Electron Microscopy I
Lecture 04

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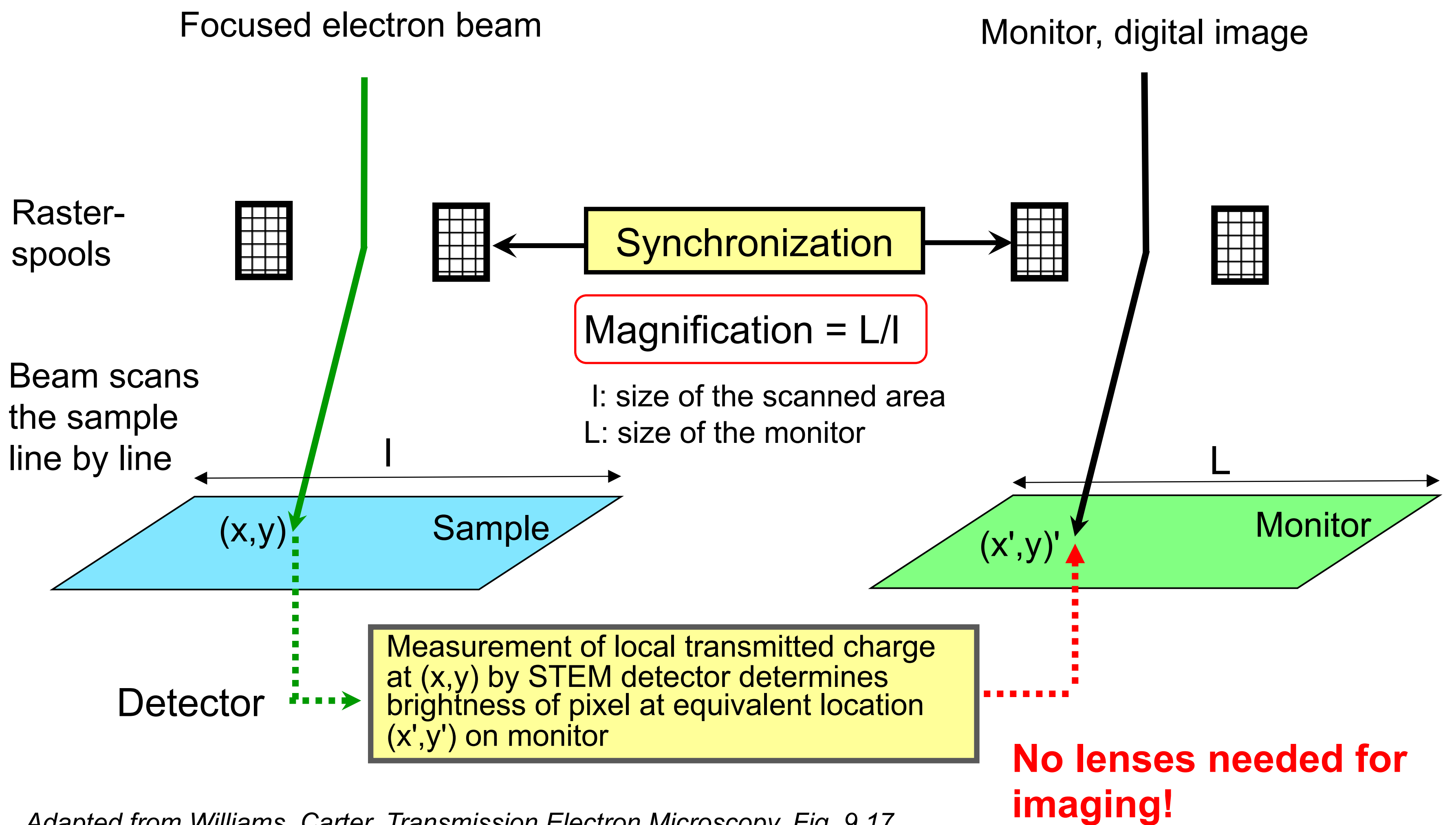
Contents

Lab course information

Information about the lab course will be

Gruppe	Praktikumstag		Name	Vorname	Mail
1	Dienstag	14:00 Uhr	Holz	Nils	ueolh@student.kit.edu
			Seidel	Johanna	johanna.seidel@kit.edu
			Yüksel	Deniz	dyuksel1@hotmail.com ???
			Lamis Saeed		ulmty@student.kit.edu
2	Mittwoch	14:00 Uhr	Nurak	Ingrit Sisilia Rosari	ingrit.nurak@kit.edu
			Kraft	Kristian	kristian.kraft@kit.edu
			Schwartz	Arne	arne.schwartz@student.kit.edu
	KW 48	28.11. + 29.11.2023			
	KW 50	12.12. + 13.12.2023			
	KW 2	09.01. + 10.01.2024			
	KW 5	30.01. + 31.01.2024			

Scanning transmission electron microscopy (STEM) in the transmission electron microscope: principle of image formation.

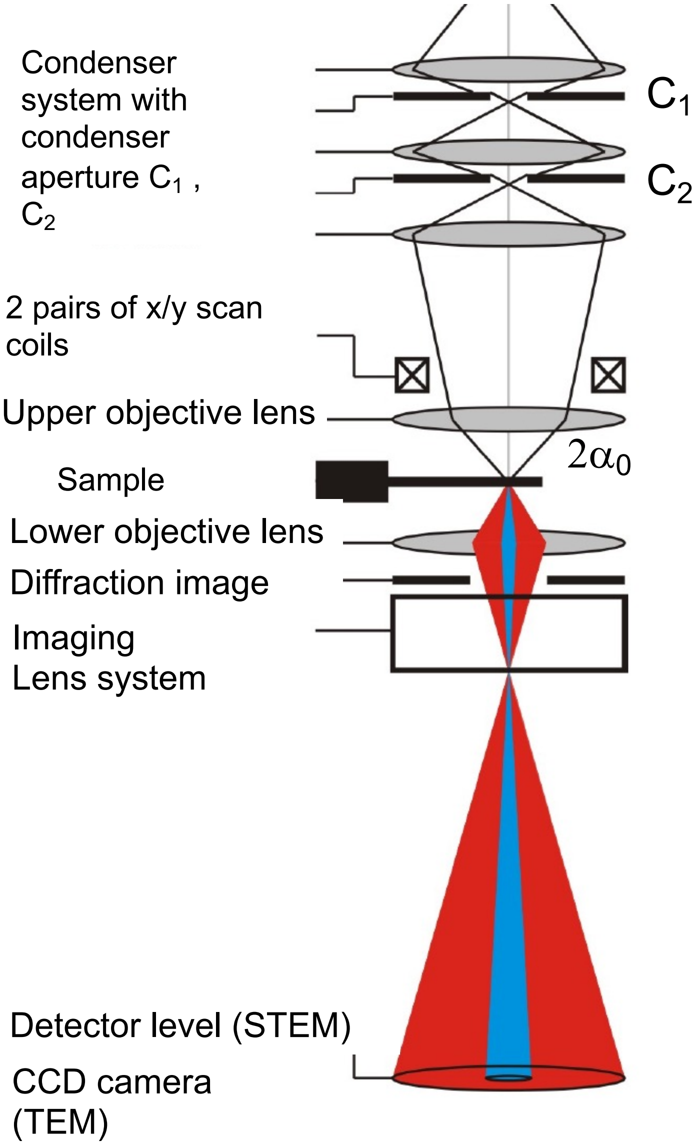


Adapted from Williams, Carter, Transmission Electron Microscopy, Fig. 9.17

STEM (Scanning Transmission Electron Microscopy)

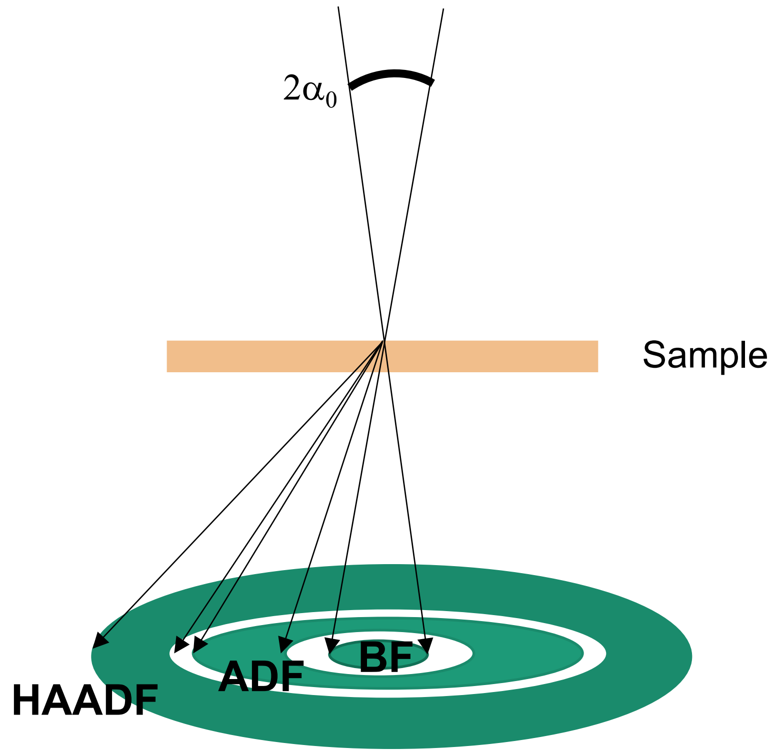
Focusing of the electron beam ("probe") on small diameter.
The beam diameter depends on the aberrations of the lowest focusing lens and the beam convergence angle α_0 . The beam diameter can vary between several nm and ~0.05 nm (in microscopes with "probe" corrector) depending on the desired resolution and imaging mode.

- The convergence angle α_0 is determined by the diameter of the C2 condenser aperture
- A diffraction image is formed in the focal plane of the lens
- No imaging lens system is necessary to generate a STEM image!
- The imaging lens system projects the diffraction pattern onto the detector plane. The magnification can be selected by the "Camera length" setting



STEM detectors

- For electrons scattered into different angular ranges
- Scattering angle ranges can also be changed by camera length of the imaging lens system



BF: Bright field detector for unscattered or electrons scattered to small angles.
ADF: Annular dark field detector for electrons scattered to large angles
HAADF: High-angle annular dark field detector for electrons scattered to very large angles.

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STEM detectors

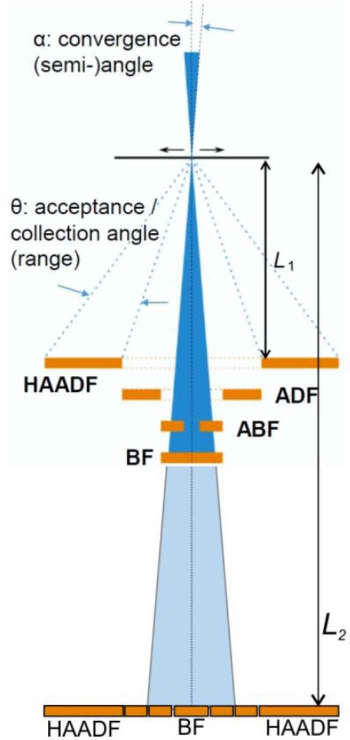
- The total signal of the STEM detector is composed of a thermal diffuse scattering and an elastic scattering. Depending on the scattering angle.

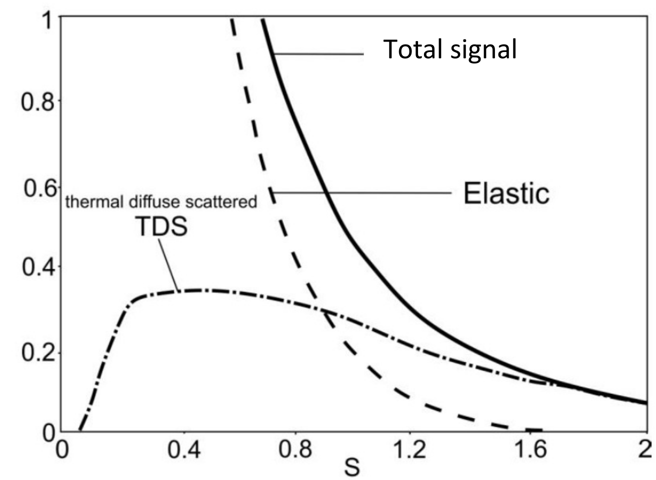
For small camera lengths L:

- For small scattering angles, elastic scattering predominates

  → Diffraction contrast on the BF Detector

- At large scattering angles and predominates the thermal diffuse scattering which is proportional to Z^2 (Z= atomic atomic number) comparable to Rutherford-like scattering
- → Z-contrast on the HAADF detector

