Electron Microscopy I
Lecture 02

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Content

Lab Course

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4 Exercises

1. Basic imaging modes of transmission electron microscopy I.
Sample: Semiconductor heterostructure

2. Basic imaging modes of transmission electron microscopy II
Sample: Polycrystalline metal alloy
Imaging of defects

3. Scanning transmission electron microscopy (STEM)
Sample: Dispersion strengthened aluminum, silicon

4. High-resolution transmission electron microscopy, digital image processing. and image simulation
Sample: Semiconductor heterostructure: GaAs layer on Si substrate

Registration **until 07.11.2023 at the latest:**

Study and Teaching → Electron Microscopy I → Lab Course Registration

Anmeldung Übungen EM 1
Bitte melden Sie sich zu den Übungen bis zum 4.11.2021 über diesen Link an.

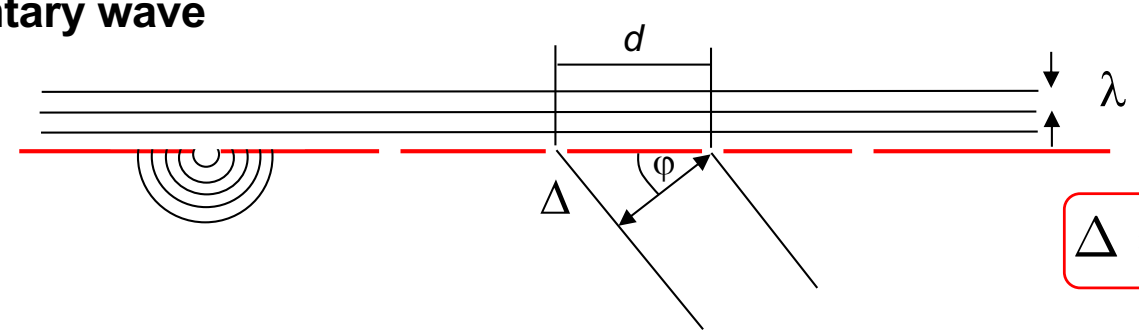
← LINK to registration form
http://www.lem.kit.edu/praktikumsanmeldung.php

General description of a wave by location and time dependent amplitude with wavenumber vector $\vec{k}$, wavelength λ and frequency ω

$$\psi(\vec{r},t) = \psi_0 \exp\left(i(\vec{k}\vec{r} - \omega t)\right)$$ Electron wave function $$|\vec{k}| = \frac{1}{\lambda}$$

- Model object slit grid, illumination by monochromatic, coherent light
  Slit width << wavelength (corresponds to situation in TEM)
  In TEM: atomic nuclei 10^{-15} m vs. wavelength 10^{-12} m
- TEM: Crystal as three-dimensional lattice with "infinitely" large "number of slits" (atoms)
- TEM: steady state → no time dependence

Huygens-Fresnel principle: each point of a wave front is starting point of an elementary wave



$$\Delta = d \sin\varphi$$

Constructive interference
Destructive interference
Δ path length difference

$$\Delta = d \sin\varphi = n\lambda$$
$$\Delta = d \sin\varphi = (2n+1)\frac{\lambda}{2} \qquad n = 0, \pm 1, \pm 2, \ldots$$

1.3 Wave optics: Abbe's theory of imaging

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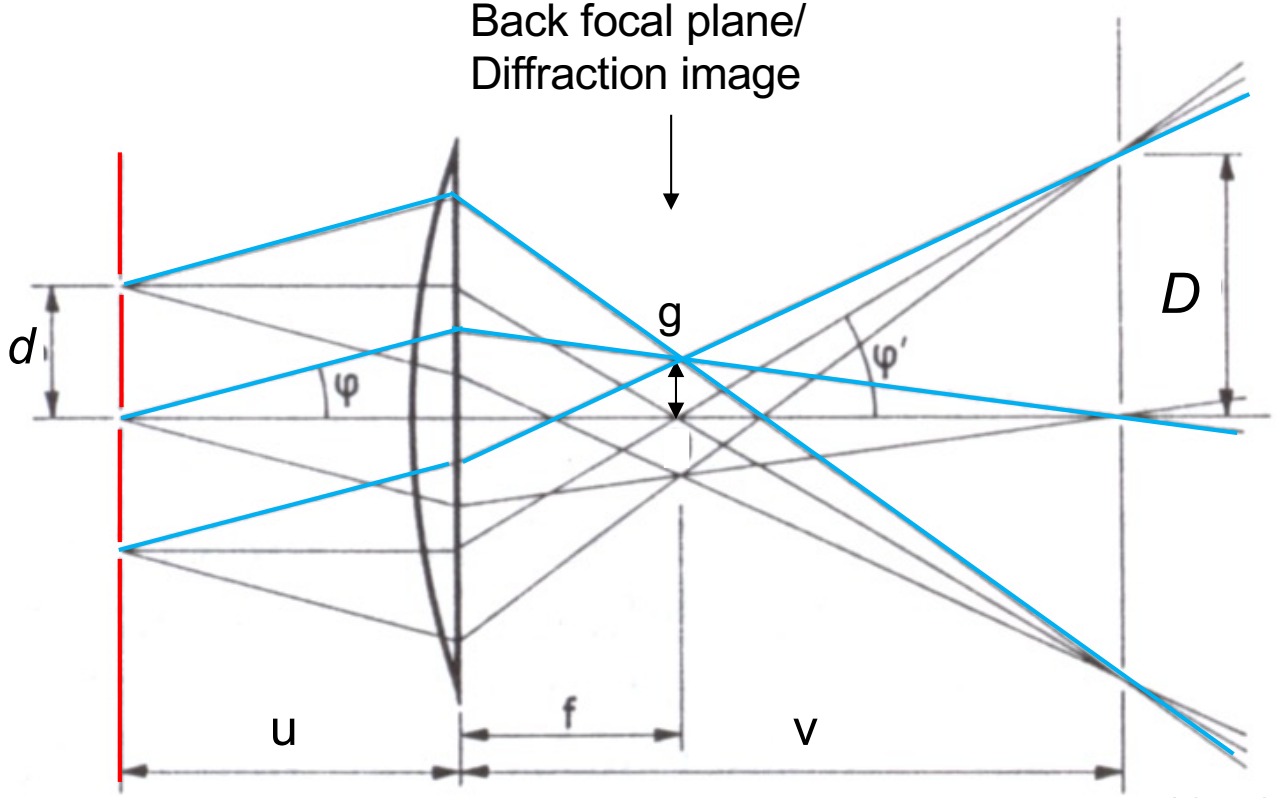
Model object: slit grid imaged through lens

Image plane

Back focal plane/
Diffraction image



g: Distance coordinate in the back focal plane

Adapted from H. Alexander, Phys. Fundamentals of Electron Microscopy, Chap. 1

Constructive interference $\Delta = d \sin \varphi = n\lambda$

With *very large number of slits*: sharply defined directions φ with constructive interference.

$$\sin \varphi = \frac{n\lambda}{d}$$

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$$\sin\varphi = \frac{n\lambda}{d}$$

Typical angles φ in transmission electron microscopy?

g: Distance coordinate in the back focal plane / spatial frequency

$$g = f\tan\varphi \approx f\sin\varphi = n\lambda f\frac{1}{d}$$

For small angles

Spatial frequency of the object

f: focal length of the lens

The spectrum of the spatial frequency (diffraction image) of an object is located in the back focal plane of the imaging lens.

Fraunhofer diffraction

Diffraction of a plane wave by an object with arbitrary transmission function f(x,y) results in amplitude $F(g_x, g_y)$ in the back focal plane of the imaging lens (lens without aberration)

$$F(g_x, g_y) = \int_{\text{Objektfläche}} f(x,y) \exp\left(-2\pi i(g_x x + g_y y)\right) dx dy$$

g_x , g_y : Spatial frequencies in the back focal plane of the imaging lens with unit length $^{-1}$

Factor 2π is usually not assigned to the spatial frequencies in electron microscopy.

Amplitude of the image wave B(x,y): Each point of the wavefront in the back focal plane is a starting point of an elementary wave.

$$B(x,y) = FT^{-1}\{F(g_x, g_y)\} = \int_{\text{Beugungsbild}} F(x,y) \exp\left(2\pi i(g_x x + g_y y)\right) dg_x dg_y$$

Intensity distribution in the image

$$I = B(x,y)B^*(x,y) = |FT^{-1}\{F(g_x,g_y)\}|^2 = FT^{-1}\{F(g_x,g_y)\}FT^{-1}\{F(g_x,g_y)\}^*$$

⟶ Phase information is lost during imaging

Important:

- In the back focal plane of the imaging lens (diffraction image) is the spectrum of the spatial frequency of the object.

Summary VL01

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- The basis of the improvement of the resolution in electron microscopy are wavelengths in the order of picometers (10^{-12} m). The wavelength can be adjusted by the voltage with which the electrons are accelerated.
- Electron lenses "consist" of electric or magnetic fields. They have a variable focal length.
- Basic principles of geometrical optics and Fourier optics from light microscopy can be applied to electron microscopy.
- Geometric optics can be applied to determine image /object position. The Gaussian lens equation applies.
- The Huygens Fresnel principle of wave optics also applies to electron microscopy. Each point of a wave front is the starting point of an elementary wave. Atoms are sources of spherical elementary electron waves.
- Constructive interference occurs when the path length difference of elementary waves emanating from adjacent "scattering" centers (atoms) is an integer multiple of the wavelength.
- The spectrum of the spatial frequency (diffraction image) of the imaged object is located in the back focal plane of the lens. Directions in which constructive interference occurs produce intensity maxima in the diffraction image.
- The amplitude of any object in the diffraction image is calculated by the Fourier transform of the object wave. The image wave results from the inverse Fourier transform of the amplitude in the diffraction image.

