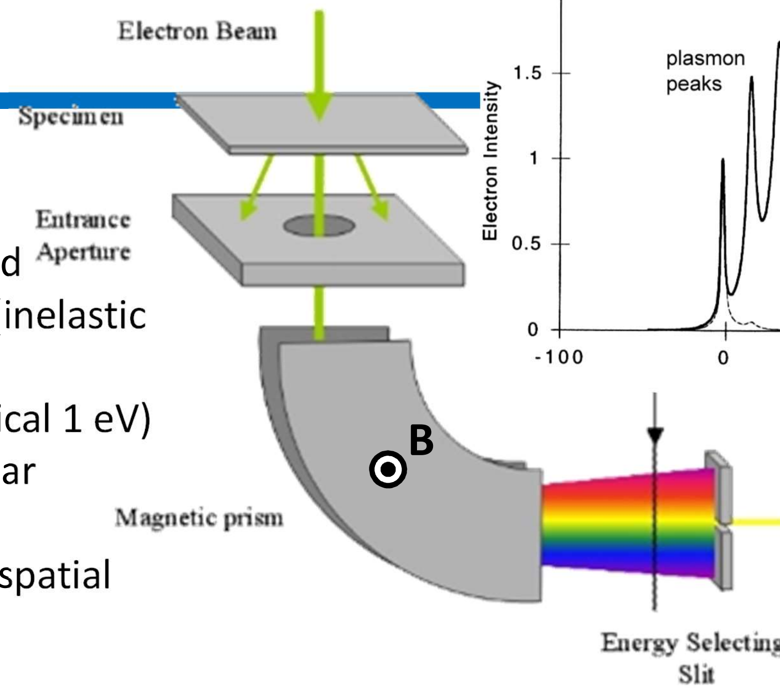
TEM Imaging modes

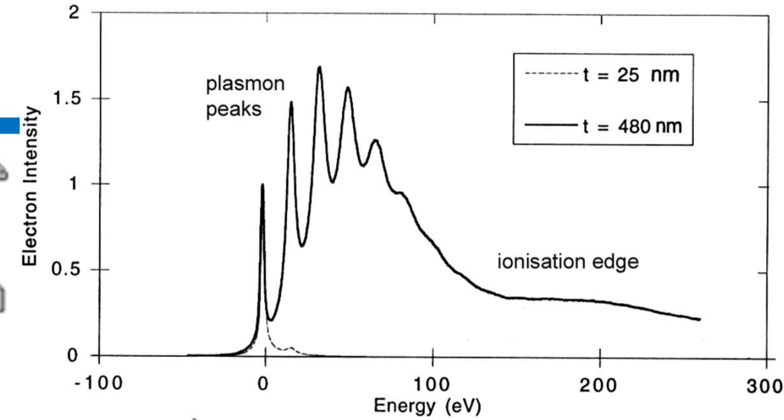
Electron energy loss spectroscopy (EELS)

precisely measuring the energies of transmitted electrons, it is possible to identify excitations (inelastic processes)

- energy resolution of as good as 0.1 eV (typical 1 eV)
- spatial resolution down to atomic level. Clear advantage compared to x-ray absorption spectroscopy (XAS), where using cyclotron spatial resolution is ~30 nm.

Good for local properties: band gap, chemical composition, thickness, plasmons, electronic density states.



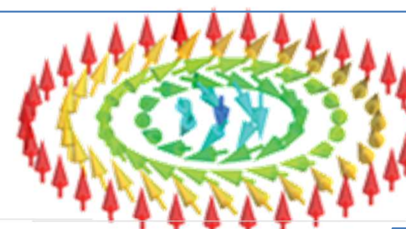


(Up) EELS spectrum of plasmon peak in Si of low thickness t=25 nm. For thicker sample: peaks at multiples of plasmon energy. 2009 *Rep. Prog. Phys.* **72** 016502. *(Left) EELS setup attached to TEM. Magnetic prism is used with perpendicular B field to separate electrons with different energy. http://www-hrem.msm.cam.ac.uk/research/EFTEM/EFTEM.html*

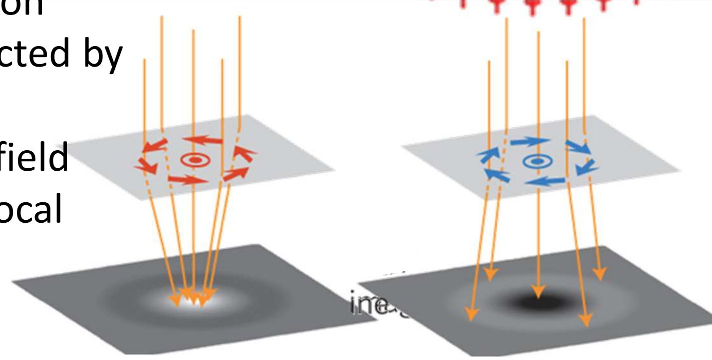
Lorentz TEM

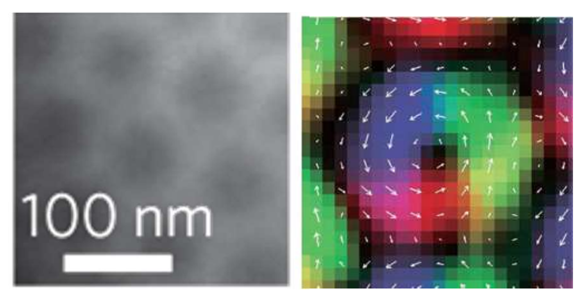
Principle: an electron beam passing through an area with a component in magnetic induction perpendicular to its trajectory will be deflected by the Lorentz force. Induction either from magnetization in the sample itself or stray field exterior of the sample. It allows to detect local magnetic property 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 (2013)