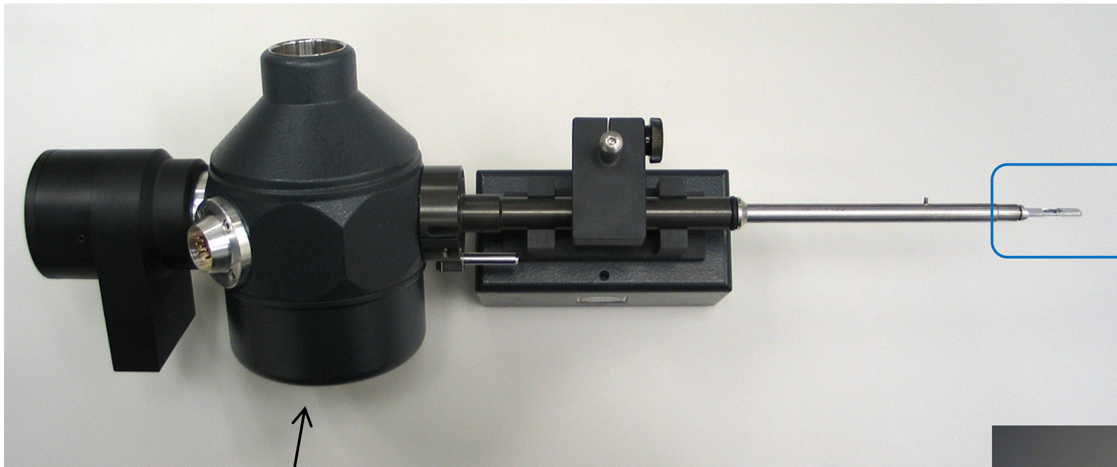


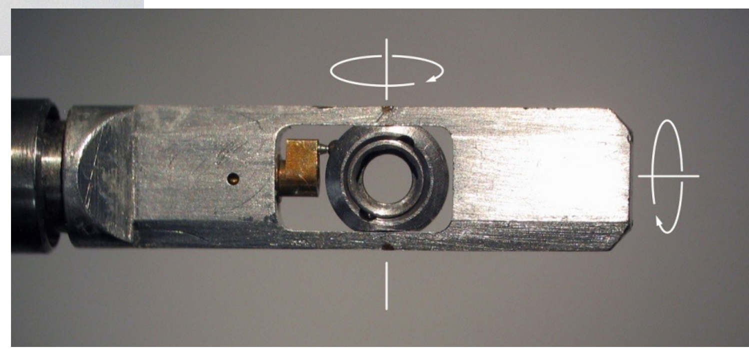


- STEM detector rings physically lie in one plane
  - Scattering angle ranges can also be changed by camera length of the imaging lens system

2.1 Design and mode of operation: Transmission electron microscope

- Analytical Double Tilt Holder for Material and Solid State Research
- Sample diameter always 3 mm



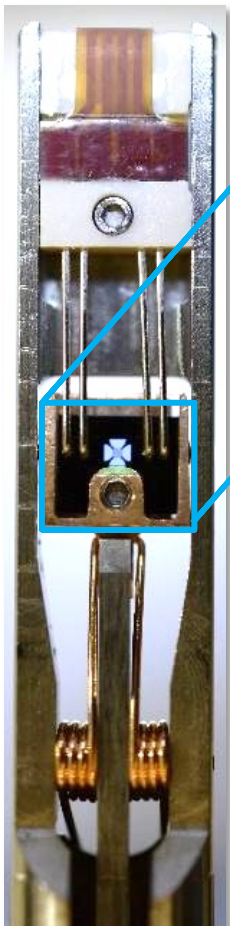


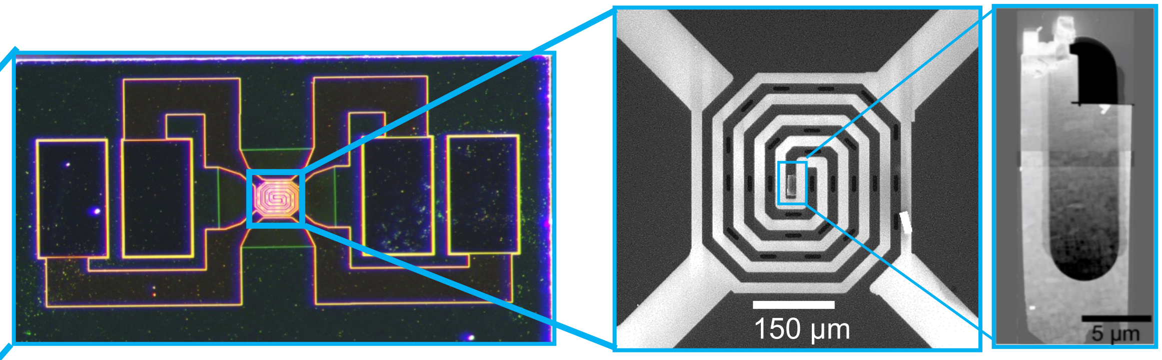
Photos: L. Dieterle (LEM)

Reservoir for liquid nitrogen -
Cooling holder

<u>Special specimen holder</u>: (in situ / in operando specimen holder)
Cooling, heating holders, specimen holders with electrical feedthroughs
"environmental cell" for experiments at elevated gas pressure, sample holder for sample deformation

In situ TEM heating holder (LEM)





- MEMS based heating chips
- Very small heating zone
- Allows very fast heating and cooling rates
- Real-time experiments in TEM possible
- For direct observations of material changes when external stimuli is applied.

http://denssolutions.com

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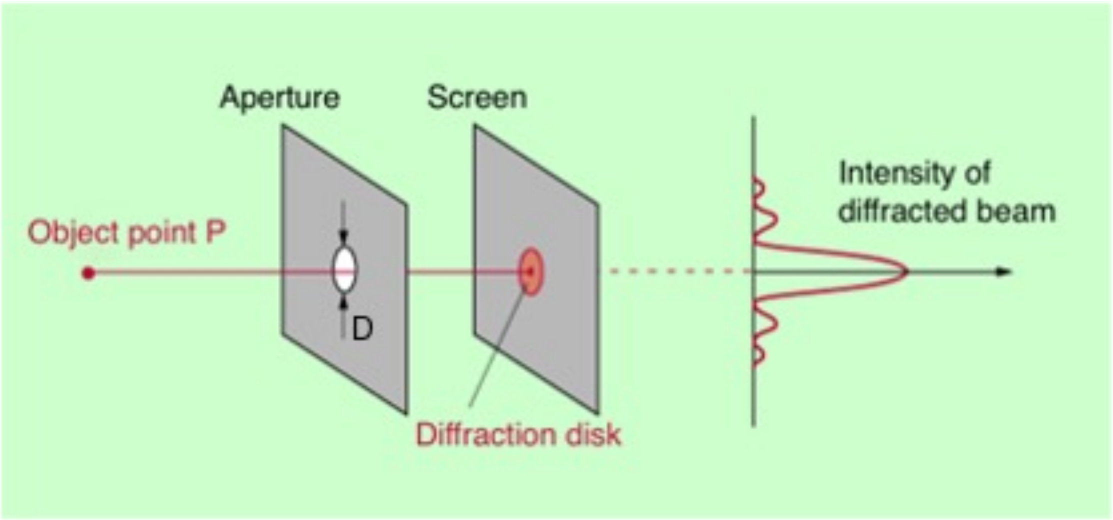
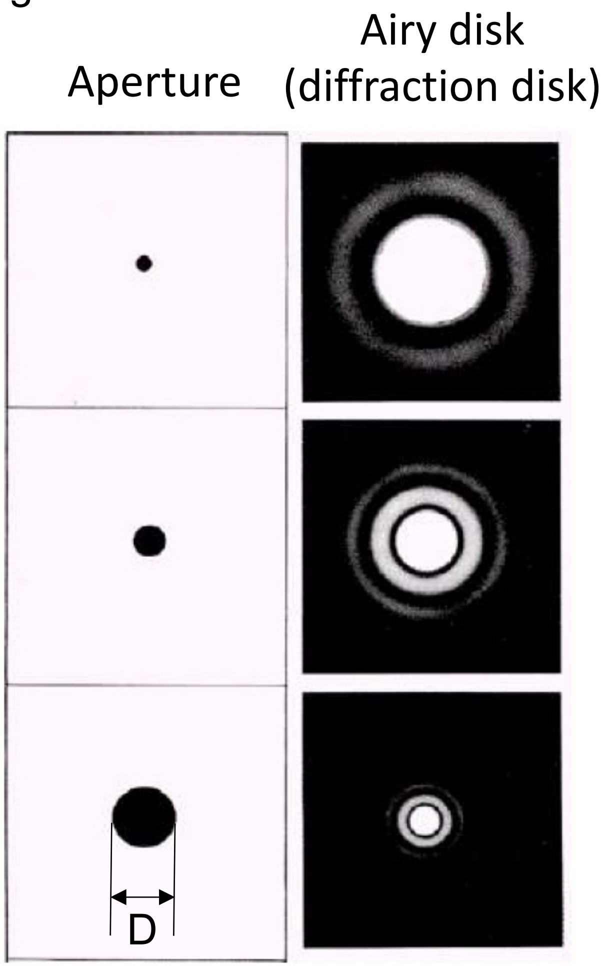
Different approaches for describing the *diffraction-confined resolution* (with negligible lens aberrations)

Rayleigh criterion: observation of diffraction at a round aperture with diameter D (e.g. edge of a lens) for light with wavelength λ





$$\sin\theta = \frac{1.22\lambda}{D}$$

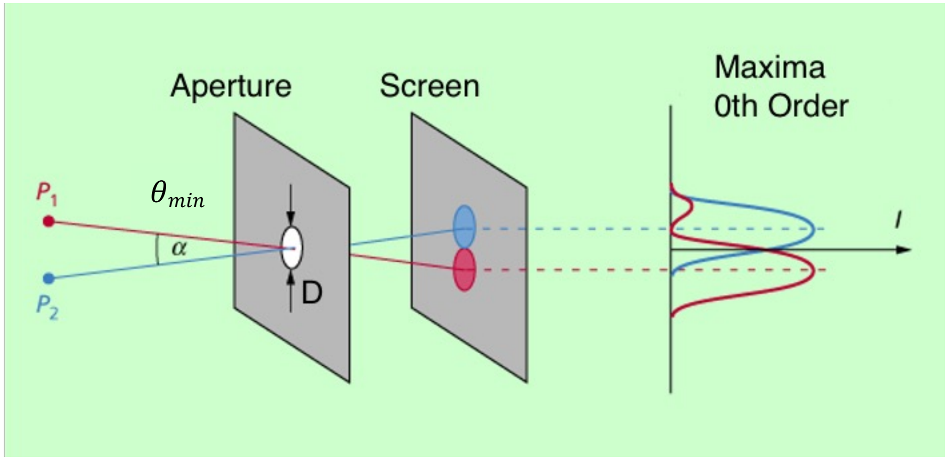
r : radius of the central intensity maximum (Airy disk)

$$r = \frac{1.22\lambda}{D} f$$

f : distance aperture (lens) to observeration screen ~ focal length f

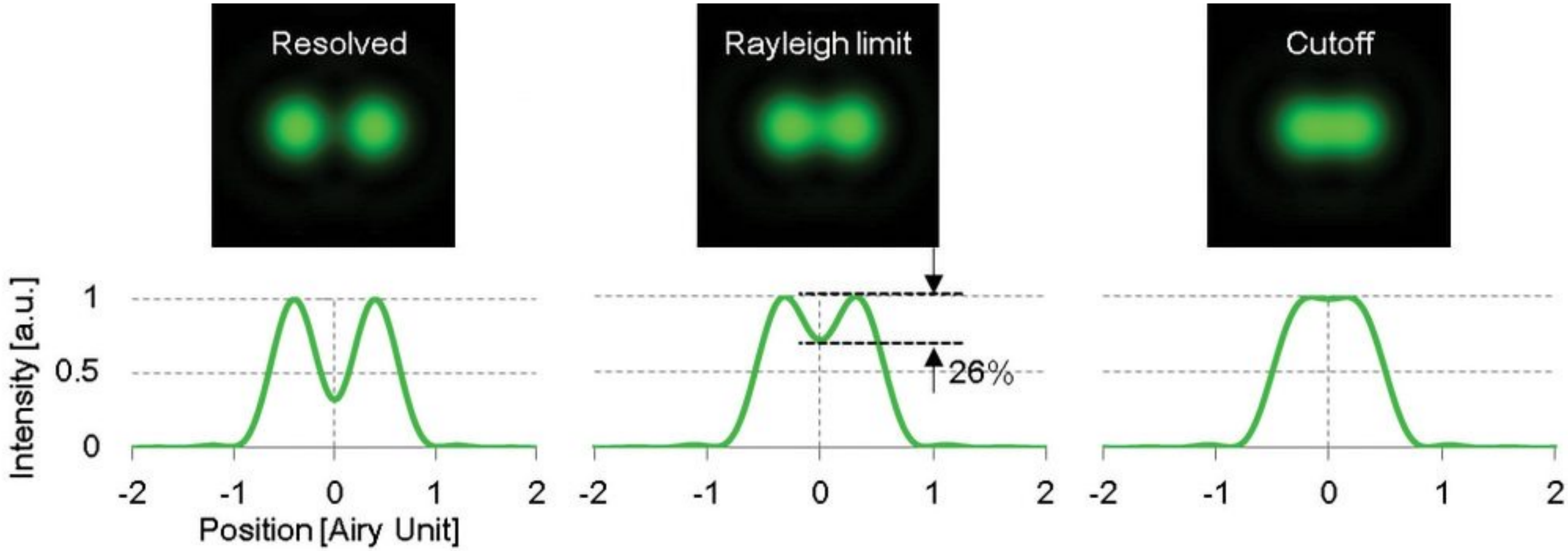
2.2 Lens aberrations in electron optics and their effect on the resolution