5 minute break

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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

2. Practical aspects of transmission electron microscopy (TEM). and scanning transmission electron microscopy (STEM).

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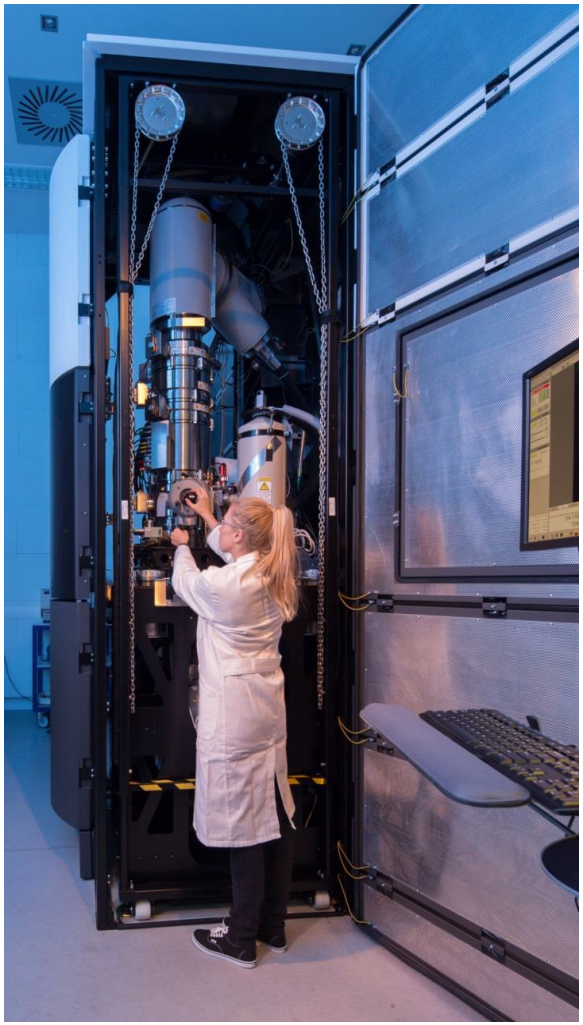
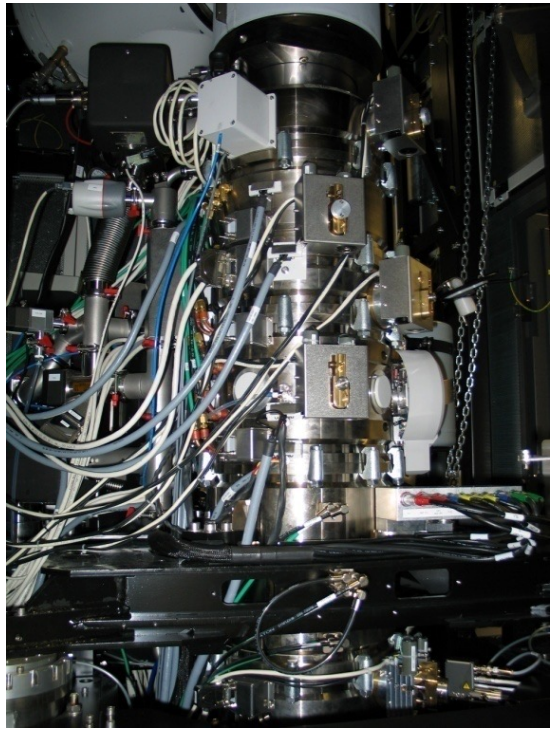
Philips CM200
Point resolution 0.24 nm
@200kV



Aberration corrected
FEI Titan^3 80-300 with point
resolution 0.07 nm @300kV



Upper part of the microscope
column in an
aberration-corrected
FEI Titan^3 80-300

2.1 Design and mode of operation: Transmission electron microscope

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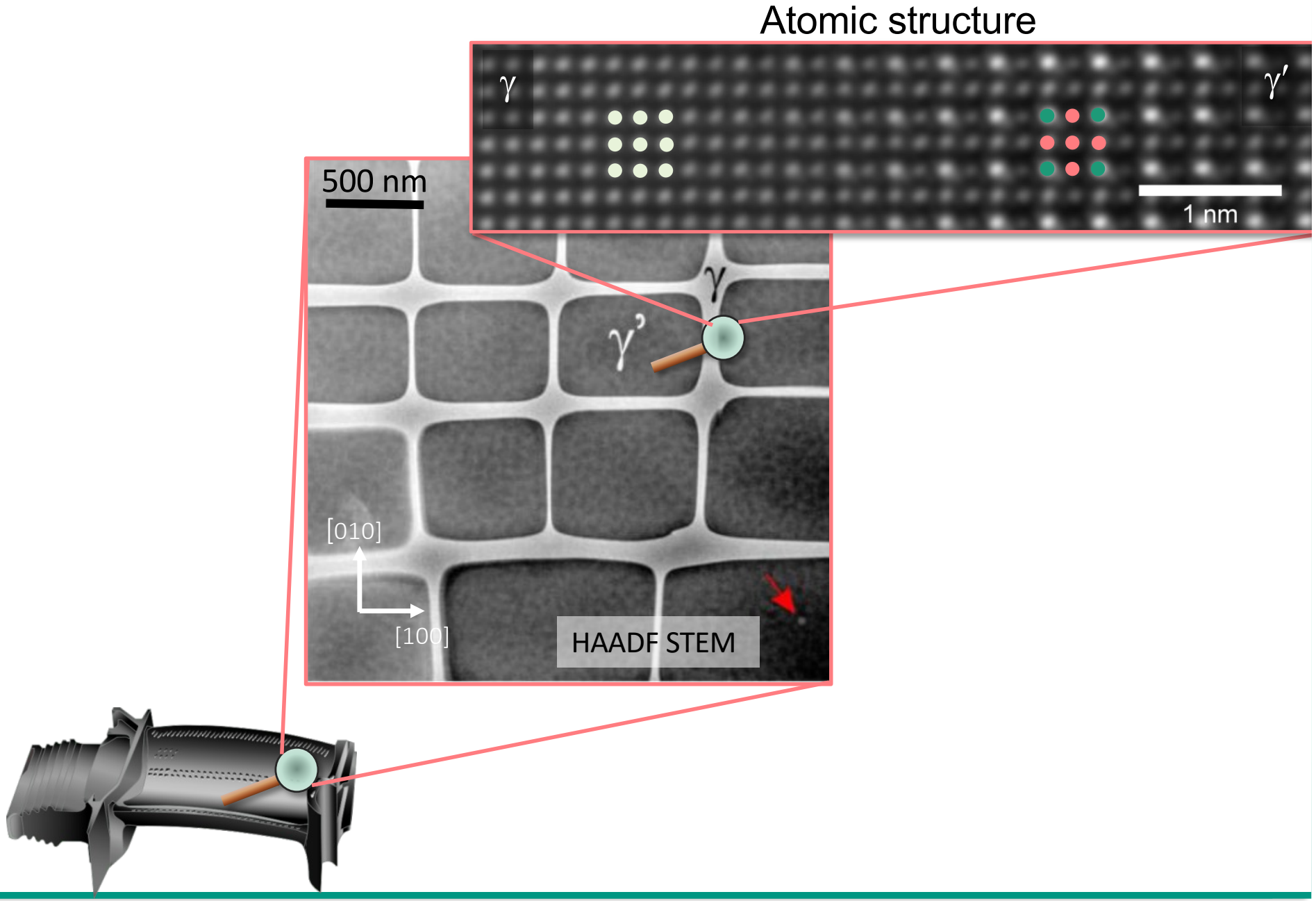
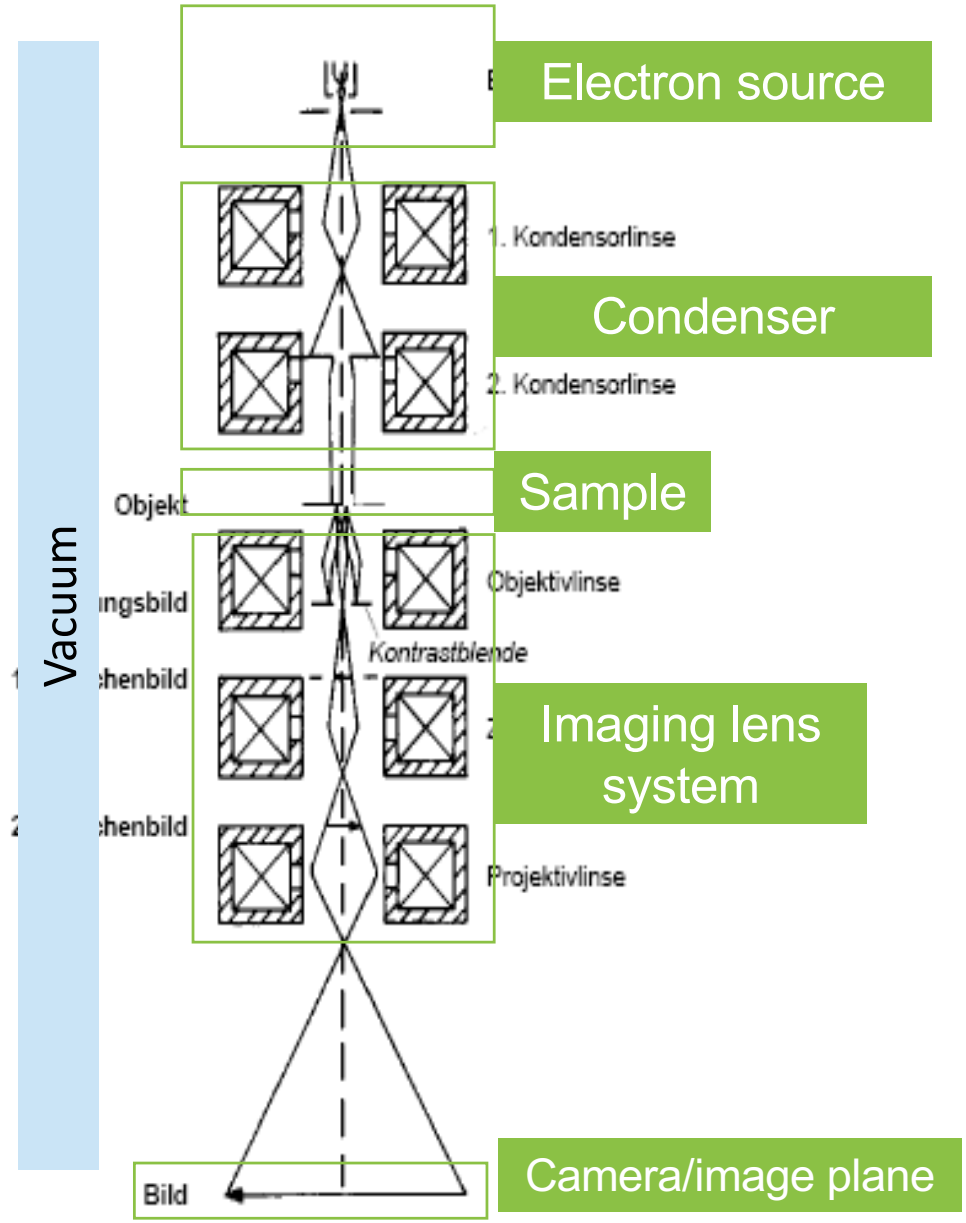


