

# Electron Microscopy I Lecture 03

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### Content

#### 1. From light microscopy to electron microscopy

- 1.1 Light and matter waves
- 1.2 Fundamentals of optical imaging: geometrical optics
- 1.3 Wave optics: Abbe's theory of imaging, Fourier optics
- 2. Practical aspects of transmission electron microscopy (TEM) and scanning transmission electron microscopy (STEM).
- 2.1 Design and operation of a transmission electron microscope
- 2.2 Lens aberrations in electron optics and their effect on the resolution
- 2.3 Specimen preparation
- 2.4 Radiation damage
- 3. Electron diffraction in the solid state/kinematic diffraction theory.
- 4. Contrast formation and practical examples of the imaging of crystalline objects in solid state and materials research.
- 5. Dynamic electron diffraction
- 6. Crystal lattice imaging/high resolution electron microscopy (HRTEM)
- 7. Scanning transmission electron microscopy
- 8. Electron holography
- 9. Transmission electron microscopy with phase plates

#### Review





Slit width << wavelength TEM: atomic nuclei=slits and starting point of our elementary spherical wave









#### **Objective aperture: High resolution or "conventional" TEM imaging**



N: Zero beam (unscattered electrons)

High resolution: Interference of at least 2 reflexes → Lens aperture with large diameter

Conventional imaging: Use of only <u>one</u> reflex (zero beam or a Bragg reflex)

 Lens aperture with small diameter





N on optical axis

#### Centered dark field image



Bright field imaging: Selection of the zero beam with small aperture

Darkfield imaging: Selection of a Bragg reflex

Centered darkfield imaging: Tilting the direction of incidence so that Bragg Reflex is on the optical axis

 $\rightarrow$  better resolution of the image because now diffracted rays lie on the well adjusted optical axis.







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



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#### **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  $\alpha_0$ . 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  $\alpha_0$  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  $2\alpha_0$  For electrons scattered into different angular ranges • Scattering angle ranges can also be changed by camera length of the imaging lens system Sample BE HAADE

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



#### **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, thermal diffuse scattering predominates, which is proportional to Z<sup>2</sup> (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



- 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

the second secon

Special specimen holder: (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) 150 µm MEMS based heating chips Very small heating zone Allows very fast heating and cooling rates ۰ Real-time experiments in TEM possible For direct observations of material changes when external stimuli is applied.

http://denssolutions.com

### Summary after lecture 2



- 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.



### 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  $\lambda$ 









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 resolving power can be achieved by an immersion fluid between lens and object with refractive index  $n_i > 1$ 

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



#### Magnetic lenses



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)









#### Focusing of electrons with velocity $\vec{v}$ by <u>homogeneous</u>

magnetic field for  $\theta \neq 0$ 

Lorentz Kraft:

$$\vec{F}_L = e(\vec{v} \times \vec{B})$$

Division of the velocity of an electron into two components:







 $v_p = v \cos \theta$  Velocity component parallel to  $\vec{B}$ 

 $v_s = v \sin \theta$  Velocity component perpendicular to  $\vec{B}$ 

D.B. Williams, C.B. Carter, Transmission Electron Microscopy, Fig.6.9









 $v_p$ : Movement parallel to the optical axis Electrons are not focused.

 $v_s$ : The Lorentz force acts as a centrifugal <u>force</u>:  $\vec{F}_z = \vec{F}_L$ 

 $m\frac{v_s^2}{r} = ev_s B \longrightarrow r = \frac{mv_s}{eB}$ 

From P to P':

Spiral path with perimeter:  $2\pi r$ 

Period of one circulation: T<sub>C</sub>

 $T_c = \frac{2\pi r}{v_s} = \frac{2\pi m}{eB}$ 

Movement of electrons on spiral paths

Regardless of the speed!







Intersection of the electron trajectories in P'

$$PP' = v_P T_C = v T_c \cos \theta = 2\pi \frac{mv}{eB} \left( 1 - \frac{1}{2}\theta^2 + \frac{1}{4!}\theta^4 \dots \right) = L_0 (1 - \frac{1}{2}\theta^2 + \dots)$$

 $L_0$ : distance for paraxial trajectories ( $P'_0$  is "image point" for small angles  $\theta$ )

Shortening the distance PP' for larger  $\theta$  by

$$PP' = v_P T_C \approx L_0 (1 - \frac{1}{2}\theta^2) = L_0 - \frac{1}{2}\theta^2 L_0 = L_0 - \Delta z$$



Electrons incident parallel to  $\vec{B}$  are not focused: Focal length f =  $\infty$ Reminder of lecture 1: "large" angles are few degrees in TEM



In reality, electromagnetic lenses have a **rotationally symmetrical inhomogeneous field**, e.g. with the Glaser bell field with B<sub>z</sub>



$$B_z = \frac{B_0}{1 + \left(\frac{z}{a}\right)^2}$$

z: coordinate parallel to the optical axis

 $B_0$ : Maximum of the field component  $B_z$  in the center of the lens a: Half width of  $B_z$ 

The Glaser's bell field has the advantage that with it the most important properties, (positions of the focal points and main planes) can be calculated.