Minimum angular distance between two point-like (self-luminous) objects



$$\sin\theta_{min} = \frac{1.22\lambda}{D}$$

$$r = \frac{1.22\lambda}{D} f$$



2.2 Lens aberrations in electron optics and their effect on the resolution

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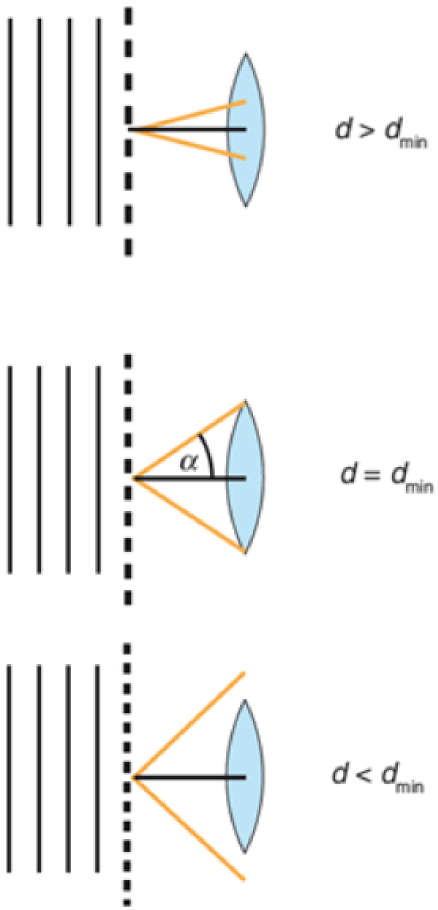
Abbe criterion: Up to which grating constant d_{min} does the imaging succeed? **Resolution based on observation of diffraction from periodic objects (e.g., slit gratings)** ***under coherent illumination.***



At least the first order of diffraction must fall within the lens: $\sin\alpha \geq \frac{\lambda}{d}$
From this follows:

$$d_{min} = \frac{\lambda}{\sin\alpha}$$

$$\sin\alpha = \frac{\lambda}{d} = g\lambda$$

In light optics, a higher resolution can be achieved by an immersion fluid between lens and object with refractive index $n_i > 1$

$$\sin\alpha = \frac{g\lambda}{n}$$

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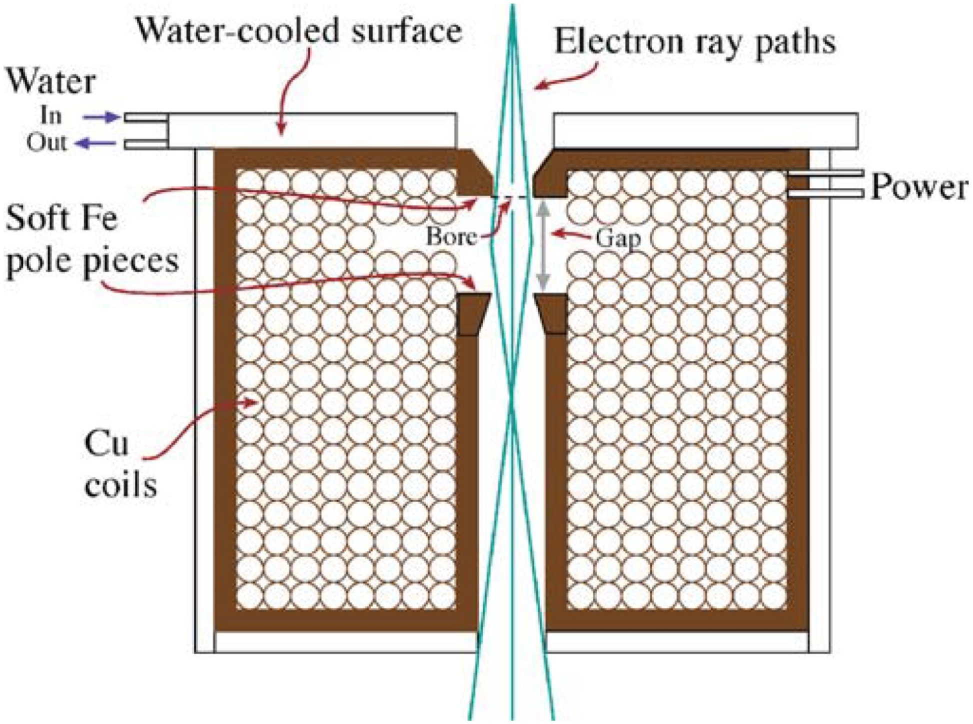
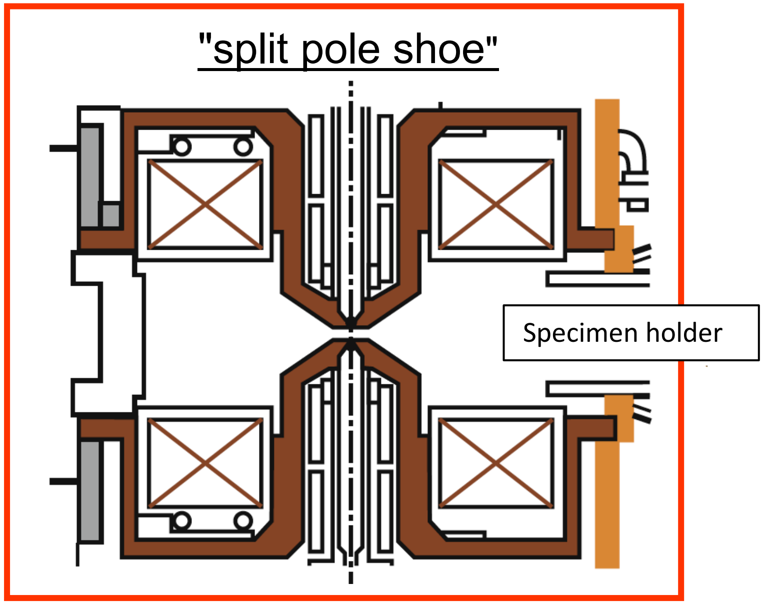
Magnetic lenses





Frequently used in TEM.
Space for holders and detectors.

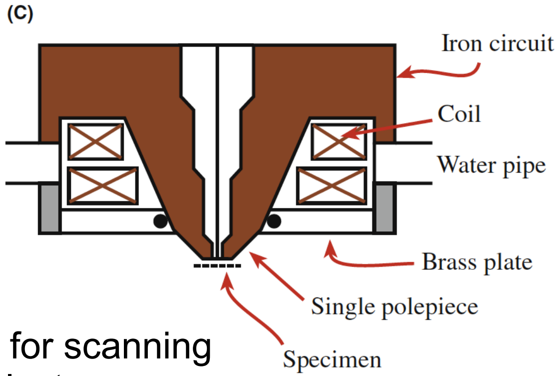
D.B. Williams, C.B. Carter, Transmission Electron Microscopy, Fig.6.6

D.B. Williams, C.B. Carter, Transmission Electron Microscopy, Fig.6.8.

Frequently used: "split polepiece" configuration consisting of lower and upper pole piece
Sample is in between the magnetic field (immersion lens)

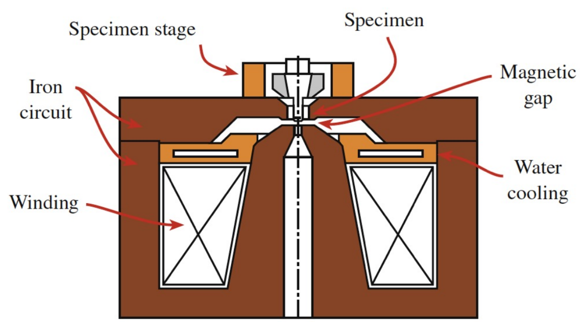
Magnetic lenses

"Mini lens"



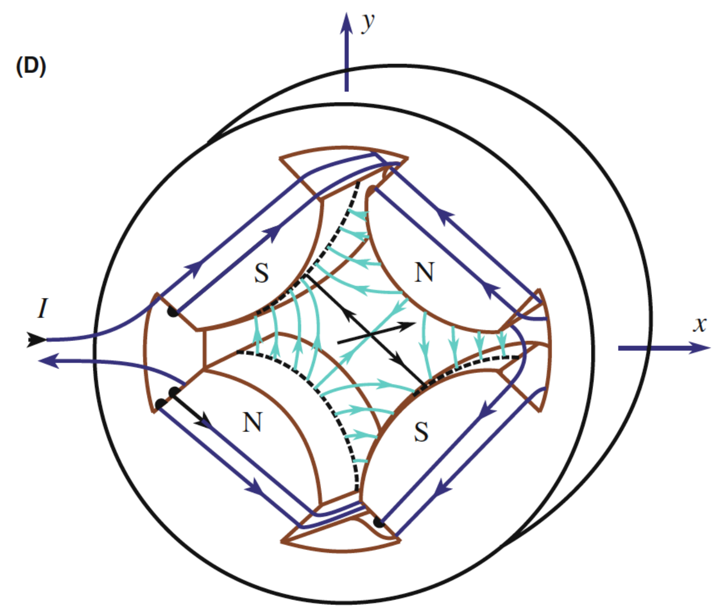
Objective lens for scanning transmission electron microscopy (STEM)

"top entry immersion lens"



Multipole lenses: e.g.

(quadrupole lens, octopole)



For the correction of lens aberrations (astigmatism)

D.B. Williams, C.B. Carter, Transmission Electron Microscopy, Fig.6.8.

Summary after lecture 2

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- Transmission electron microscopes are technically complex instruments with a wide variety of components. Electron source, condenser lens system, sample, imaging lens system, and camera/screen. Pumps create a high vacuum, which is needed to avoid interactions and thus deflections of the electrons by air molecules.
- Cathodes are electron emitters that emit electrons based on thermal emission and a combination of thermal and field emission. Thermal emission: Heating of the cathode material until some electrons have enough kinetic energy to overcome the work function (posterior part of the Fermi distribution). Field emission: A strong electric field releases electrons with low energy width from the cathode. Several criteria determine the quality of the cathode.
- The condenser system consists of the C1 lens and a C2 lens. The focal length of the C1 lens can be adjusted over several fixed values ("spot size"). The C2 lens is continuously adjustable. Thus focusing of the beam or parallel illumination on the specimen is possible.
- The objective lens, produces a diffraction image in the 1st back focal plane, and in its image plane the 1st intermediate image. By different excitation of the first intermediate lens, we project either a diffraction image or the real image of the sample onto the screen.
- In diffraction mode, local crystal structure analyses can be performed by acquiring diffraction images of small sample areas. The sample can be tilted, and thus precise excitation conditions can be defined in the back focal plane using the objective aperture. Setting of dark field, bright field, etc.
- Scanning transmission electron microscopy: Rastering of the beam probe line by line over the sample area. Measurement of local transmitted charge at (x,y) by STEM detector determines brightness of pixel at equivalent location (x',y') on monitor. STEM detector consists of several rings with different diameters. These cover different scattering angle ranges, which can also be determined with the camera length.
- Various specimen holders for transmission electron microscopy. Double tilt for diffraction analysis. "in situ" specimen holders to perform experiments directly in the TEM.

Magnification

According to Newton's lens law, we define the magnification as the ratio of the focal length (d_i) and the object width (d_O):

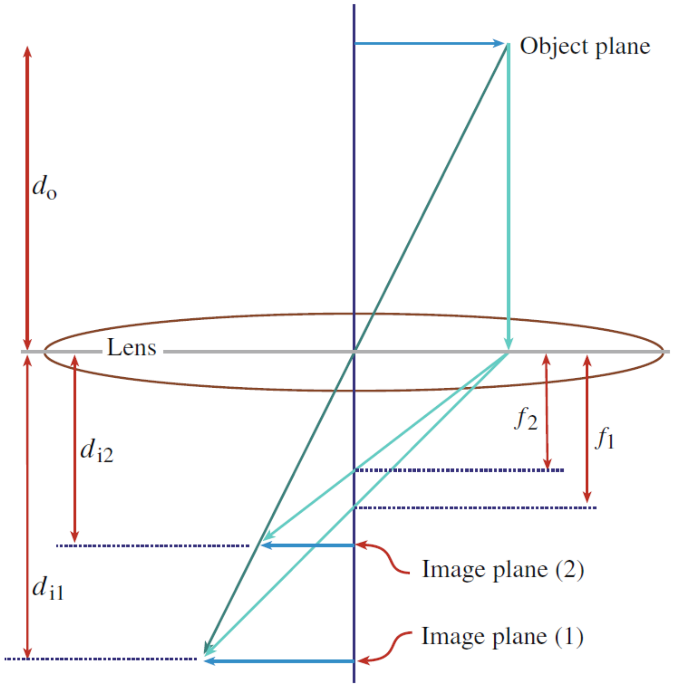

FIGURE 6.4. Strengthening the lens shortens the focal length f. So a weaker lens ($f1$) produces a higher magnification of the object than a stronger lens ($f2$) since the image distance d_i increases, but the object distance, d_o, is unchanged.

$$M = \frac{d_i}{d_o}$$

In the TEM small angles, it also applies approximately:

$$M = \frac{\beta}{\alpha}$$

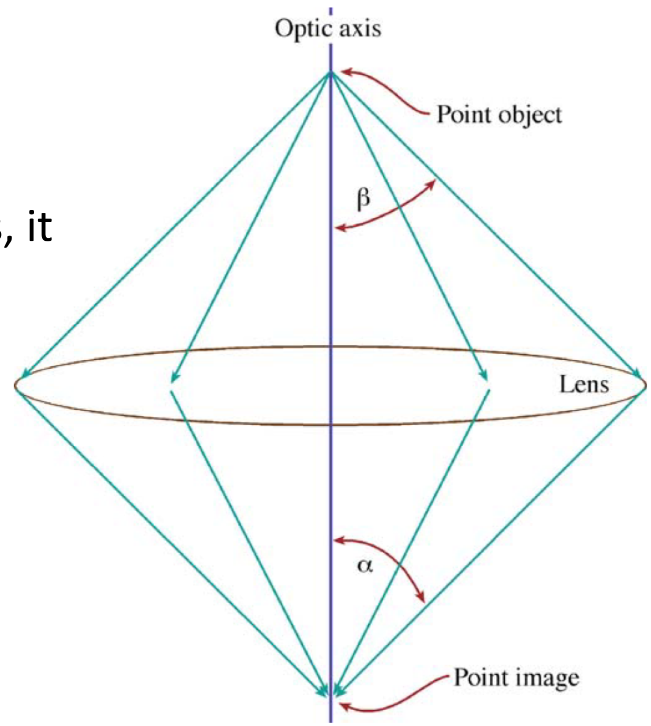

FIGURE 6.1. Image formation by a convex lens. A point object is imaged as a point and the collection angle of the lens is defined relative to the object (β) or the image (α).