2.1 Design and mode of operation: Transmission electron microscope



L. Reimer, Transmission Electron Microscopy, Fig. 1.3

Comparable structure of light and Transmission electron microscope:
- "Light Source"
- Condenser
- Sample
- imaging lens system
- Image (camera)

Vacuum:
Between 10^{-5} torr (image plane, camera) and 10^{-11} torr in the area of the electron source (field emitter)
10^{-7} - 10^{-8} torr in the sample area

2.1 Design and mode of operation: Transmission electron microscope

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Philips CM 200

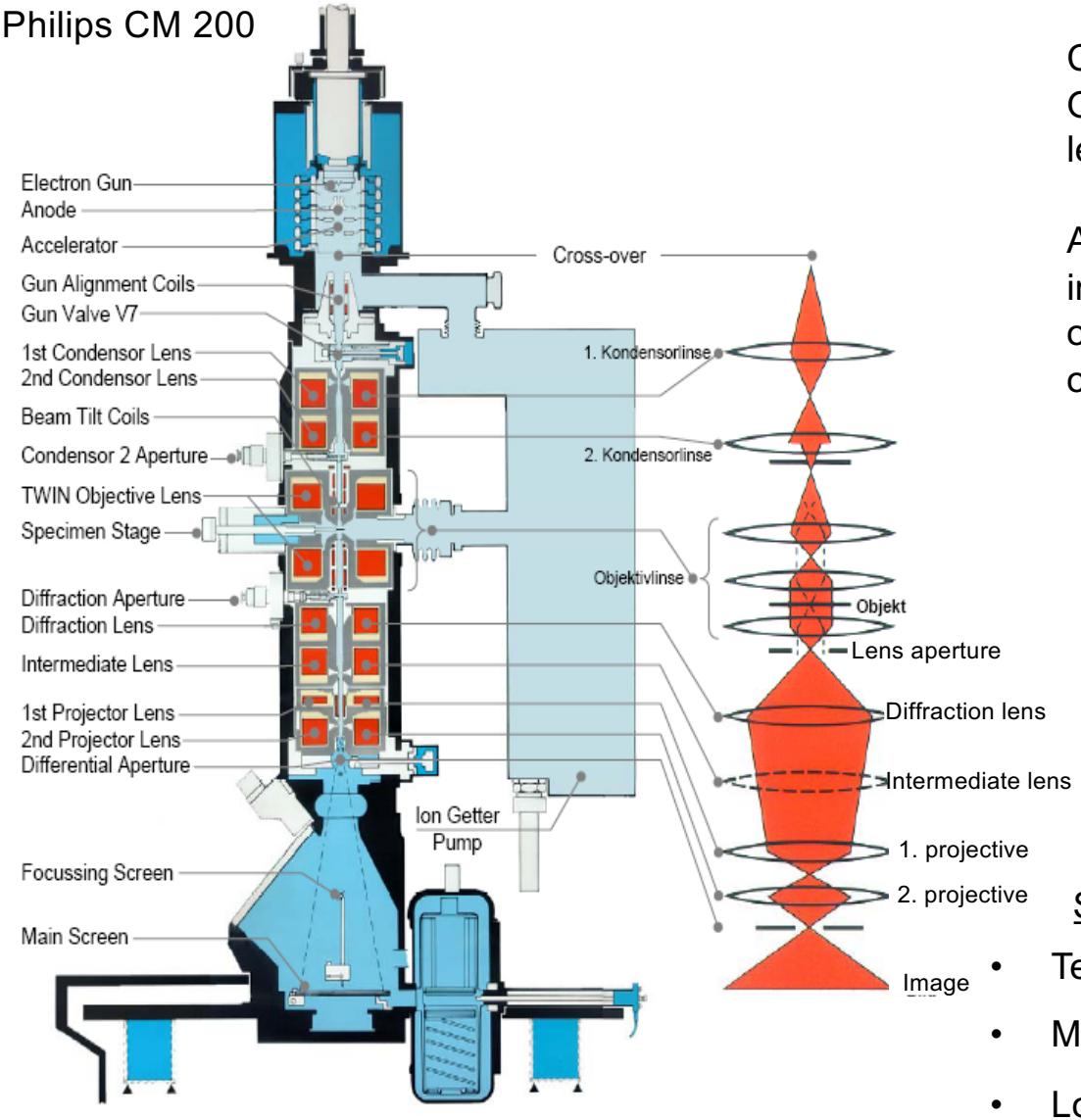

Abbildung 3.2: Schnitt durch ein Elektronenmikroskop der Philips CM-Serie mit Strahlengang für Hellfeldabbildung (©Philips)

Coil pairs in the area of the condenser, Objective lens, intermediate lens and projection lenses for beam tilting and beam shifting

Apertures with different diameters in the condenser, in the back focal plane of the lens (diffraction image) and in the plane of the 1st intermediate image (not shown)

In order to achieve maximum resolution we need sufficient lens and Beam current stabilities.

<u>Strict requirements for the laboratory environment</u>

- Temperature stability ± 0.5 degrees/hour
- Maximum stray magnetic fields: ± 30 nT
- Low building vibration amplitudes
- (1.1 m thick concrete foundation in CFN building)

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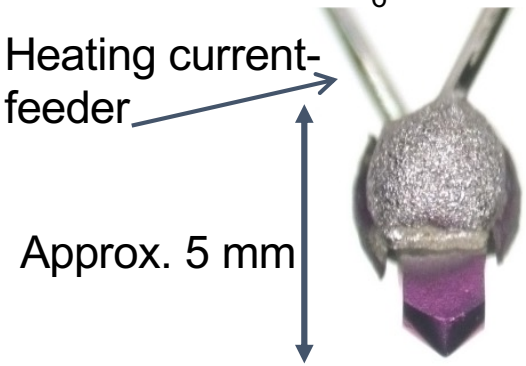
Electron emitter (cathode): Thermal emission and field emission

Electron emission: emission of electrons from a material.

Emission based on *thermal emission* or *combination of thermal and field emission*

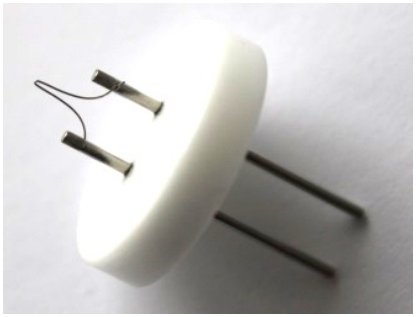
LaB_6 Cathode



Operating temperature: 1700K

Cathodes for thermal emission:
LaB_6 or W "hairpin" cathode

W-"hairpin" cathode

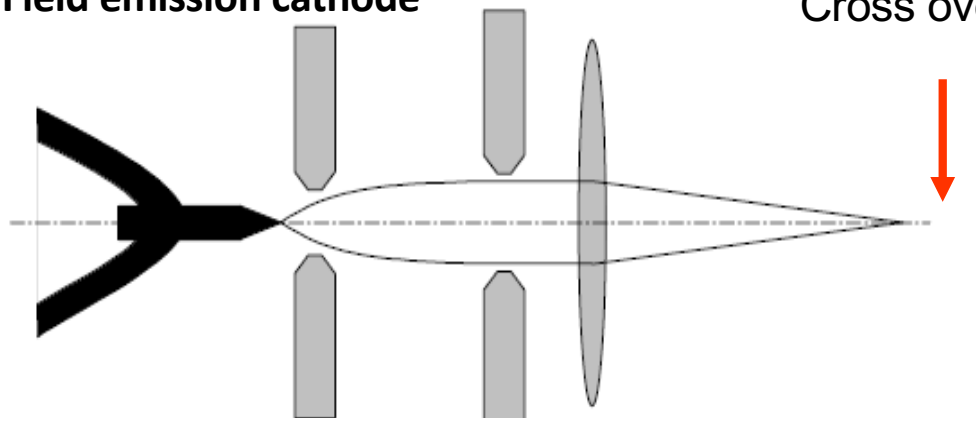


Operating temperature: 2700K

Structure of a field emission cathode
(FEG: field emission gun)
Cathode: tip made of W/ZrO_2 with very small radius of curvature
Typical voltage between Extractor and cathode: 3 - 4 kV

Field emission cathode

Cross over



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Electron emitter (cathode): Thermal emission and field emission