B<sub>z</sub> is sufficient to calculate the paraxial rays assuming the Gaussian law for magnetic fields. (Book: L.Reimer pages 26-33)

$$k^2 = \frac{eB_0^2 a^2}{8m_0 U^*}$$

**k:** dimensionless camera constant (includes all "operating parameters")

L. Reimer, Transmission Electron Microscopy, Fig.2.5



Focusing of axis-parallel electron trajectories by rotationally symmetric **inhomogeneous field**, e.g. Glaser's bell field with B<sub>z</sub>



L. Reimer, Transmission Electron Microscopy, Fig.2.9









Error slice diameter in the Gaussian Image layer:

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

 $\alpha_0$ : 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 error disc diameter "disk of least confusion"

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

c<sub>s</sub> = 1.2 mm, opening angle  $\alpha_0 \cong 10^{-2}$  rad ( $\alpha_0$  for 0.25 nm,  $\lambda$  = 2.5 pm)

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



#### **Real electron lenses**

Inhomogeneous fields:

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

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



#### **Scherzer Theorem:**

Imaging errors (aperture 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<sub>s</sub> value due to incomplete compensation of higher order errors in hexapole lenses, selective adjustment of small positive and negative C<sub>s</sub> values possible





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Electron Microscopy I







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

- $\rightarrow$  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.









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 emitter temperature
- High voltage fluctuations  $\Delta U/U \cong 10^{-6}$
- Additional slight focal length variation by lens current fluctuations  $\Delta I/I \cong 10^{-6}$

Error discs with minimum diameter

 $d_c = 0.5\Delta f \alpha_0$ 

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

 $C_{\rm c}$  : Chromatic error constant

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



### Discussion slide for students

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

Which aberration limits the resolution the most?

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



#### **Imaging error**

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Chromatic error \propto \alpha_0
Spherical aberration \propto \alpha_0^3
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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 error* 

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

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U = 200 kV, C_s = 1.2 mm, C_c = 1.2 mm, DE=1eV \alpha_0 = 0.01 rad
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$$2\frac{\Delta I}{I} = 2 \times 10^{-6} \qquad \frac{\Delta U}{U} = 10^{-6} \qquad \frac{\Delta E}{E} = \frac{1}{200000} = 5 \times 10^{-6}$$
$$d'_{s,min} = \frac{1}{2} \ 1.2mm \quad 0.01^3 = 0.6nm$$
$$d_c = 0.6 \ \mathrm{mm} \sqrt{10^{-12} + 4 \times 10^{-12} + 25 \times 10^{-12}} \ 0.01 = 0.033 \ \mathrm{nm}$$

the image plane

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





Typical sample thickness: 10 nm - 1 mm

 $\widecheck{\alpha}$ 

L. Reimer, Transmission Electron Microscopy, Fig.4.23

B'

δsM

 $\alpha' = \alpha_0 / M$ 

 $2r = T\alpha < \delta_S$ 

Specimen m

Objective

Image plane

 $2r' = T\alpha_0 M$ 

Lens

+ T/2



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



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

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

 $\alpha'$ : aperture angle of the beam on the image side  $\alpha_0$ : aperture angle of the beam on the object side

Example:  $M = 10^4$ ,  $\alpha_0 = 0.01$  rad,  $\delta_s = 5$  nm (pixel size  $\Delta = 50$  mm)  $\longrightarrow$  S = 50 m!

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

L. Reimer, Transmission Electron Microscopy, Fig.4.23



- Rayleigh criterion and Abbe criterion are two approaches for describing resolution power in LM.
- Electron microscopy uses magnetic lenses where electrons move on spiral electron trajectories through a rotationally symmetric inhomogeneous magnetic field (approximation with Glaser's bell field).
- All electrons passing through an object point P pass through a point P', which can be regarded as the image point of P. All electrons started in a point at different angles  $\theta$  are thus reunited in a point P<sub>o</sub> ' of the z-axis passing through their starting point as long as their axis-parallel velocity components are equal. This is the case when the angle  $\theta$  is so small that  $\cos \theta \approx 1$  to a good approximation and so the Gaussian image plane is defined.
- Spherical aberration: For off-axis rays (larger angles), the image width is shortened by Dz. Error discs with radius r can be calculated. From this, an spherical aberration error constant C<sub>s</sub> is derived. For larger aperture angles, the spherical aberration error dominates, since  $d_{min} = \frac{1}{2}c_s\alpha_0^3$ .
- Scherzer theorem: aberrations (spherical and chromatic errors) are unavoidable in rotationally symmetric electromagnetic electron lenses. But: With a double hexapole lens, a correction for the spherical aberration was developed in 1997.
- In the case of axial astigmatism, the points ( $P_M$  and  $P_s$ ) are not points but *focal lines in the* respective other plane. In front of and behind the two focal planes, an oval is formed instead of a circle, since each ray bundle of a plane becomes an ellipse and has a different aperture angle in each point. Multipoll lenses are used to correct axial astigmatism. Error disc:  $d_A = \Delta f_a \alpha_0$
- Projection lenses are usually not of such high quality. These can be the cause of distortions in the image.
- The chromatic error: Lens focal length depends on the wavelength, since electrons are not ideally emitted monochromatically, but have an energy distribution due to thermal emission, therefore they are focused differently.
- Depth of field is a measure of the extent of the sharp area in the object space of the 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), detectors or cameras can be shifted over a large area.