Strong lenses enlarge less and reduce more.

Electron Microscopy I
Lecture 05

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Lab course information

Information about the lab course will be uploaded to ILIAS.

Gruppe	Praktikumstag		Name	Vorname	Mail
1	Dienstag	14:00 Uhr	Holz	Nils	ueolh@student.kit.edu
			Seidel	Johanna	johanna.seidel@kit.edu
			Yüksel	Deniz	dyuksel1@hotmail.com
			Lamis	Saeed	ulmty@student.kit.edu
2	Mittwoch	14:00 Uhr	Nurak	Ingrit Sisilia Rosari	ingrit.nurak@kit.edu
			Kraft	Kristian	kristian.kraft@kit.edu
			Schwartz	Arne	arne.schwartz@student.kit.edu
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	KW 5	30.01. + 31.01.2024			

2.2 Lens aberrations in electron optics and their effect on the resolution

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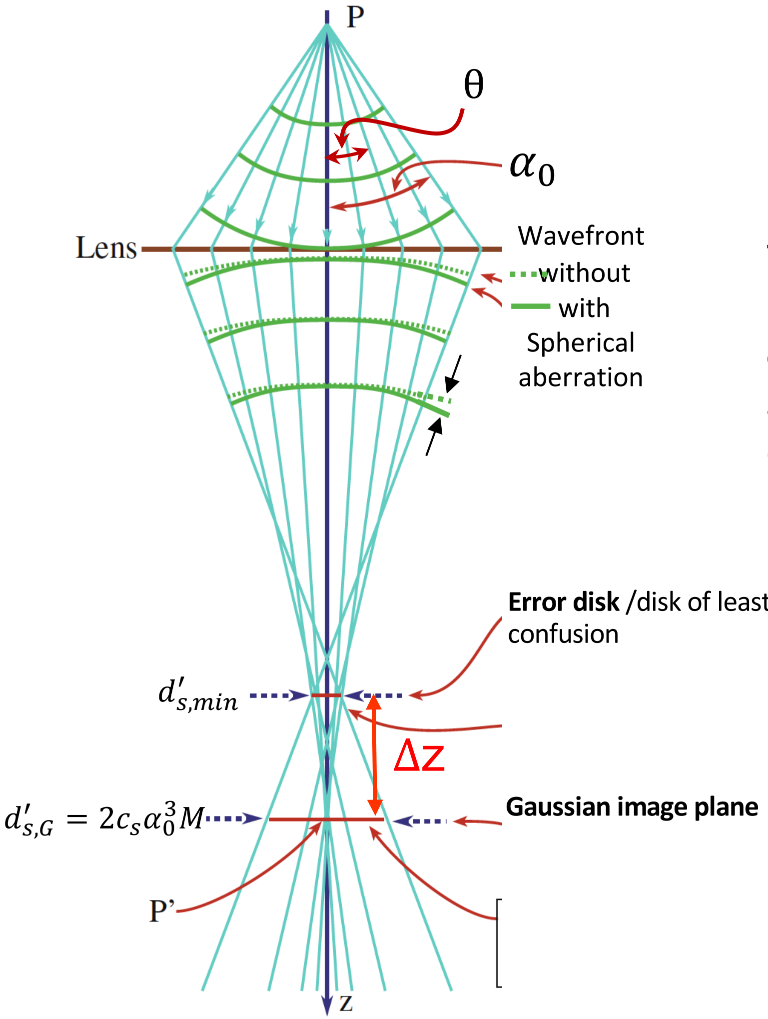
Spherical aberration $PP' = v_P T_C = vT_c \cos\theta = 2\pi \frac{mv}{eB}\left(1 - \frac{1}{2}\theta^2 + \frac{1}{4!}\theta^4 \ldots\right) = L_0(1 - \frac{1}{2}\theta^2 + \ldots)$

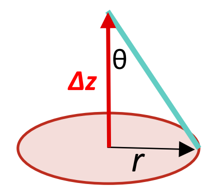


L. Reimer, Transmission Electron Microscopy, Fig.2.13

Shortening the image width by Δz

$$\Delta z = \frac{1}{2} L_o \theta^2$$

The point P is mapped in P' of the Gaussian image plane for paraxial rays with small angle (serves as reference plane). For off-axis rays (large angles), the image width is shortened by Δz. aberration disc / **disc of least confusion** with radius **r** can be calculated.

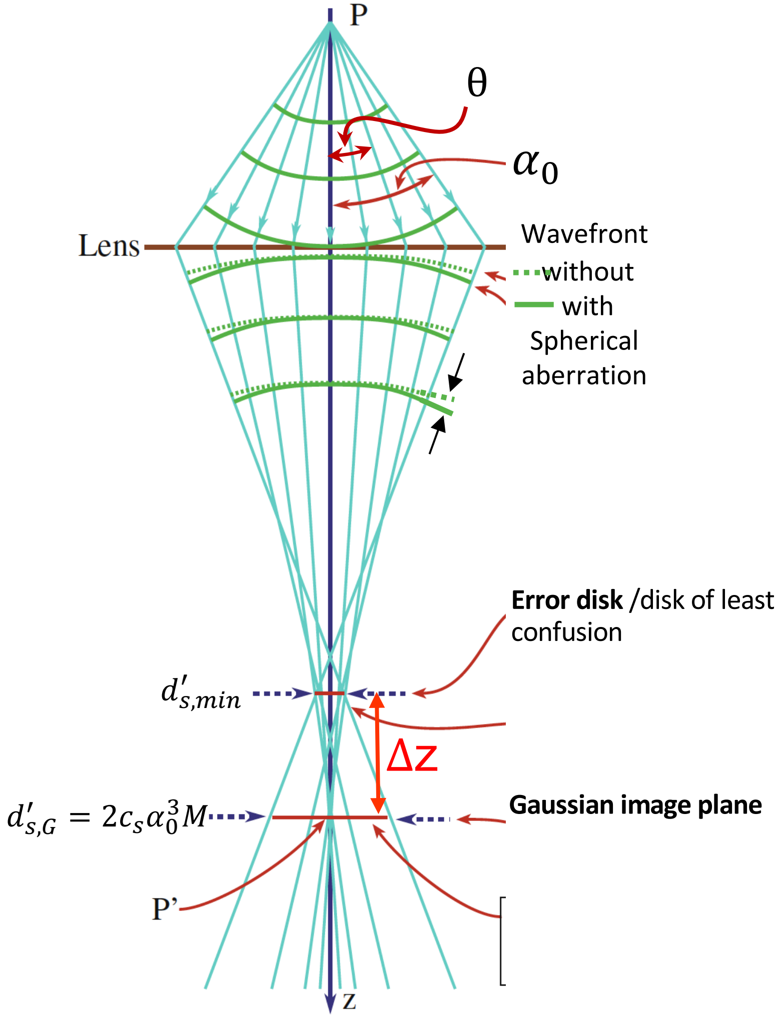


$$r = \Delta z \tan\theta \, M \approx \Delta z \theta M = \frac{1}{2} L_o \theta^3 M = c_s \theta^3 M$$

M: Magnification

Spherical aberration coefficient $c_s = \frac{1}{2} L_0$

Spherical aberration



L. Reimer, Transmission Electron Microscopy, Fig.2.13

Aberration disc diameter in the Gaussian Image layer:

$$d'_S = 2c_s\alpha_0^3M$$

α_0 : Opening (semi) angle of the object side Beam (limited by aperture)

Error slice diameter referred to object plane (without magnification M):

$$d_S = 2c_s\alpha_0^3$$

Minimum aberration disc diameter "disk of least confusion"

$$d'_{s,min} = \frac{1}{2}c_s\alpha_0^3$$

c_s = 1.2 mm, opening angle $\alpha_0 \cong 10^{-2}$ rad (α_0 for 0.25 nm, λ = 2.5 pm)

$$d'_{s,min} = \frac{1}{2}\ 1.2mm\ \ 0.01^3 = 0.6nm$$

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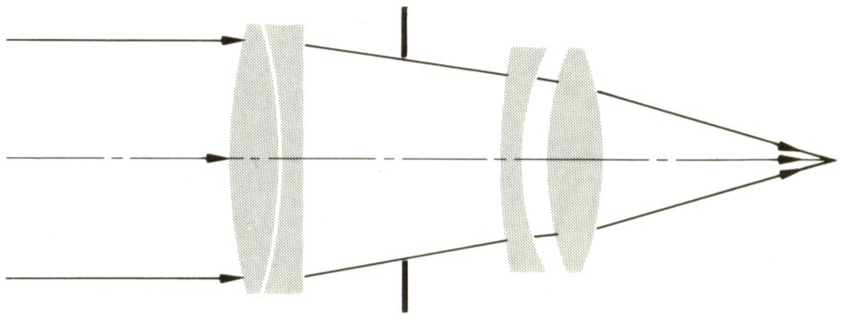
Real electron lenses

Inhomogeneous fields:

- Complex, not always ideal spiral trajectories
- Mostly no complete rotations → Image rotation 180º + additional angle, which depends on the magnetic field, i.e. the magnification

Correction of the spherical abberation in light optics by <u>suitable combination of convex and concave (round) lenses</u>



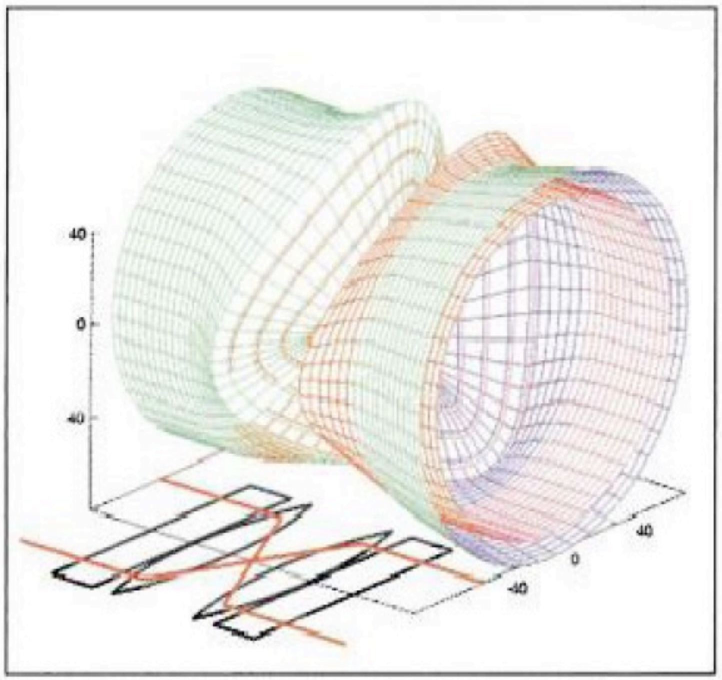
Scherzer Theorem:

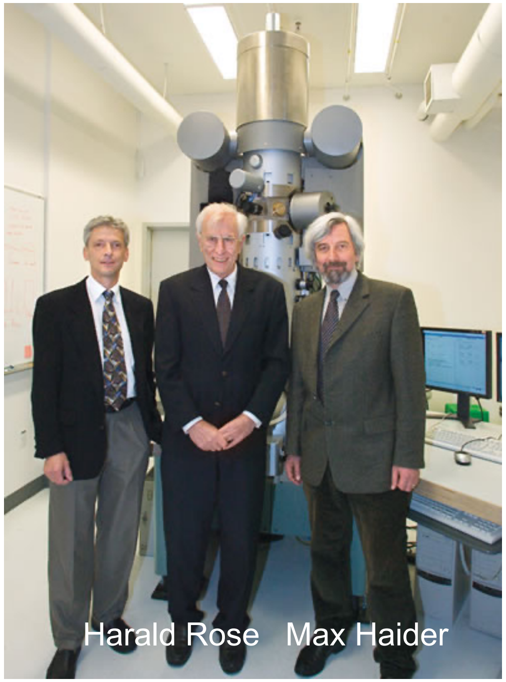
Imaging abberations (spherical and chromatic aberrations) in rotationally symmetric electromagnetic electron lenses are unavoidable!

→ No correction as in light microscopy!