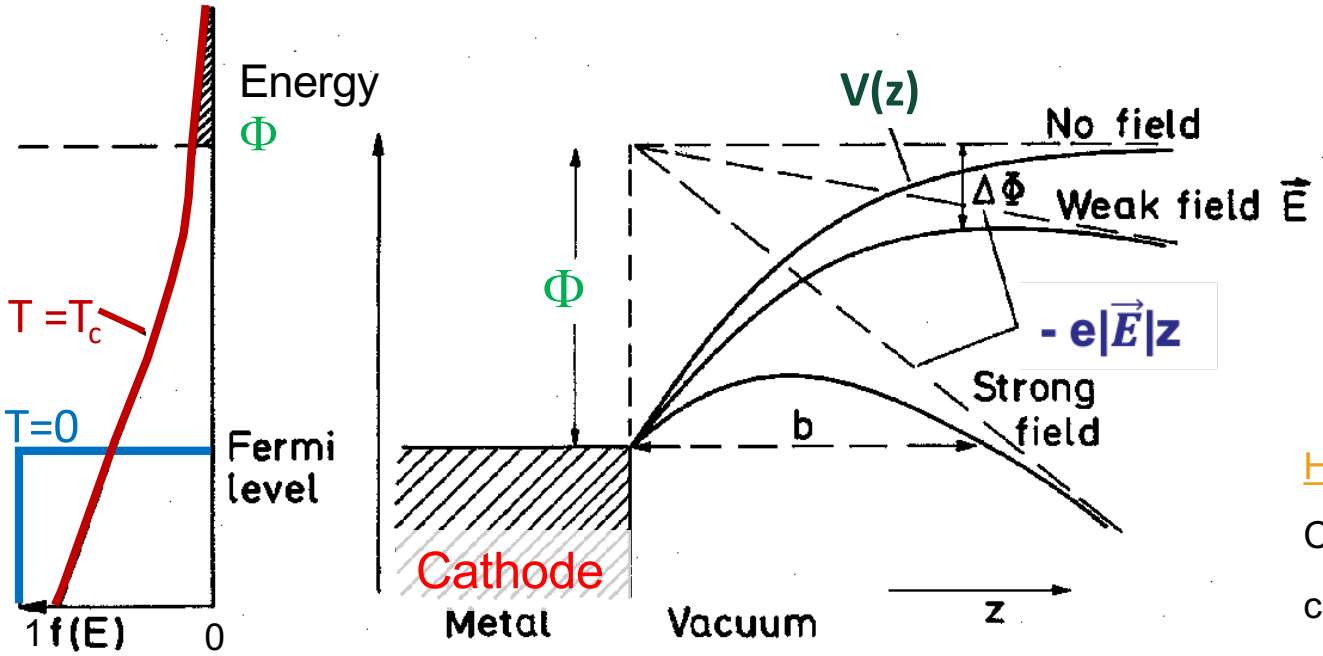

Fig. 4.1. Fermi distribution $f(E)$ and potential energy of electrons at the metal-vacuum boundary L. Reimer, Transmission Electron Microscopy, Fig. 4.1

(1) Φ: Work Function

$_{W}$ =4 ΦeV,

$_{LaB6}$ =2Φ.7 eV

$_{ZrO2}$ =2Φ.5 eV

High local electric field

Cathode tip with small radius of curvature, extractor

(2) Potential energy V(z) of an electron in front of a conducting surface:

$$V(z) = \underbrace{\Phi}_{(1)} - \underbrace{\frac{e^2}{16\pi\varepsilon_0}\frac{1}{z}}_{(2)} - \underbrace{e|\vec{E}|z}_{(3)}$$

(3) When applying an **electric field E**: effectively reduced work function

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Electron emitter

Criteria for the quality of an electron **emitter**

- high current density (high brightness at the object)
- High directional beam value
- Small width of the energy distribution of the electrons (minimization of the color error)
- High spatial coherence of electrons (interference capability in lattice mappings) through a point (small) source as possible

Gun Brightness $\beta = \frac{j}{\Omega}$

Ω: solid angle
j: beam current density

	Brightness value / A (sec sr)$^{-1}$	ΔE / eV	Spatial coherence
W-Hairpin cathode	10^4 - 10^5	3	low
LaB_6 -cathode	10^5 - 10^6	1.5 - 2.5	medium
Schottky FEG (approx. 1700 K)	10^7 - 10^9	0.8 - 1.2	high
FEG ("cold", approx. 1000 K)	10^7 - 10^9	0.3 - 0.8	high

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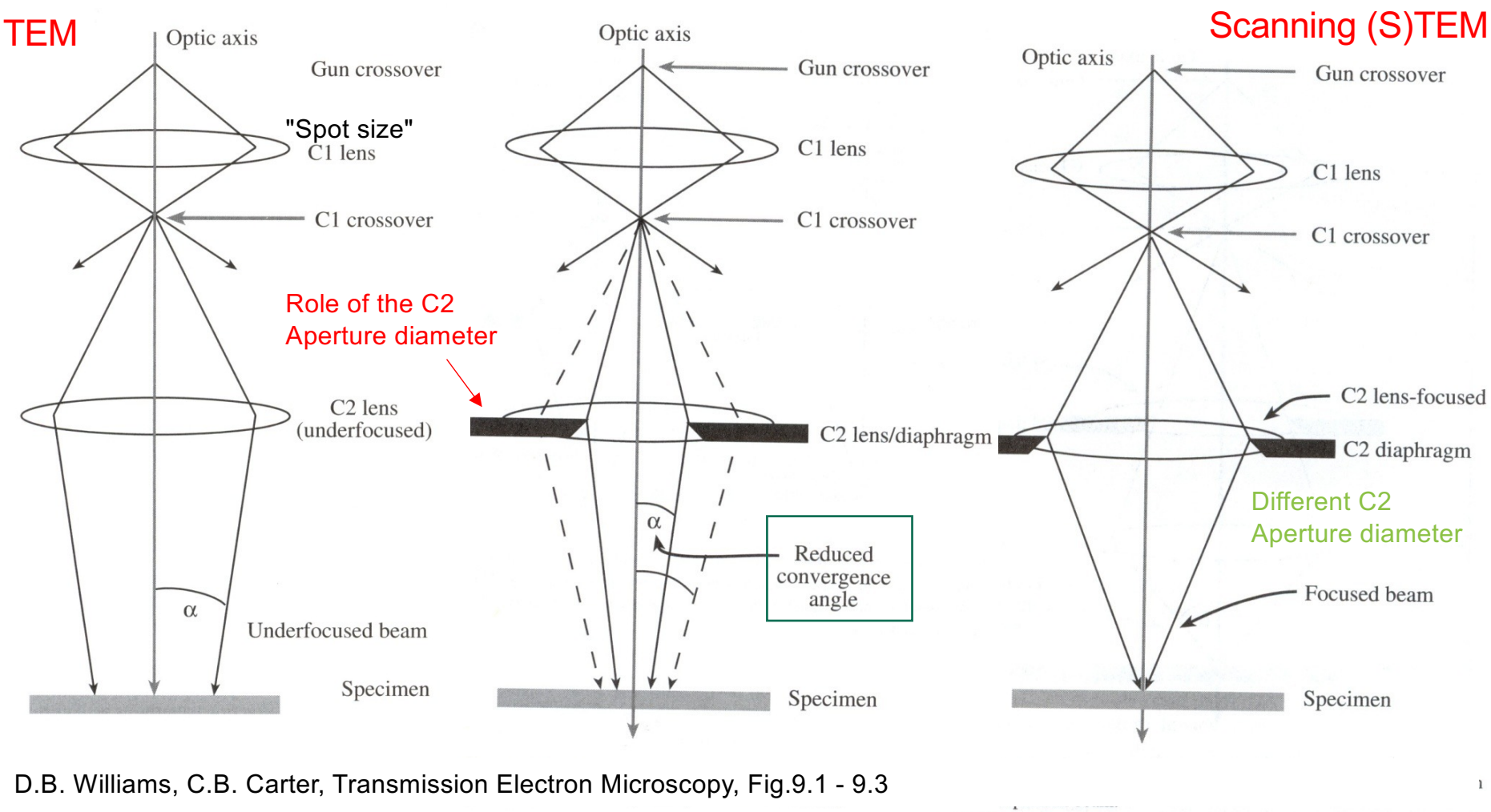
Condenser: adjustment of beam intensity, beam convergence, beam coherence



D.B. Williams, C.B. Carter, Transmission Electron Microscopy, Fig.9.1 - 9.3

Several fixed values for C1 focal length ("spot size"), continuous focusing of C2 by the operator

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TEM: imaging and diffraction mode