Nattelson Section 4.1.3 also Wikipedia

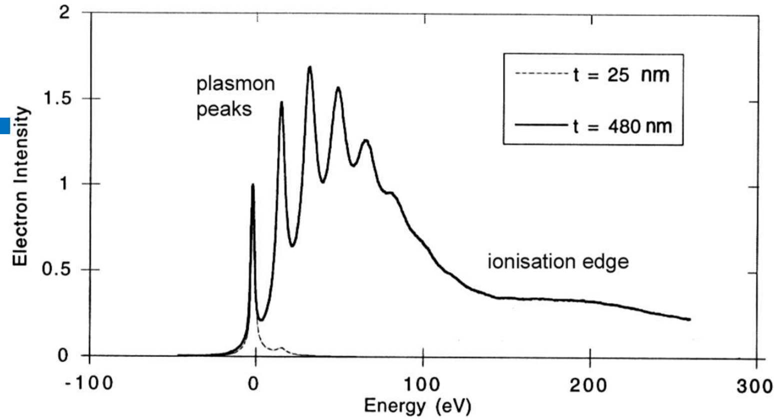
Electron microscopy

When a fast-moving electron passes through a solid, the nearby atomic electrons are displaced by Coulomb repulsion, forming a correlation hole (a region of net positive potential of size ~1 nm) that trails behind the electron. Provided the electron speed exceeds the Fermi velocity, the response of the atomic electrons is oscillatory, resulting in regions of alternating positive and negative space charge along the electron trajectory; see Figure 5.

The effect is known as a ***plasmon wake***, in analogy to the wake of a boat travelling on water at a speed higher than the wave velocity or an aircraft flying faster than the speed of sound.

As the electron moves through the solid, the backward attractive force of the positive correlation hole results in energy loss [of the electron]. The process can be viewed in terms of the creation of pseudoparticles known as *plasmons*, each of which carries a quantum of energy equal to *E*p.

The energy loss is interpreted as the creation of plasmons, giving an EELS spectrum consisting of a peak at Ep and its multiples (top right).



(Up) EELS spectrum of plasmon peak in Si of low thickness t=25 nm. For thicker sample: peaks at multiples of plasmon energy.
2009 *Rep. Prog. Phys.* **72** 016502.

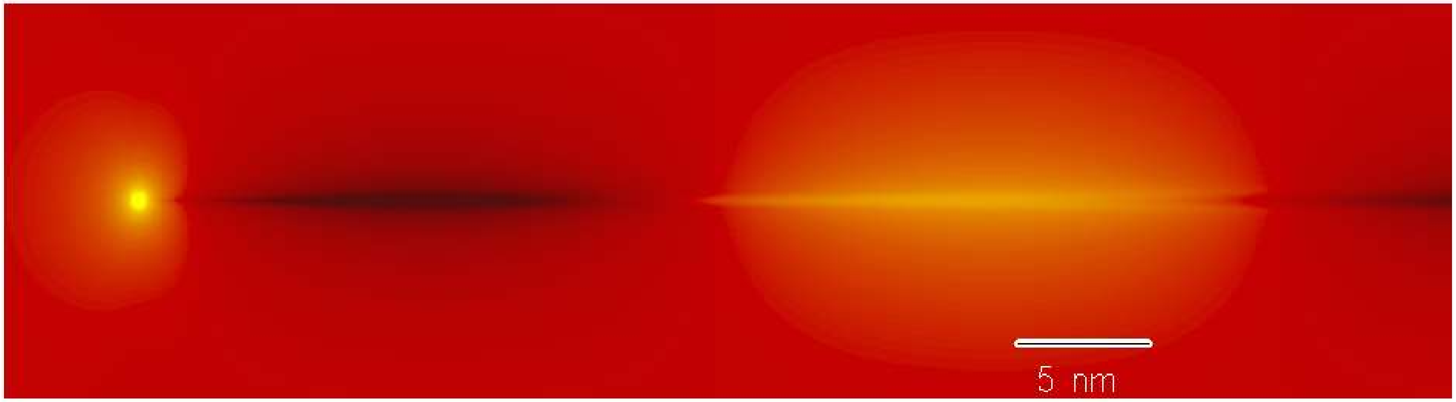


Figure 5. Plasmon wake of a 100 keV electron travelling through aluminium, calculated from the dielectric properties (P E Batson, personal communication). The electron is represented by the bright dot on the left; alternate dark and bright bands represent positive and negative regions of space charge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