Revolution in electron microscopy: spherical aberration correction since 1997

M. Haider et al, Nature 392, 768 (1998).





- "Double hexapole corrector" for spherical aberration since 1997 based on the theoretical concept of O. Scherzer and H. Rose.
- Experimental realization by Maximilian Haider
- Negative C_s value due to incomplete compensation of higher order errors in hexapole lenses, selective adjustment of small positive and negative C_s values possible

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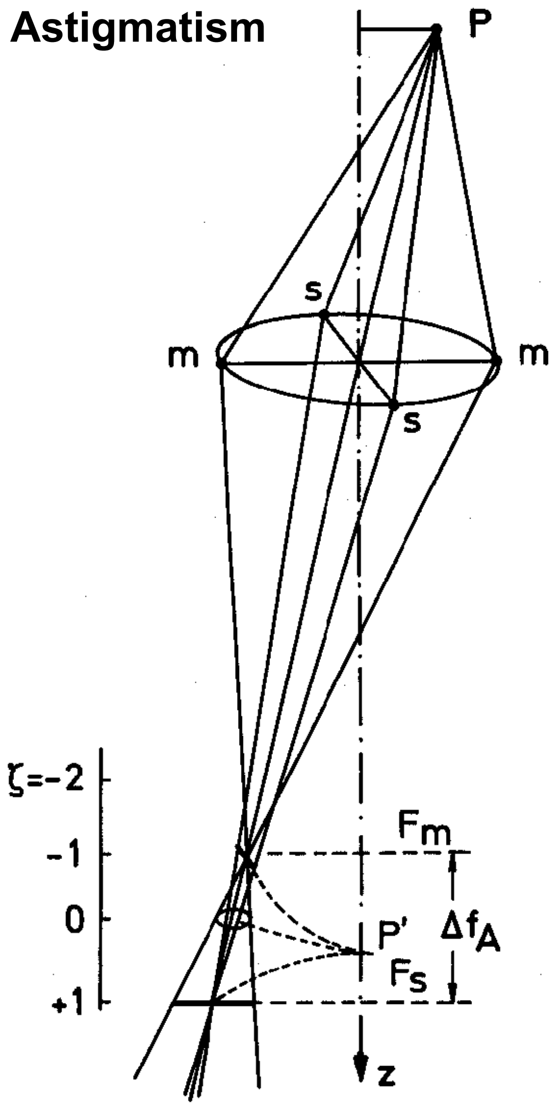
Astigmatism

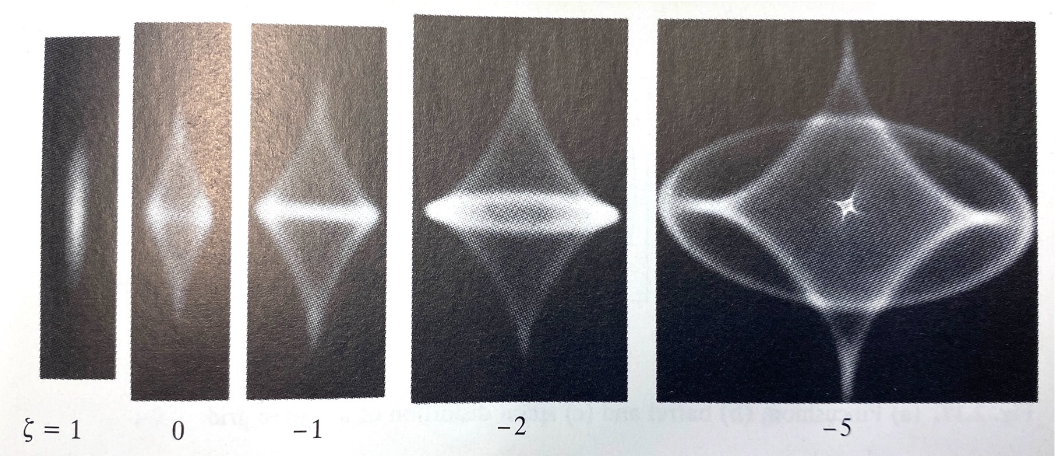


Causes of axial astigmatism:
Deviations of the lens field from rotational symmetry due to cylindrically symmetric field components caused by

- Field variations due to microstructural inhomogeneities of the pole shoe material
- Charges on apertures (lens aperture)
- Charging of the sample

$$d_A = \Delta f_A \alpha_0$$

d_A : Minimum disc of least confusion diameter
Δf_A : Focal length difference for sagittal and meridional level
α_0 : Beam opening angle



L. Reimer, Transmission Electron Microscopy, Fig.2.16 and Fig.2.16

2.2 Lens aberrations in electron optics and their effect on the resolution

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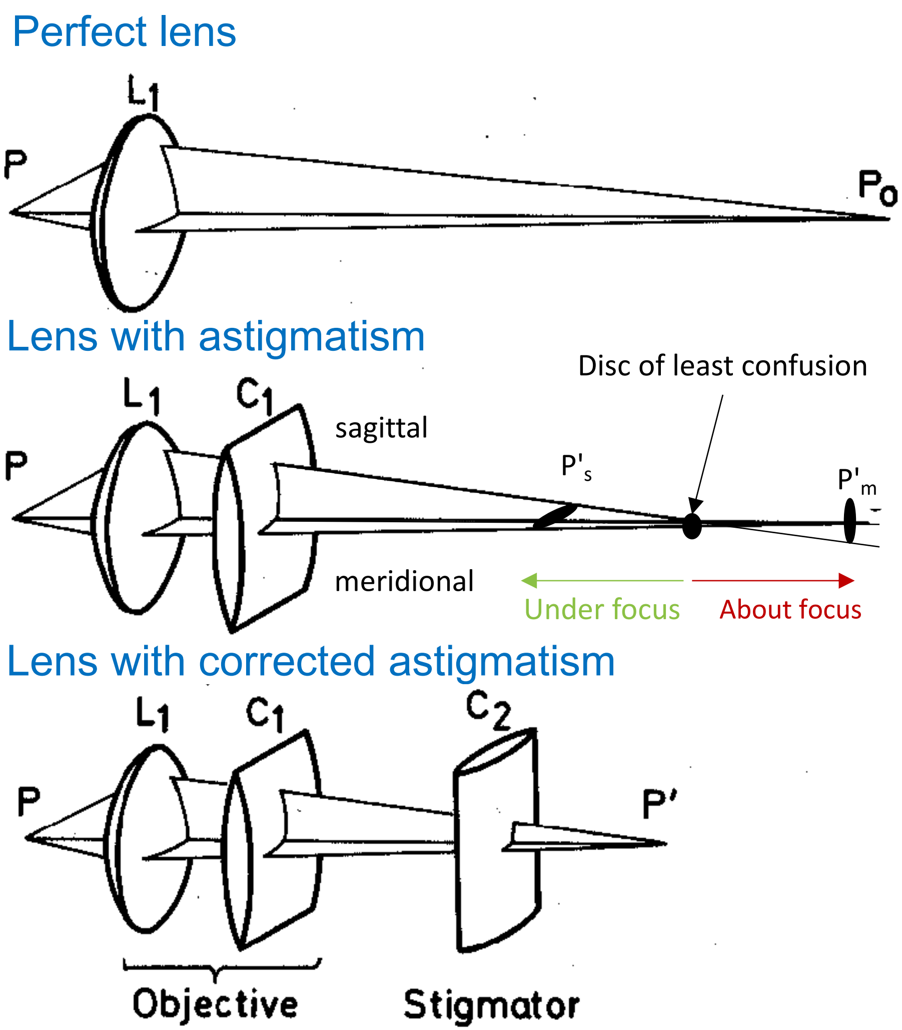
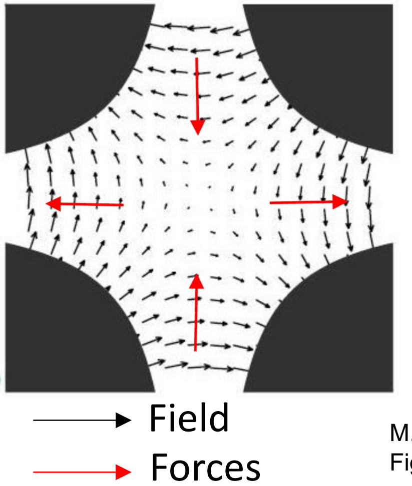
Astigmatism



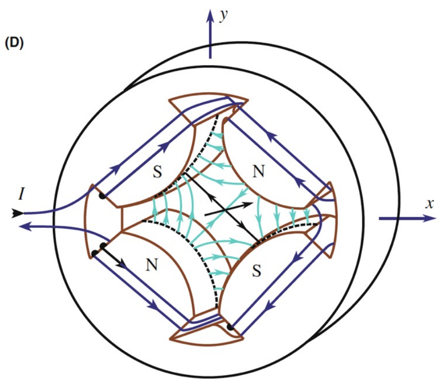
L. Reimer, Transmission Electron Microscopy, Fig. 2.21

Stigmators

Fields & forces acting on the electron beam

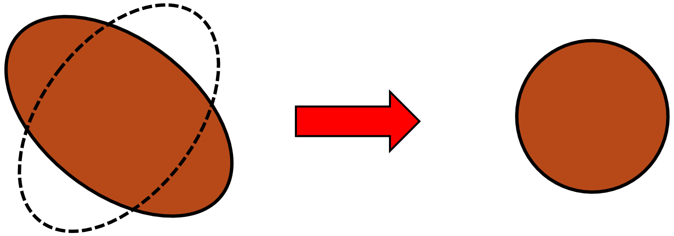


Quadrupole / Octopole lenses



M.De.Graef, Introduction to conventional TEM Fig. 3.20

Beam before and after correction



- There are a total of three stigmators in the transmission electron microscope in the region of the condenser, objective and intermediate lens

2.2 Lens aberrations in electron optics and their effect on the resolution

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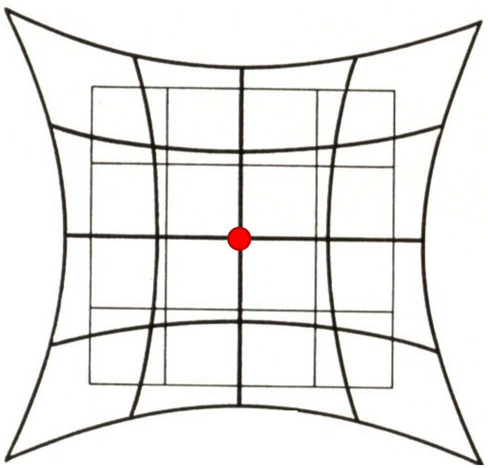
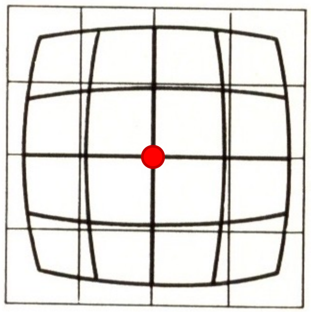
Distortion due to poor lens quality of the projector lens
(last post-magnifying lens):

Cushion distortion



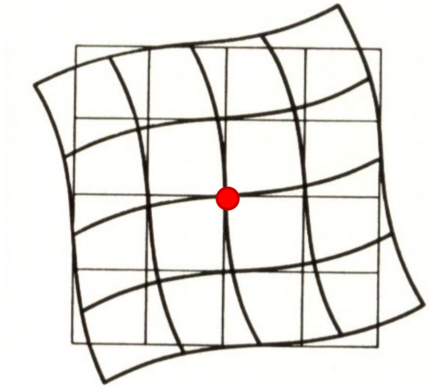
Magnification M **increases** with distance from the optical axis

Barrel distortion



Magnification M **decreases** with distance from the optical axis

spiral distortion



Rotation and distortion of the image

L. Reimer, Transmission Electron Microscopy, Fig.2.17

- → Caution when making precise distance measurements in TEM images
- Correction of distortions is possible by post-processing of the images, since distortions are generally constant in time.

2.2 Lens aberrations in electron optics and their effect on the resolution

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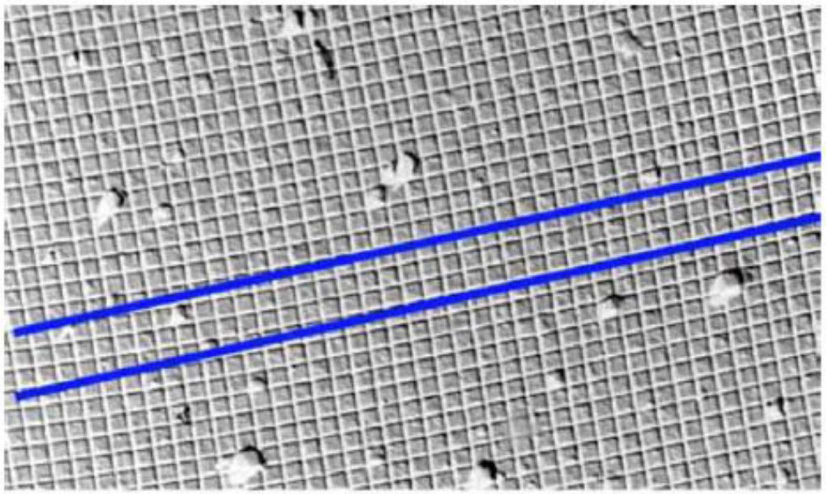
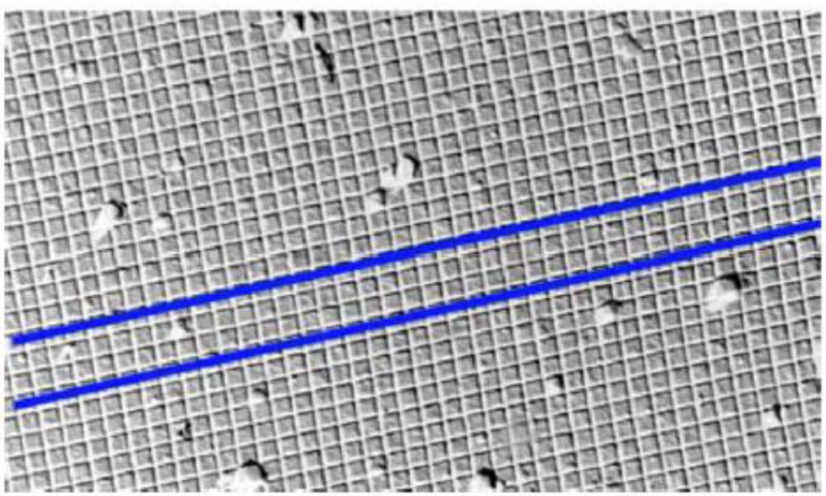
Example: Cushion, barrel and spiral distortion



Before correction

Grating spacing: 460 nm



After correction

V.Kaynig et al, Journal of Structural Biology 171 (2010), 163-173.