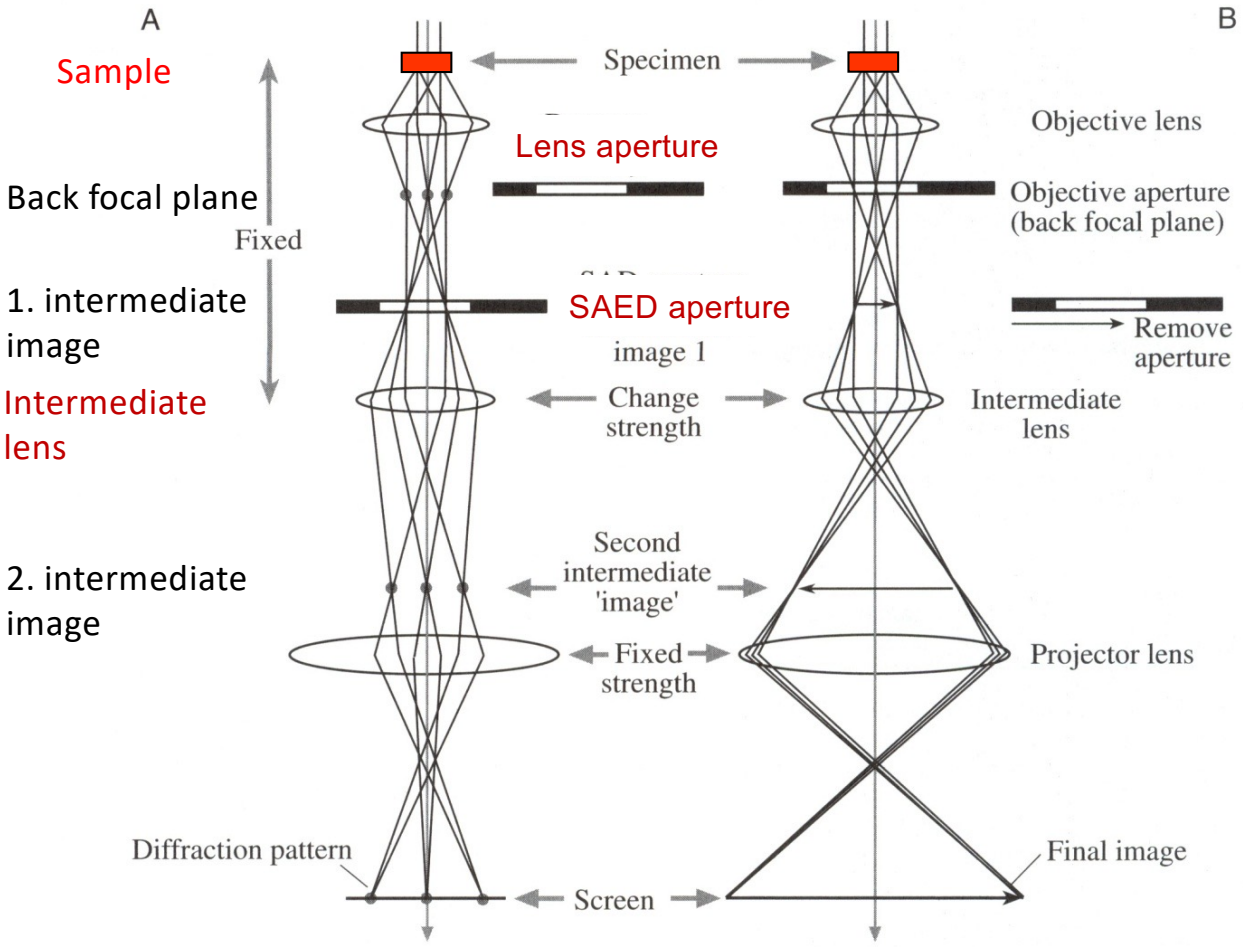

Figure 9.12. The two basic operations of the TEM imaging system involve (A) projecting the diffraction pattern on the viewing screen and (B) projecting the image onto the screen. In each case the intermediate lens selects either the back focal plane or the image plane of the objective lens as its object.

D.B. Williams, C.B. Carter, Transmission Electron Microscopy, Fig.9.12

Switching from image to diffraction mode by excitation change of the intermediate lens

Control of the imaging mode by diameter and position of the **lens (contrast) aperture** in the back focal plane of the lens

Selection of a sample area for diffraction analysis by position and diameter of the **fine area aperture** in the the 1st intermediate image plane (SAED: selected area electron diffraction)

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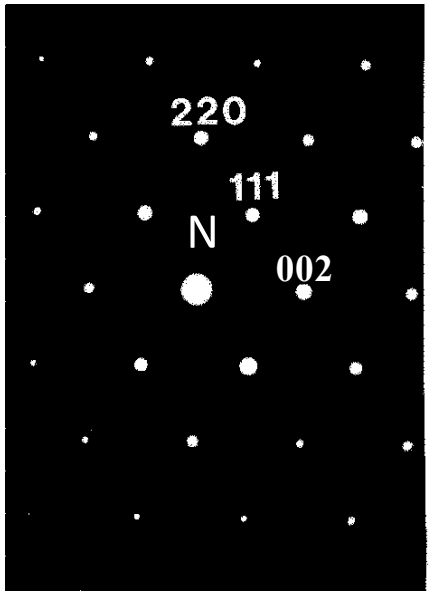
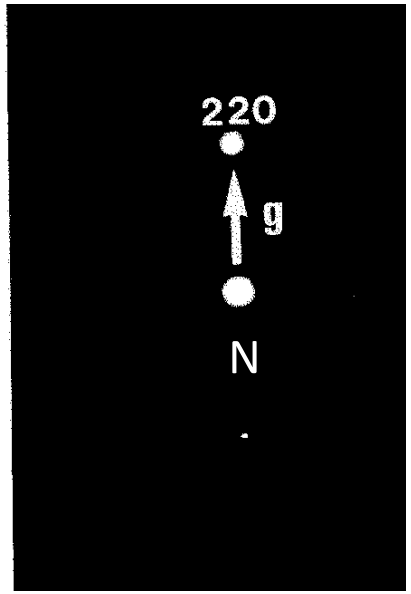
Significance of the diffraction mode



GaAs sample: electron beam oriented parallel to the [$\bar{1}$10] direction



GaAs dual beam condition

- *Local* crystal structure analysis by acquisition of diffraction images of small sample areas by SAED.
- Determination of lattice plane distances
- Orientation of the sample in the microscope by tilting in relation to the electron beam direction
  → Control of the "excitation conditions“

Orientation of the sample / crystal lattice to the electron beam is decisive for the information content of the image!

N: Zero beam (unscattered electrons)

Occurrence of reflexes when Bragg condition is fullfilled

$$2d_{hkl} \sin \theta_B = \nu\lambda$$

d_{hkl} : Lattice plane spacing in the sample
θ_B : Bragg angle
λ: Wavelength

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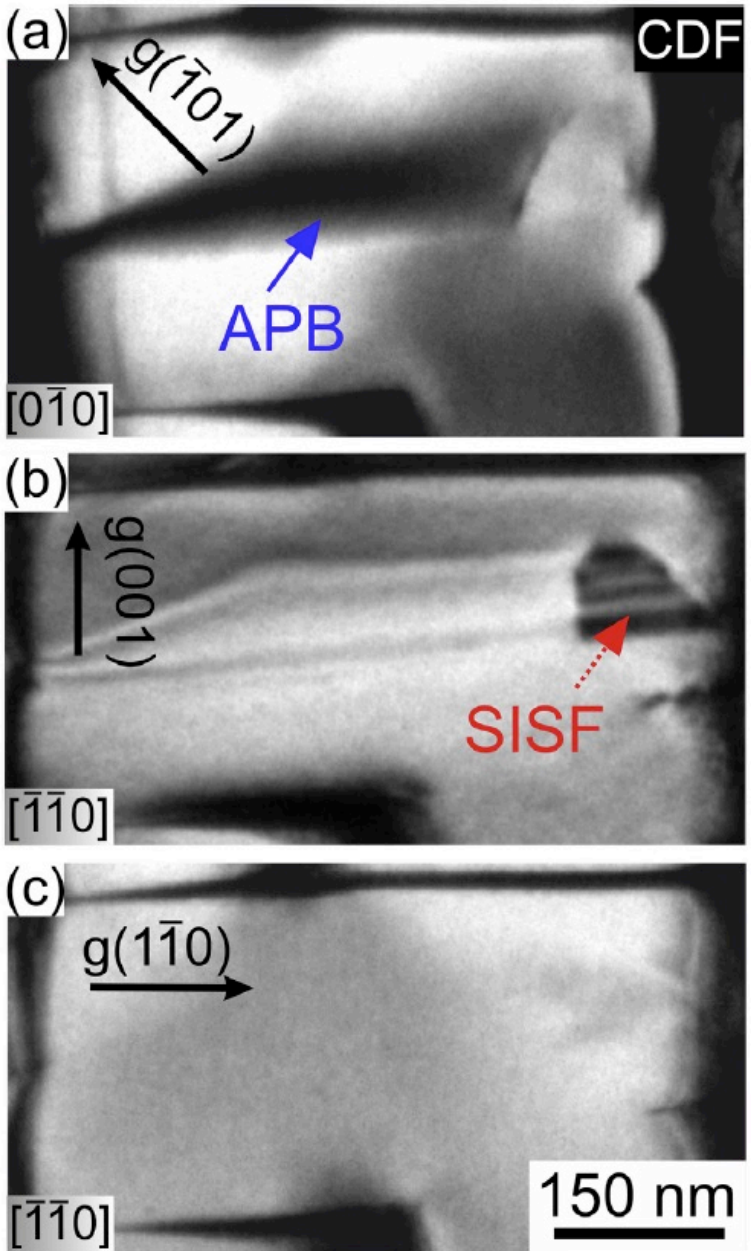
Effect of the orientation of the crystal lattice of the sample relative to the electron beam on the information content of the image.

Crystal defects in the cube-shaped precipitate of a creep-deformed two-phase superalloy.



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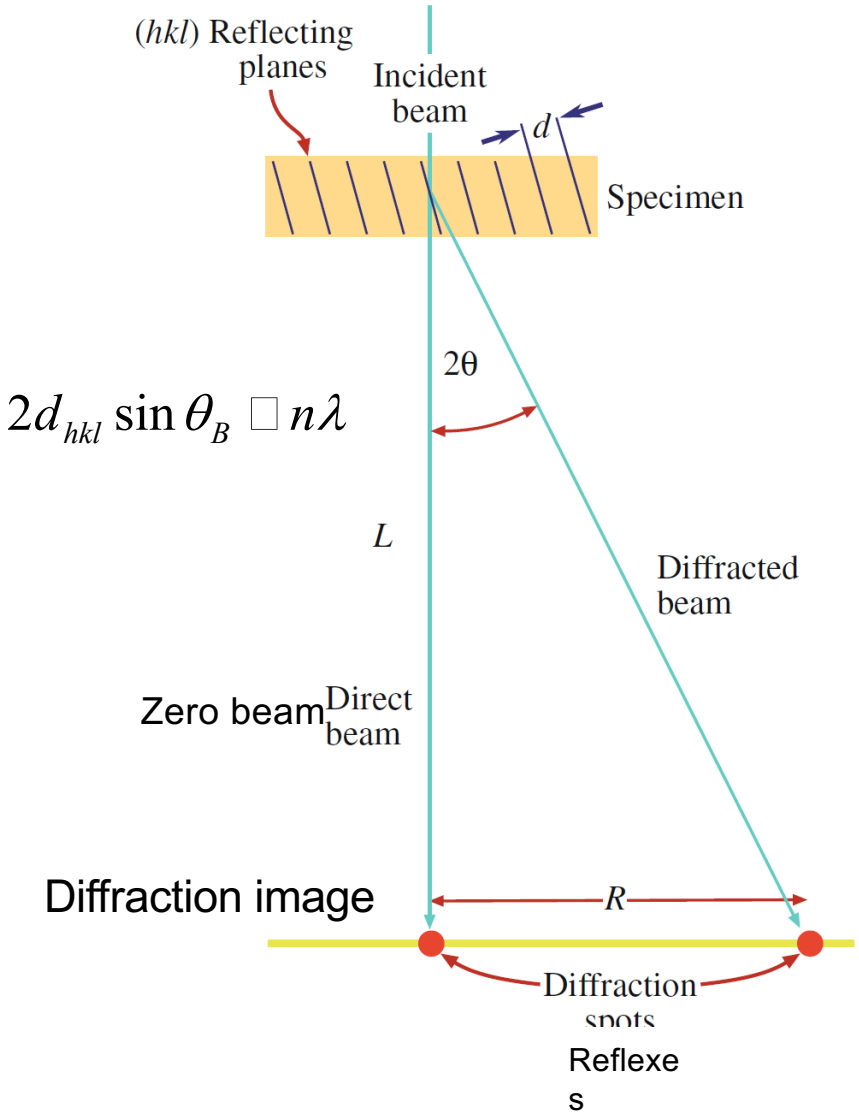
Magnification of the diffraction image: Camera length



$2d_{hkl} \sin\theta_B \square n\lambda$

R: Distance between Bragg reflex and zero beam
L: Camera length (effective focal length)
λL: Camera constant

$$\frac{\lambda}{d_{hkl}} = 2\sin\theta_B \approx \tan 2\theta_B = \frac{R}{L}$$

Determination of lattice plane spacing d_{hkl} from reflection position

$$d_{hkl} = \frac{\lambda L}{R}$$

Check the manufacturer's specifications for L using of a reference sample with known d_{hkl}

D.B. Williams, C.B. Carter, Transmission Electron Microscopy, Fig.9.22/9.23 (new edition).

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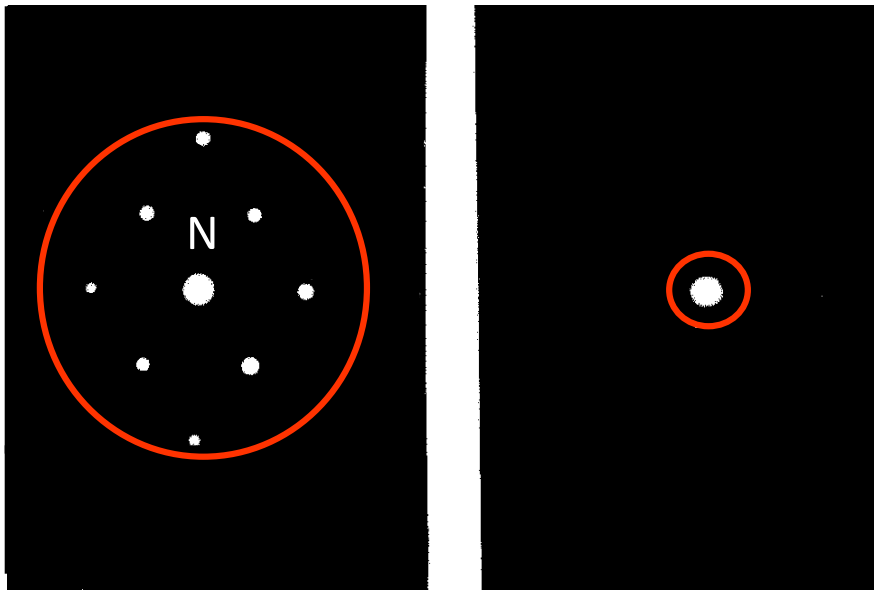
Objective aperture: High resolution or "conventional" TEM imaging



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

Conventional illustration:
Use of only one reflex
(zero beam or a Bragg reflex)
→ Lens aperture with small Diameter

N: Zero beam (unscattered electrons)

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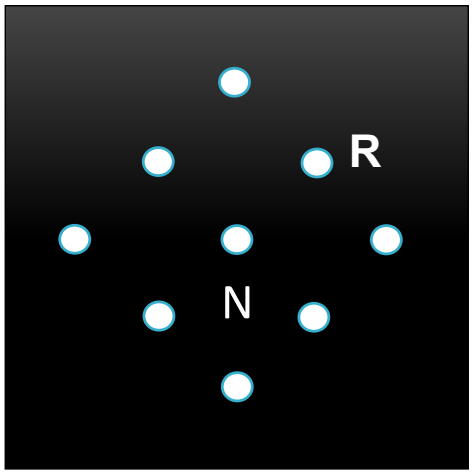
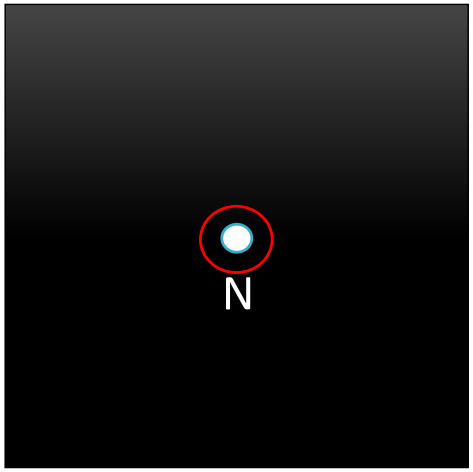
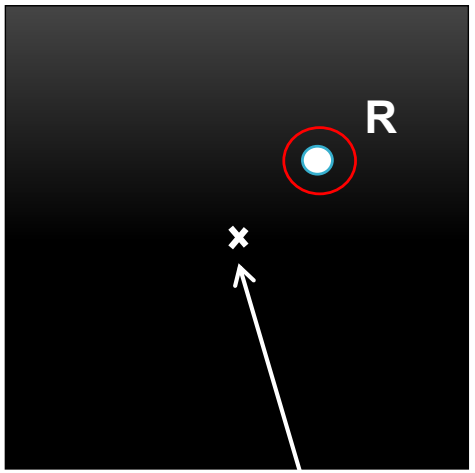
Without lens aperture



Brightfield image

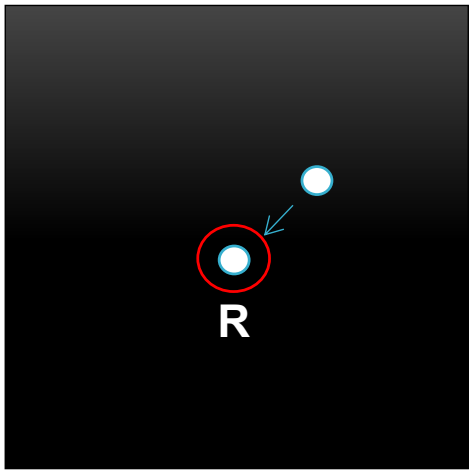


Dark field imaging



N on optical axis

Centered dark field imaging



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

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

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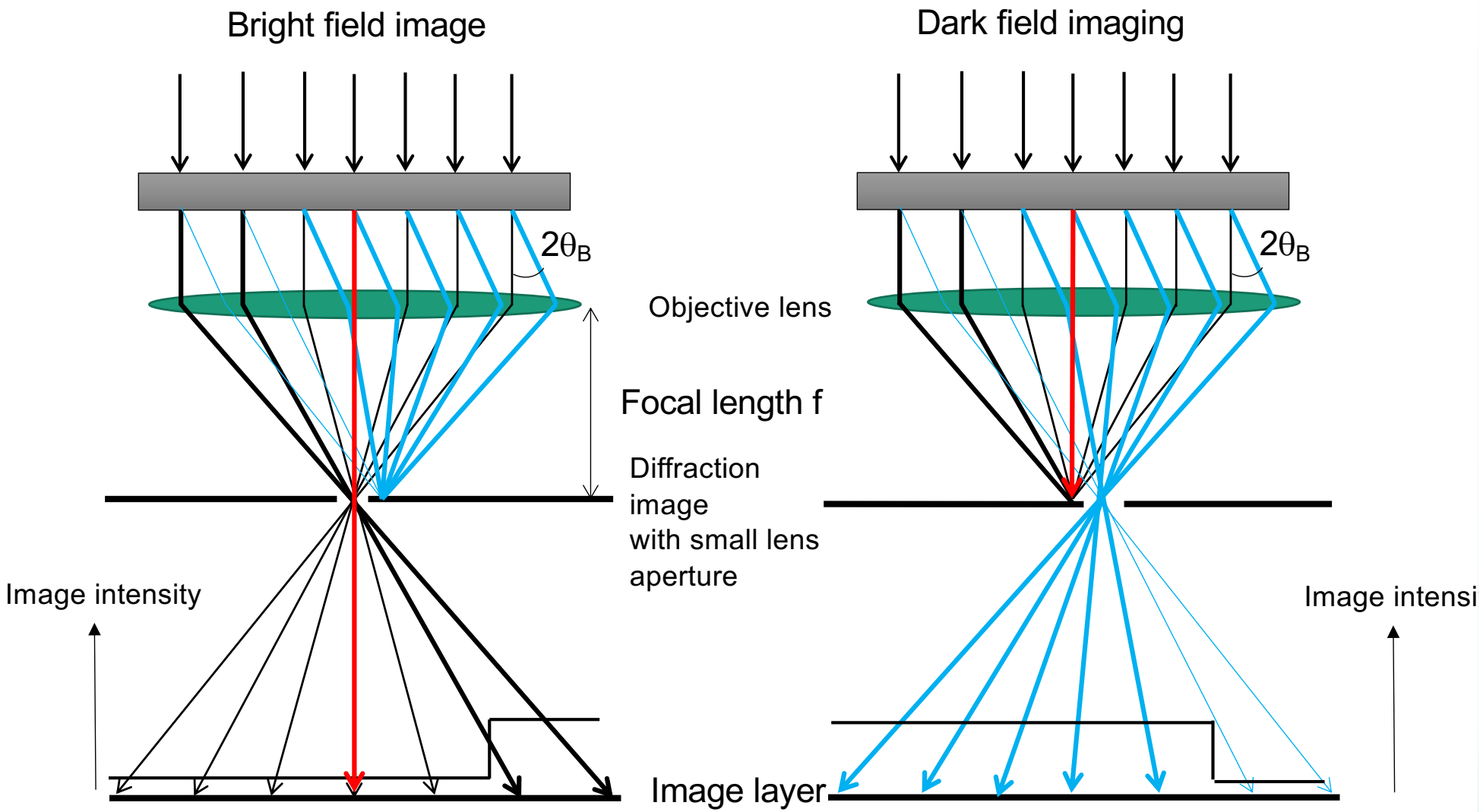
Conventional imaging: Bright field and dark field imaging