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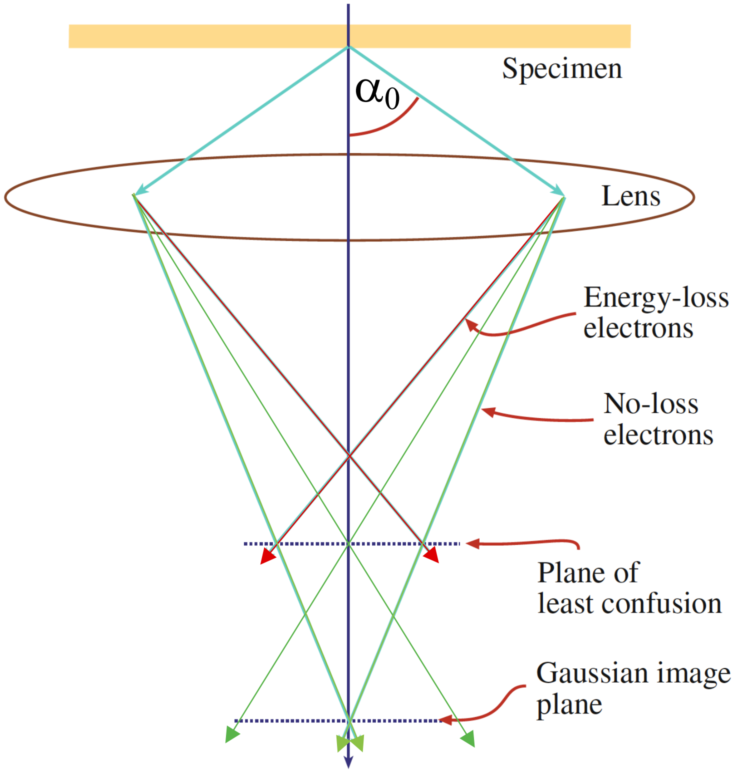
Chromatic aberration



D.B. Williams, C.B. Carter, Transmission Electron Microscopy, Fig. 6.13

Focal length of lens dependent on wavelength

Electrons not ideally monochromatic:

- Energy distribution of electrons at thermal emission, ΔE from 0.3 eV to 2.5 eV depending on electron emitter temperature
- High voltage fluctuations $\Delta U/U \cong 10^{-6}$
- Additional slight focal length variation by lens current fluctuations $\Delta I/I \cong 10^{-6}$

Disc of least confusion minimum diameter

$$d_c = 0.5\Delta f \alpha_0$$

$$\Delta f = C_c \sqrt{\left(\frac{\Delta U}{U}\right)^2 + \left(\frac{2\Delta I}{I}\right)^2 + \left(\frac{\Delta E}{E}\right)^2}$$

C_c : Chromatic aberration constant

Inelastically scattered electrons in the sample with Energy losses of several 100 eV!

Discussion slide for students

$$d_c = 0.5 \Delta f \alpha_0 \qquad d'_{s,min} = \frac{1}{2} c_s \alpha_0^3 \qquad d_A = \Delta f_A \alpha_0$$

Which aberration limits the resolution the most?

a) Spherical aberration
b) Chroatic aberration
c) Astigmatism
d) Influence of post-magnifying lenses (typically 4-5): Multiplication of error disc diameters?

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Imaging aberration

Chromatic aberration $\infty\ \alpha_0$
Spherical aberration $\infty\ \alpha_0^3$

Answer: At small angles, the chromatic aberration predominates, and at large angles, the spherical aberration dominates.

In electron microscopes without corrector:
Limitation of the resolution due to *spherical aberration*

In electron microscopes with spherical aberration (C_s) corrector:
Limitation of the resolution due to *chromatic aberration*

Philips CM 200 FEG/ST transmission ***electron microscope***

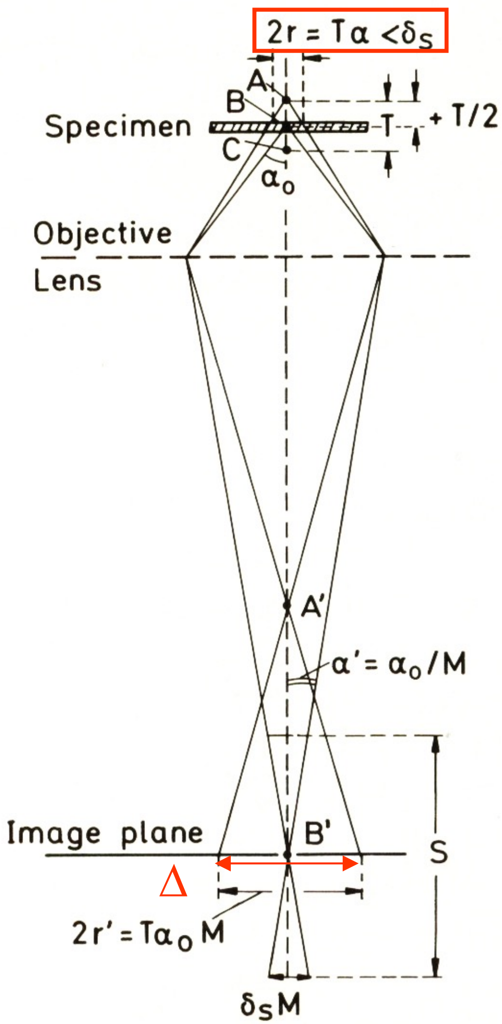
U = 200 kV, C_s = 1.2 mm, C_c = 1.2 mm, ΔE=1eV
α_0 = 0.01 rad

$$2\frac{\Delta I}{I} = 2\times10^{-6} \qquad \frac{\Delta U}{U} = 10^{-6} \qquad \frac{\Delta E}{E} = \frac{1}{200000} = 5\times10^{-6}$$

$$d'_{s,min} = \frac{1}{2}\ 1.2mm\ \ 0.01^3 = 0.6nm$$

$$d_c = 0.6\,\mathrm{mm}\sqrt{10^{-12} + 4\times10^{-12} + 25\times10^{-12}}\,0.01 = 0.033\,\mathrm{nm}$$

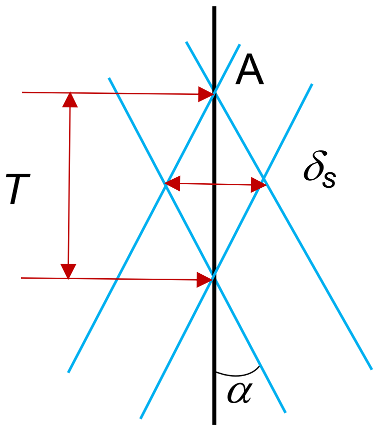
Depth of field T: Maximum extension of an object without loss of resolution in the image



L. Reimer, Transmission Electron Microscopy, Fig.4.23

Point A is focussed with a resolution M δ_s in the image plane

$$T < \frac{\delta_s}{\alpha}$$



δ_s : Resolution

T: Specimen thickness

Δ = M δ_s must be < pixel size (typical for CCD camera: 20 µm)

Typical sample thickness: 10 nm - 1 µm

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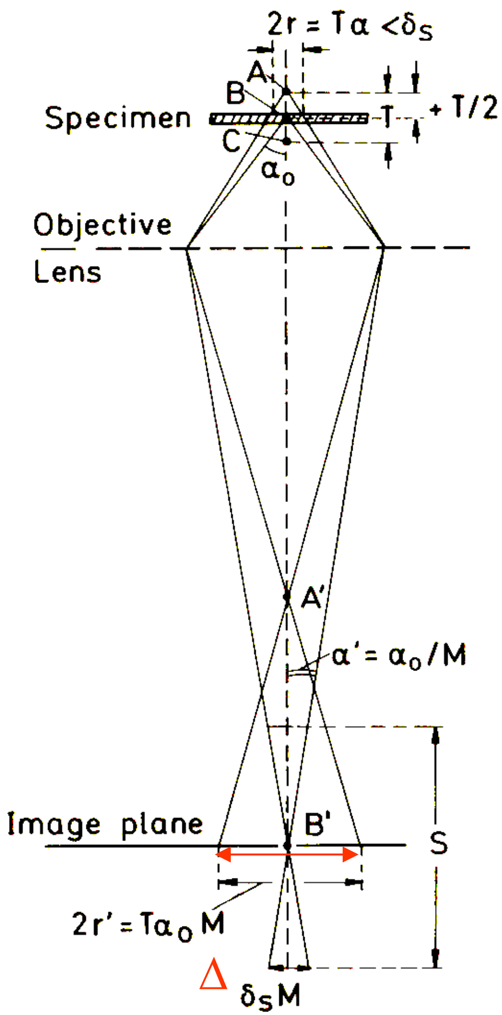
Depth of focus S: Shift in the image plane without loss of sharpness



L. Reimer, Transmission Electron Microscopy, Fig.4.23

δ_s M at $\pm$ S/2 must be smaller than pixel size

$$\delta_s M = \alpha' S \longrightarrow S = \frac{\delta_s M}{\alpha'} = \frac{\delta_s M^2}{\alpha_0}$$

α': aperture angle of the beam on the image side
α_0 : aperture angle of the beam on the object side

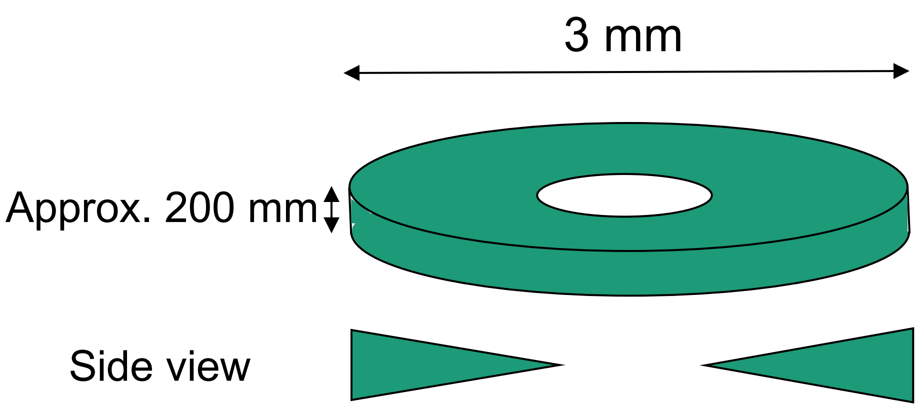
Example:
M = 10^4 , α_0 =0.01 rad, δ_s = 5 nm
(pixel size Δ= 50 mm)
$\longrightarrow$ S = 50 m!

Positioning of different detectors via a larger range possible without loss of resolution (photographic plates, CCD cameras, ..)

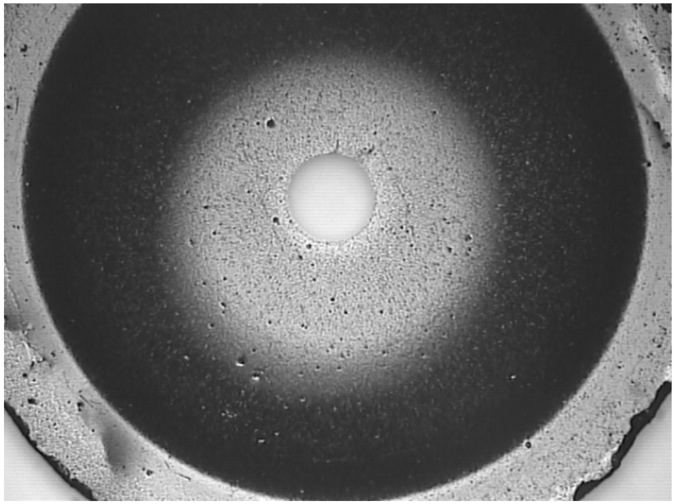
Sample preparation

Goals:

- Electron-transparent sample with a thickness of a few 10 nm for high-resolution TEM up to ~ 100 nm for conventional TEM
- maximum sample thickness depending on the density of the sample material and electron energy used
- Avoiding artifacts through preparation



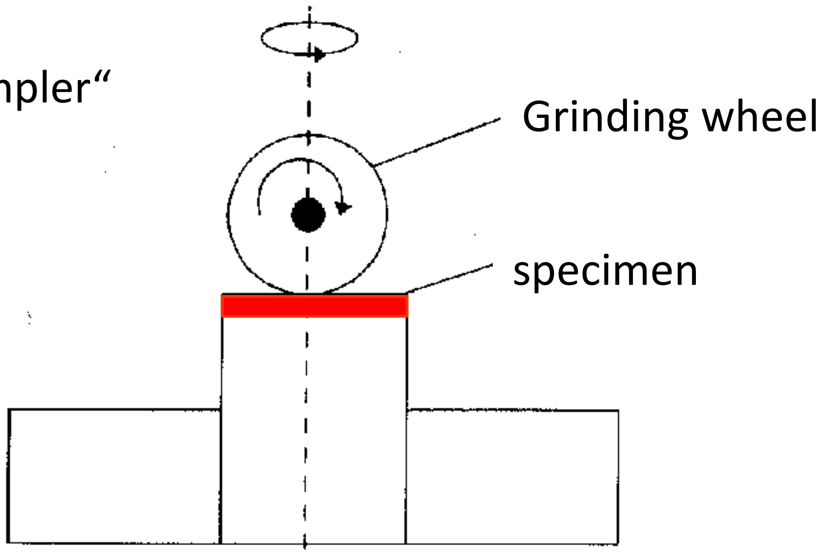
Light microscope image of a TEM sample



- Sawing and drilling out a thin material disk with a diameter of 3 mm
- Surface grinding to a thickness of approx. 200 µm