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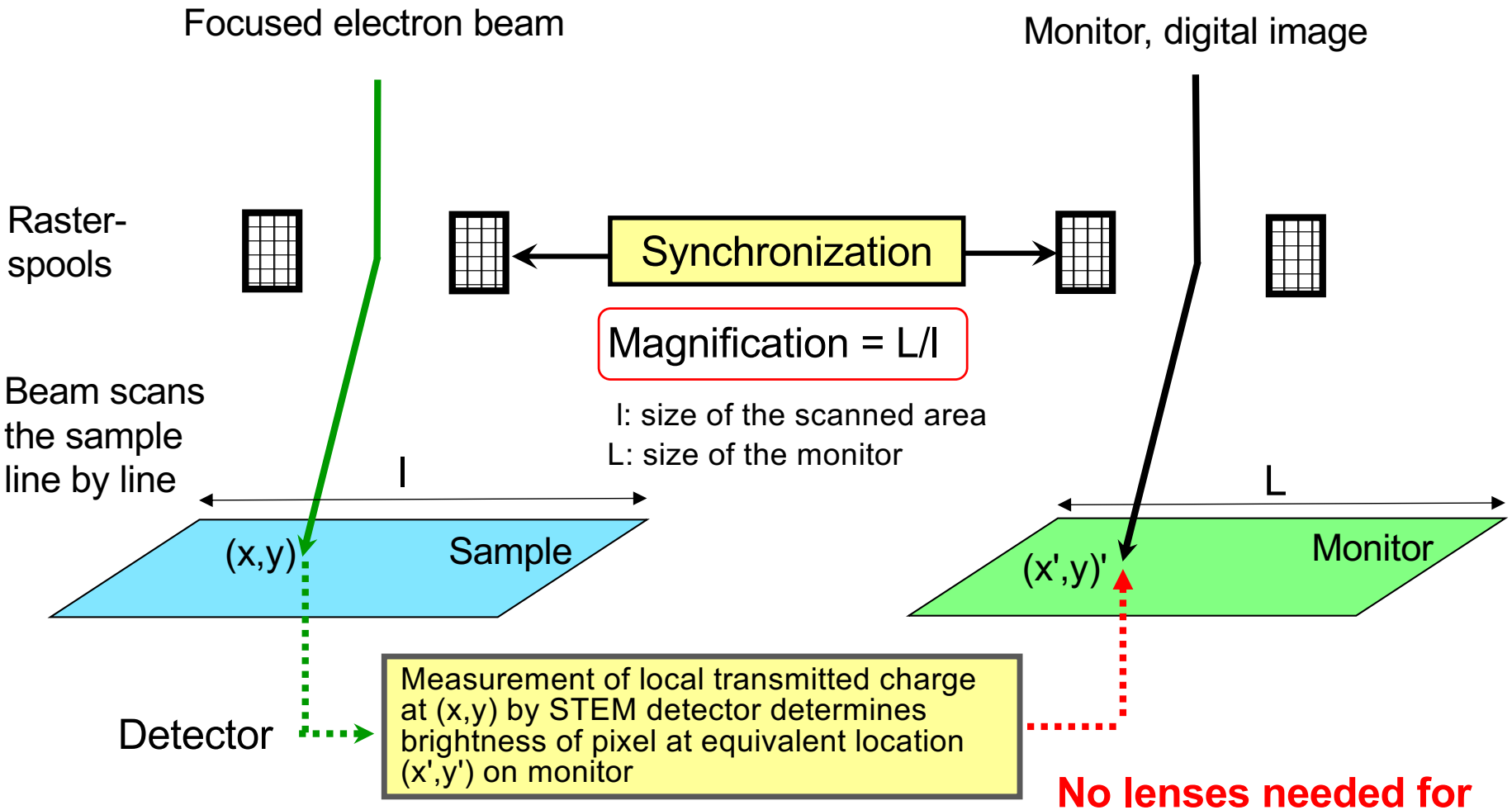
Scanning transmission electron microscopy (STEM) in the transmission electron microscope: principle of image formation.



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

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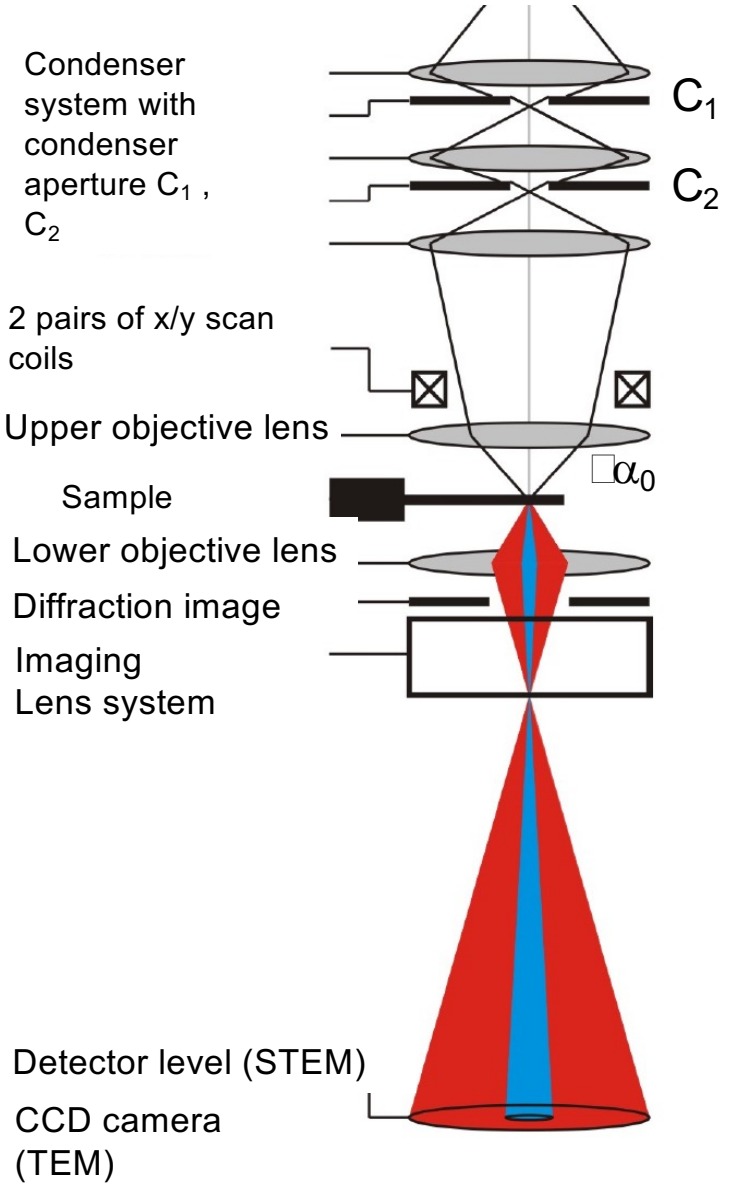
STEM (Scanning Transmission Electron Microscopy)

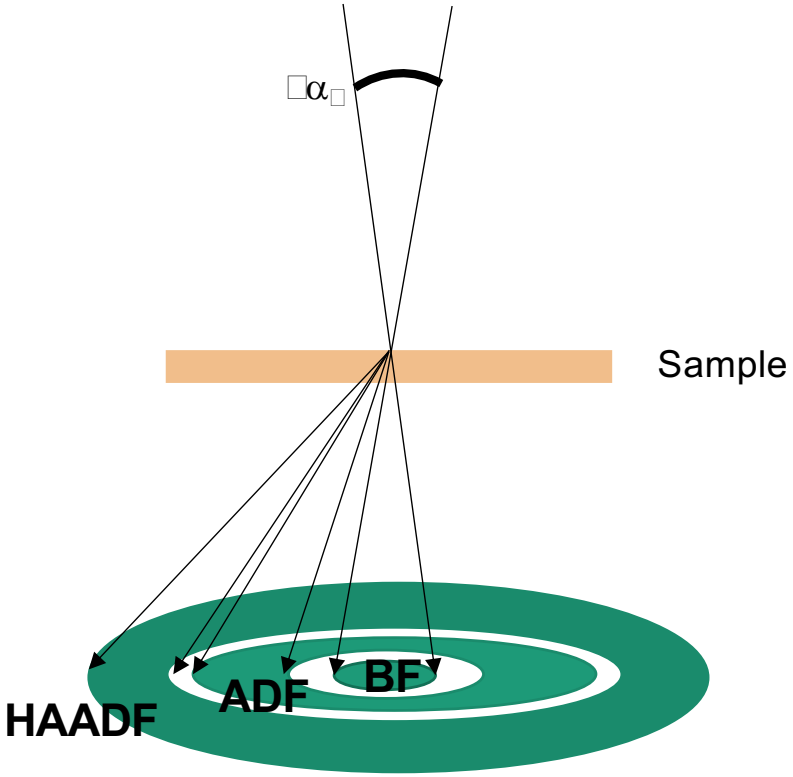
Focusing of the electron beam ("probe") on small diameter.
The beam diameter depends on the aberrations of the lowest focusing lens and the beam convergence angle α_0 . The beam diameter can vary between several nm and ~0.05 nm (in microscopes with "probe" corrector) depending on the desired resolution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

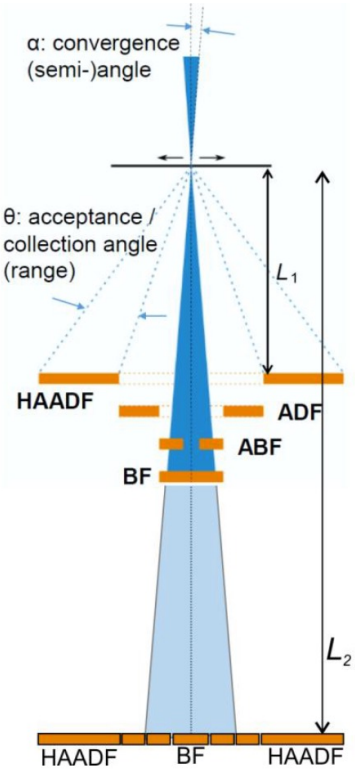
- For small scattering angles, elastic scattering predominates

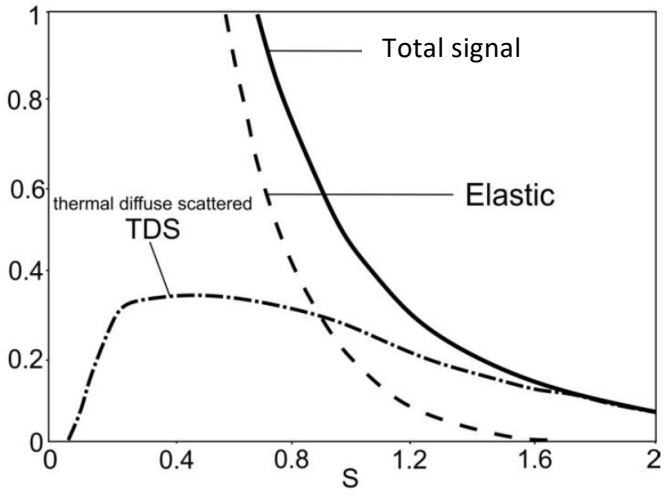
  → Diffraction contrast on the

  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

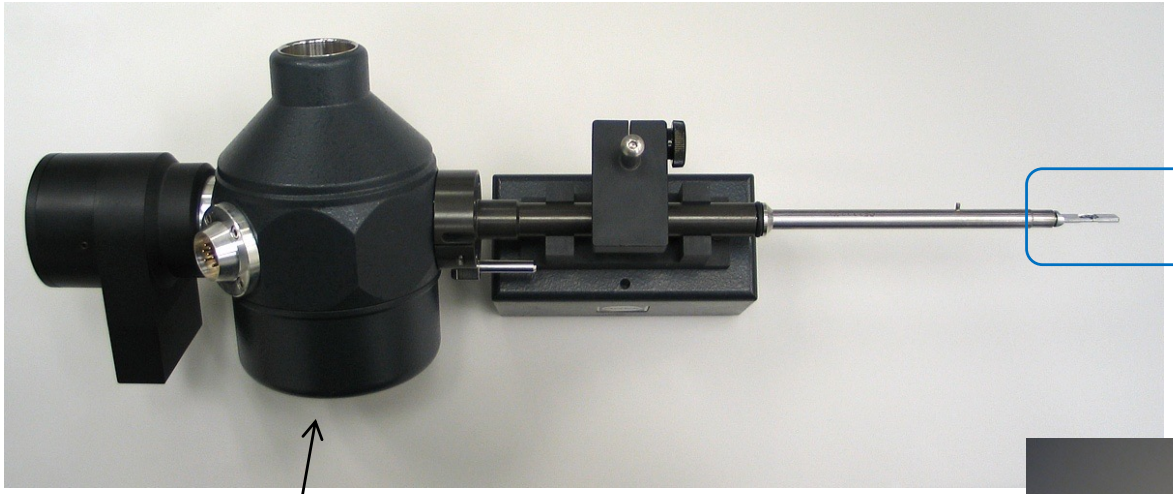
  HAADF detector





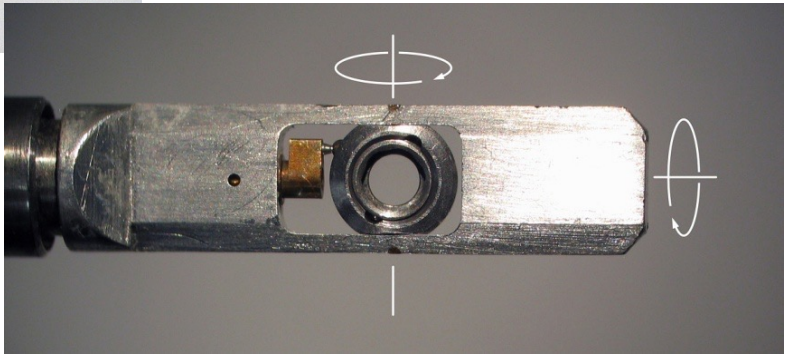
- STEM detector rings physically lie in one plane
- Scattering angle ranges can also be changed by camera length of the imaging lens system

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



Photos: L. Dieterle (LEM)

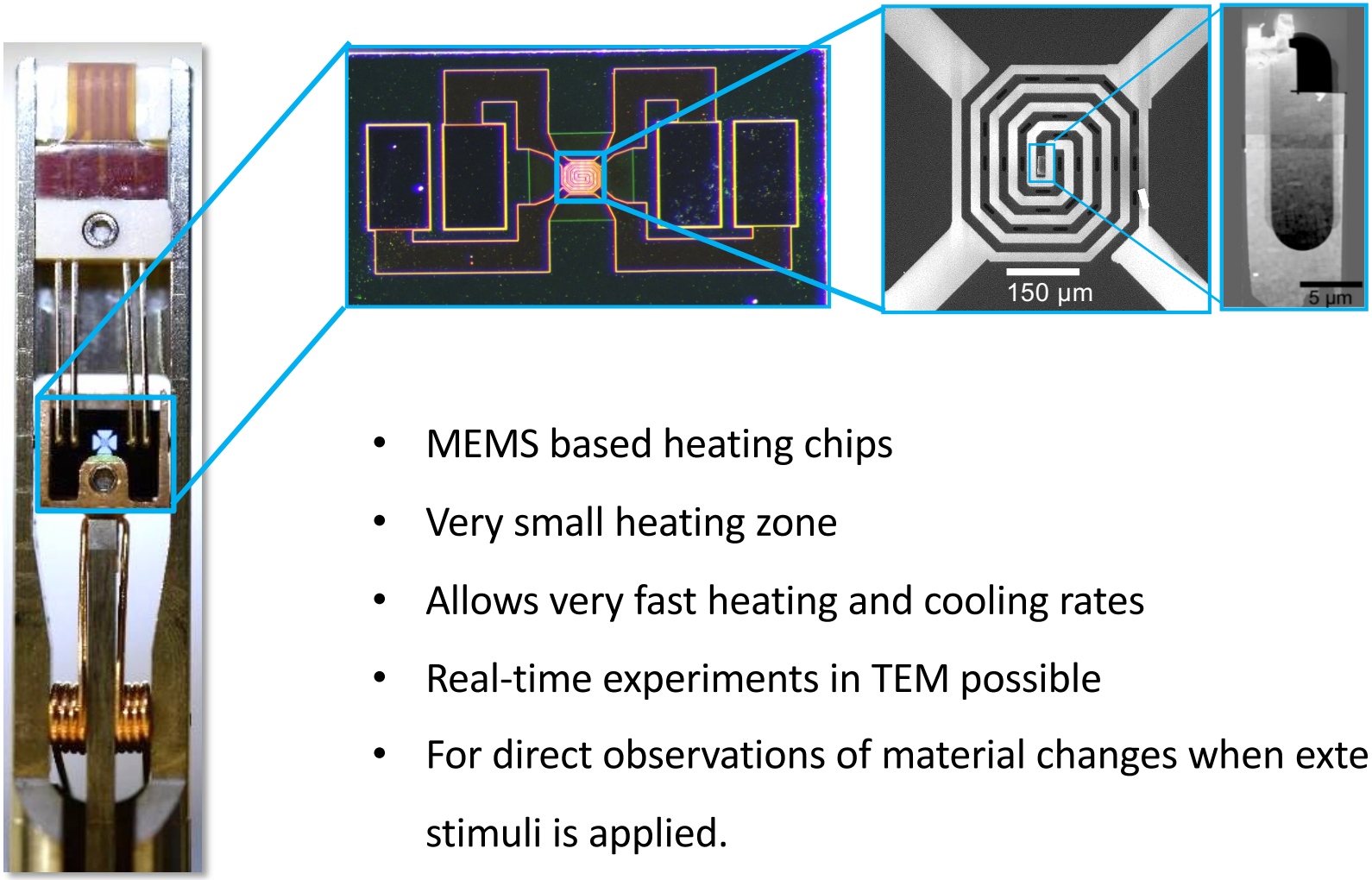
Reservoir for liquid nitrogen -
Cooling holder



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

Laboratory for Electron Microscopy

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.

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Summary after lecture 2

Laboratory for Electron Microscopy

- 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.