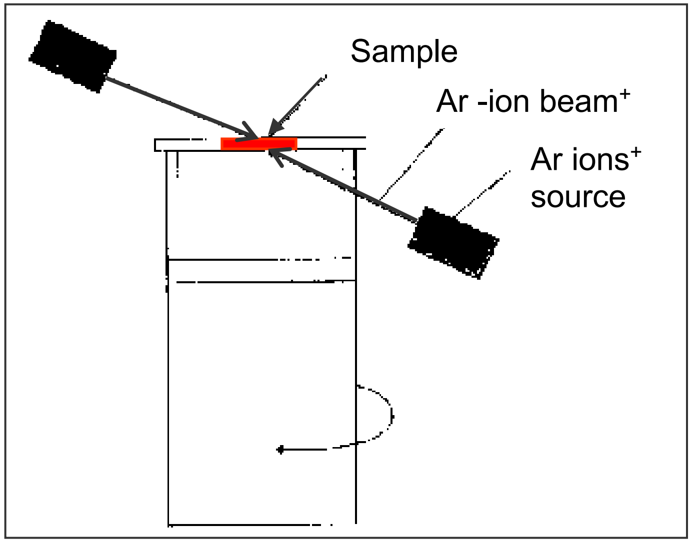
Sample preparation

„dimpler“



E. Hornbogen, B. Skrotzki, Materials microscopy, Fig.2.1

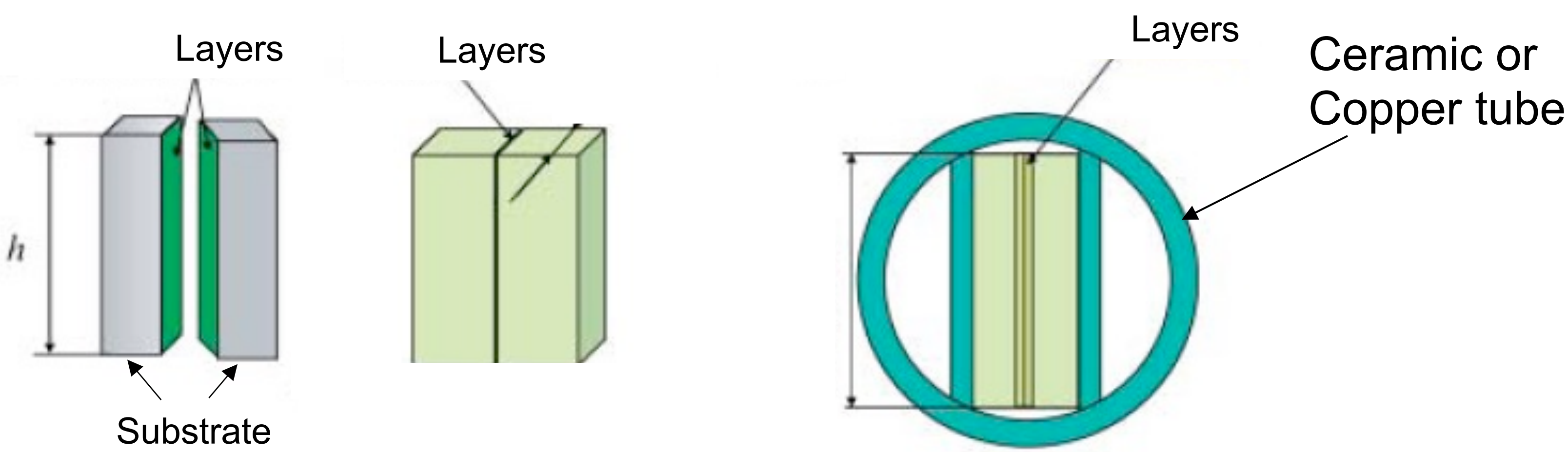
Through grinding with a dimpler up to a thickness of a few µm in the center of the 3 mm slice



E. Hornbogen, B. Skrotzki, Materials microscopy, Fig.2.10

Electron transparency through sputtering with Ar^+ ions with 1 - 5 keV energy under grazing incidence to hole in the center of the sample is created

Cross-section samples: Side view of layer systems



Subsequent preparation as already shown

Other preparation methods:
- Electrochemical / chemical etching (metals, semiconductors)
- Ultramicrotomy ("soft matter": polymers, biological/medical samples)
  Production of thin sections
- ...

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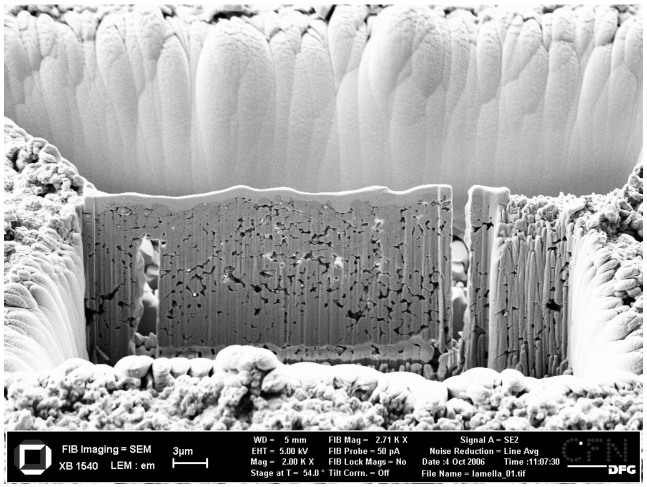
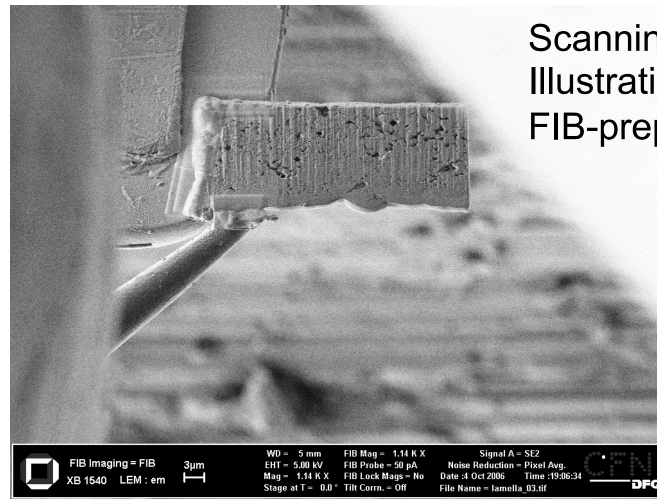
Preparation with a focused ion beam system (FIB)

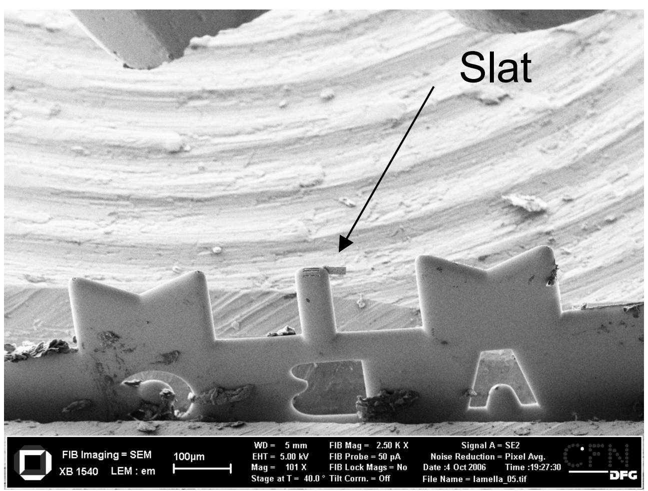
Milling out a thin lamella from a solid sample with a focused Ga -ion beam^{+}





Scanning electron microscopy Illustrations of a FIB-prepared TEM lamella

Erich Müller (LEM)



- Precise target preparation possible
- Implantation of Ga into the sample and thus associated radiation damage

More about FIB and scanning electron microscopy in the in EM II Lecture

Summary: *Lens aberrations in electron optics and their effect on* resolving power

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- Rayleigh criterion and Abbe criterion are two approaches for the description of the resolving power in LM.
- Magnetic lenses are used in electron microscopy, where electrons move on spiral electron trajectories through a rotationally symmetrical inhomogeneous magnetic field (approximation with Glaser's bell field).
- All electrons that pass through an object point P pass through a point P', which can be regarded as the image point of P. All electrons started at a point at different angles θ are thus reunited at a point P_o ' on the z-axis passing through their starting point - as long as their axis-parallel velocity components are the same. This is the case if the angle θ is so small that cos θ is equal to one in a good approximation and thus the Gaussian image plane is defined.
- spherical aberration: For off-axis rays (larger angles), the image width is shortened by Dz. Error slices with radius r can be calculated. An aperture error constant C_s is derived from this. At larger aperture angles, the aperture error increases significantly, as $d_{min} = \frac{1}{2} c_s \alpha_0^3$
- Scherzer's theorem: Imaging errors (spherical and chromatic aberration) are unavoidable in rotationally symmetrical electromagnetic electron lenses. However, a correction for the aperture error was developed in 1997 using a double hexapole lens.
- With axial astigmatism, the points (P_M and P_S) are not points but *focal lines* in the other plane. Instead of a circle, an oval is formed in front of and behind the two focal planes, as each beam of a plane becomes an ellipse and has a different opening angle at each point. Axial astigmatism is corrected with multi-pole lenses. Error disk $d_A = \Delta f_A \alpha_0$
- Projection lenses are usually not of such high quality. This can cause distortions in the image.
- The chromatic aberration: Lens focal length depends on the wavelength, since electrons are not ideally emitted monochromatically, but have an energy distribution due to thermal emission, they are focused with different strengths.
- Depth of field is a measure of the extent of the sharp area in the object space of an imaging optical system.
- Depth of focus is the area in the image plane that ensures a sufficiently sharp image of a focused object. In the TEM, these areas are very large (several meters), so detectors or cameras can be moved over a larger area.

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Beam damage

„direct" beam damage in the microscope

- **Atomic displacement („**knock-on damage“)
- **Elektronenanregungsschädigung** (Radiolyse)

„Indirect" beam damage in the microscope

- **Contamination** *(polymerisation of the Hydro Carbons (C_nH_m) on the sample.)*
- **Heating** (Radius r_0 of the illuminated area is proportional to the temperature increase.)

„Direkte" Strahlenschädigung im Mikroskop

Atom displacement
(„knock-on damage“)

Electron excitation damage
(Radiolysis)

Electron – Atmo nucleus
Interaction

Atom displacement
→ Leads to direct binding dissociation

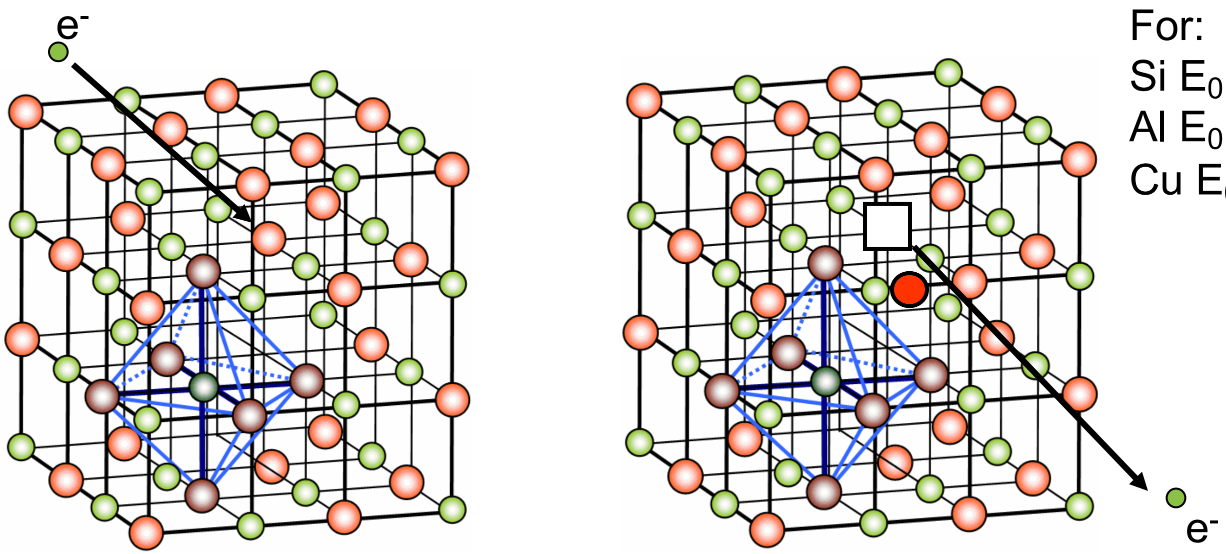
Elektron – valence electron
Interaction

Atom displacement
→ Leads to ionization
--> Leads to local sample heating

„Direct“ radiation damage within the microscope

- atom displacement („knock-on damage“)

Atom displacement damage



Minimale Elektronenenergie
„activation/threshold energy“
For:
Si E_0 > 145 keV
Al E_0 > 170 keV
Cu E_0 > 300 keV

- Formation of vacancies and interstitial atoms when binding energy of the atom < energy transferred during impact

- Formation of extensive defects (stacking faults, cavities) with high void and interstitial atom concentrations

→ Limitation of the irradiation time of an object, reduction of electron energy

Atomic displacement damage („knock-on damage“)

Grain boundary in Si, 300 keV Elektronenenergie

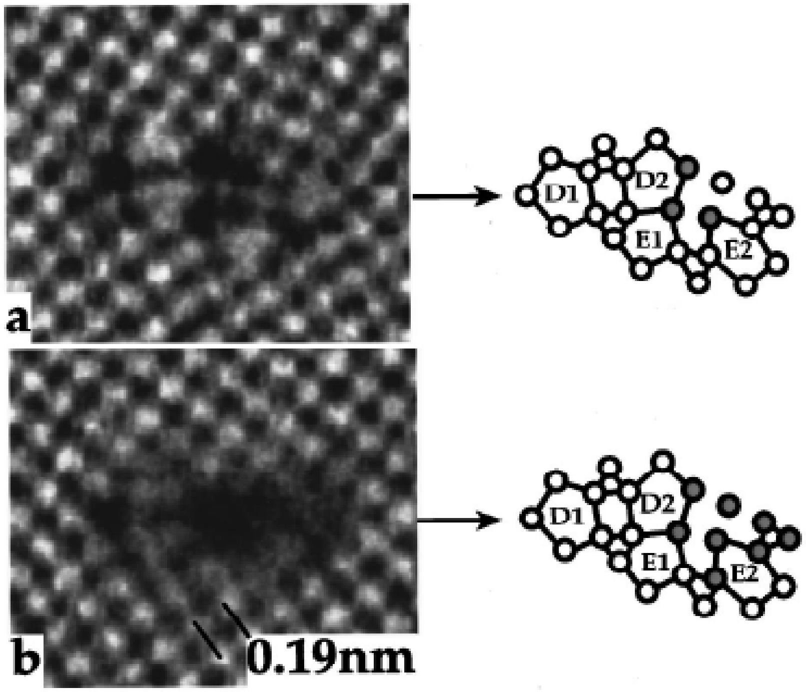
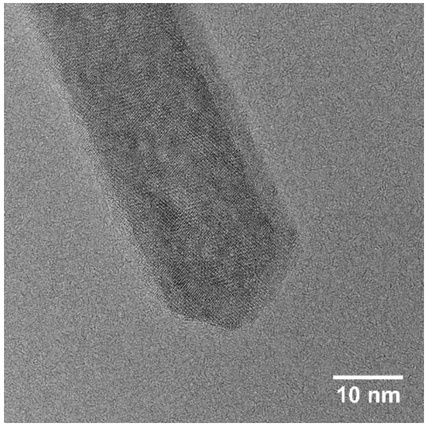
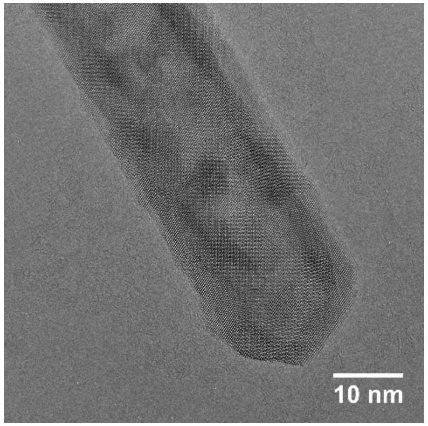


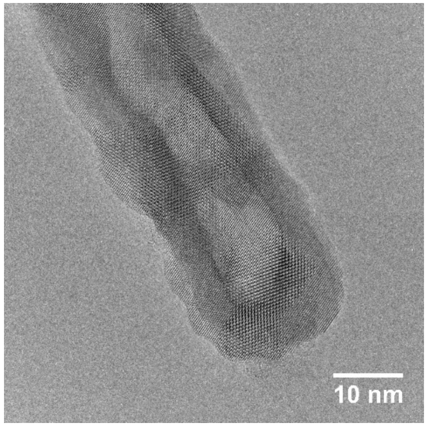
FIG. 1. Z-contrast images and derived structures of the $\Sigma = 25$ {710} ⟨001⟩ symmetric tilt boundary at two stages of exposure to electron irradiation: (a) a nearly unaffected core with all columns visible but those shaded showing reduced intensity; (b) a partially affected core with several columns appearing darker.

A. Maiti et al., Appl. Phys. Lett. 75, 2380 (1999)

Electron-beam induced transformation of a nanocolumn into a nanotube



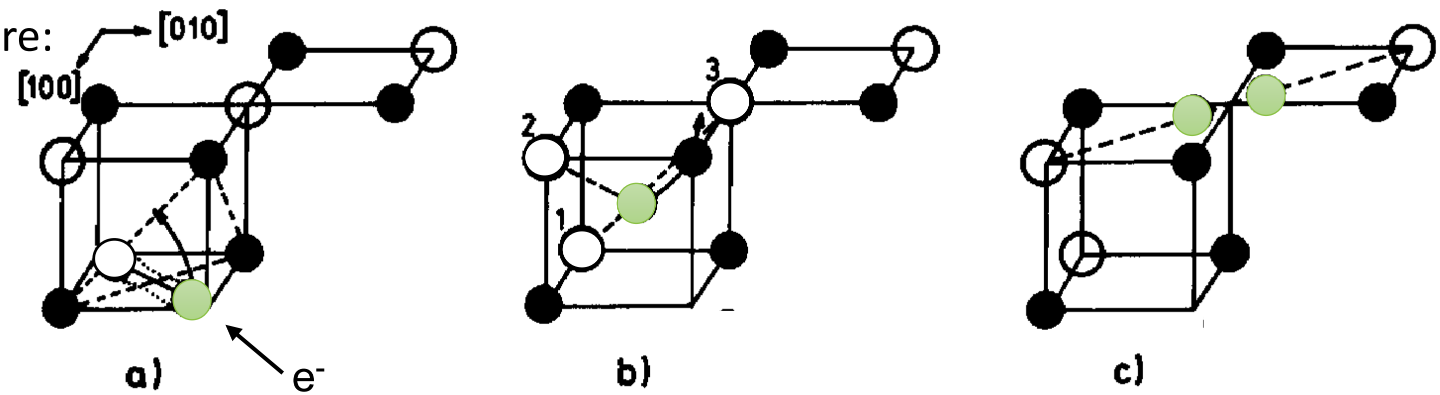




R. Popescu (LEM)

Radiolysis in electrical isolating materials

Example
NaCl structure:



L. Reimer, Transmission Electron Microscopy, Fig. 11.8

Cl Na

Structural rearrangement through the excitation of electrons in the sample material into higher energetic or non-binding states

➔ Reduction by cooling the sample in a sample holder cooled with liquid nitrogen

➔ Increase in electron energy (probability of an electronic excitation process decreases)

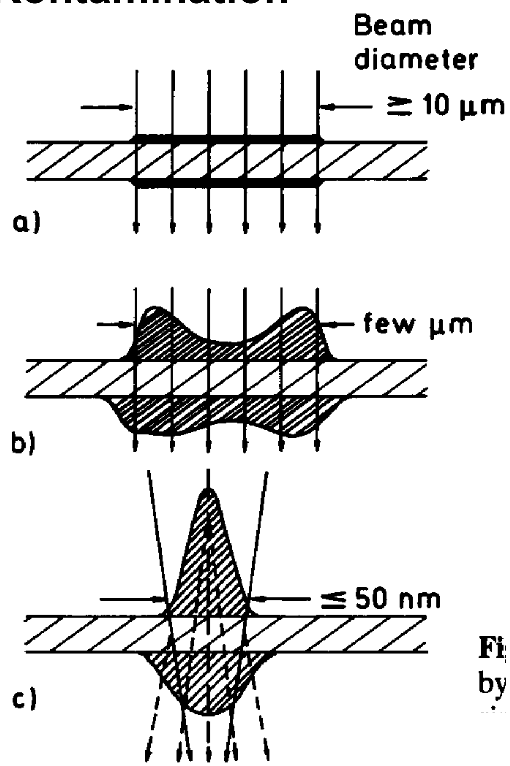
Radiolysis in electrically conductive samples is insignificant.

Radiolysis is dominant in ionic crystals, (non-conductive) ceramic oxides, polymers, and samples from life sciences.

2.3 Beam damage

„Indirct" beam damage
- Contamination
- heating

Kontamination



L. Reimer, Transmission Electron Microscopy, Abb. 10.10

Polymerization of hydrocarbon molecules (C_nH_m) from:

- Residual gas atmosphere (e.g., pump oils, vacuum grease from rubber seals)
- Contamination from residues of the sample preparation

on the sample surface due to the influence of the electron beam

Remedy:
- Extremely clean work (gloves)
- Cleaning in Ar/O plasma
- Improvement of the vacuum through a cold trap at liquid nitrogen temperature (condensation of C_nH_m)
- Liquid nitrogen cooling holder (reduction of surface diffusion)

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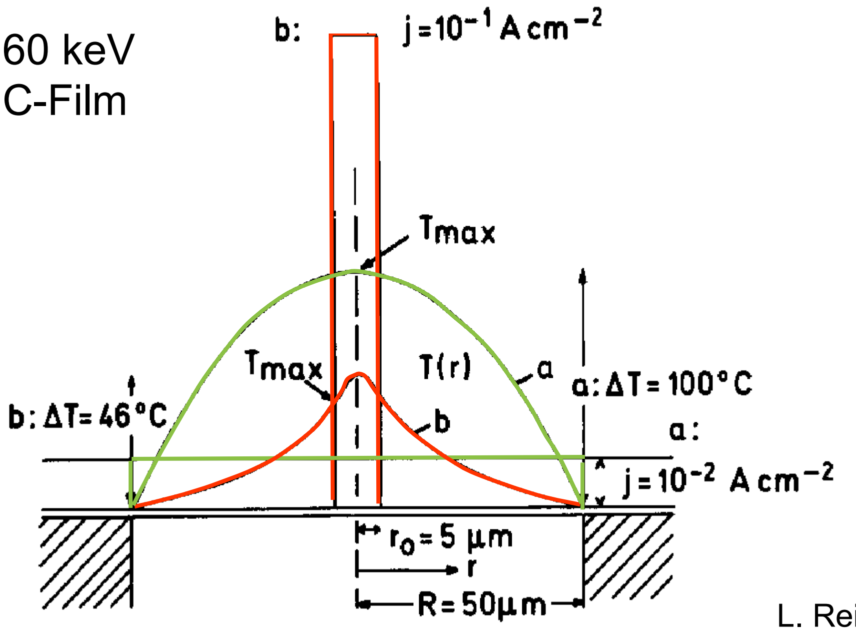
Sample heating

60 keV
C-Film



- There are hardly any experimental data available.
- The thermal conductivity of the sample and thermal coupling to the sample holder are crucial.
- Melting of small particles and small inclusions in the microscope observed without heating.
- Calculation: dissipated heat by electron beam = heat dissipated through thermal conduction.

L. Reimer, Transmission Electron Microscopy, Abb. 11.2 und Tab. 11.3

Table 11.3. Rise of temperature ΔT in the center of a circular diaphragm ($R = 50$ μm) covered with a supporting film and irradiated with 100 keV electrons.

	Uniform illumination	Small-area illumination
Substance	$R = 50\ \mu$m, $j = 100$ A m^{-2}	$r_0 = 0.5\ \mu$m, $j = 10^4$ A m^{-2}
Formvar	62°C	6°C
Glass (SiO)	27°C	2.5°C
Metal (Cu)	0.3°C	0.03°C

The radius r_0 of the illuminated sample area is crucial for the temperature increase.

Summary chapter 2

Laboratory for Electron Microscopy

- Rayleigh criterion and Abbe criterion are two approaches for the description of the resolving power in LM.
- Magnetic lenses are used in electron microscopy, where electrons move on spiral electron trajectories through a rotationally symmetrical inhomogeneous magnetic field (approximation with Glaser's bell field).
- All electrons that pass through an object point P pass through a point P', which can be regarded as the image point of P. All electrons started at a point at different angles θ are thus reunited at a point P_o ' on the z-axis passing through their starting point - as long as their axis-parallel velocity components are the same. This is the case if the angle θ is so small that cos θ is equal to one in a good approximation and thus the Gaussian image plane is defined.
- The structure of a Transmission Electron Microscope (TEM) is comparable to that of a light microscope: "light source," condenser, extensive illumination of the sample, imaging lens system, image plane with a camera.
- In Scanning Transmission Electron Microscopy (STEM) mode, illumination is done with a focused electron beam that is scanned across the sample. The image is generated by the locally detected electron intensity, which controls the brightness of the corresponding pixel.
- An imaging lens system is not necessary for image formation. However, images in TEM and STEM modes are comparable (see Chapter 4) because the interaction of electrons with the sample determines the image.
- Images (real space) and diffraction patterns (reciprocal space) of the same sample area can be obtained by extending the focal length of the intermediate lens. Diffraction patterns provide local crystal structure information and allow the sample to be selectively oriented with respect to the electron beam, for example, in a two-beam condition or zone axis orientation.
- In the back focal plane of the objective lens, there are apertures with different diameters that determine the imaging mode.
- "Conventional" TEM bright-field or dark-field images (images with one reflection) are created by selecting a smaller aperture. A large aperture must be used for high-resolution TEM images, so that electrons from at least 2 reflections interfere in the image.

Summary chapter 2

- A homogeneous magnetic field focuses electrons, except for electrons moving parallel to the optical axis.
- Real electron lenses, therefore, create inhomogeneous magnetic fields.
- Magnetic and electrostatic round lenses for electrons are subject to lens aberrations that depend on the object-side opening angle α_0 of the beam bundle.
- The resolution of electron optical systems is limited by lens errors, not by the electron wavelength. The opening error is dominant at large α_0 . The aberration disc diameter is given by $1/2\, c_s \alpha_0^3$, the spherical aberration constant. Modern microsciopes include a corrective lens system for the spherical aberration .
- Other lens aberrations include chromatic aberration and astigmatism, with astigmatism routinely corrected in the microscope.
- Samples for TEM must be very thin as electrons strongly interact with solids. Maximum sample thickness depends on the electron energy and material density. Typical maximum thicknesses are 1 µm for "conventional" and a few 10 nm for high-resolution TEM.
- Various techniques exist for sample preparation, depending on the material—purely mechanical, chemical, electrochemical, or with a focused ion beam for "target preparation." The goal is to prepare the sample material free from artifacts.
- The interaction of the sample with electrons can cause damage in the microscope. Direct damage (displacement damage and radiolysis) and indirect damage (contamination and sample heating) are distinguished.

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