

# Electron Microscopy I Lecture 01

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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
- 1.4 Limitation of resolution due to lens aberrations in electron optics

### 2. Practical aspects of transmission electron microscopy (TEM).

- 2.1 Structure and function of a transmission electron microscope
- 2.2 Specimen preparation
- 2.3 Radiation damage

### 3. Electron diffraction in the solid state/kinematic diffraction theory

Electron Microscopy I

- 3.1 Interaction of electrons with individual atoms
- 3.2 Interaction of electrons with crystalline objects: Kinematic diffraction theory



# Content



# 4. Contrast formation and practical examples of the mapping of crystalline objects in solid state and materials research

- 4.1 Mass thickness contrast
- 4.2 Column approach
- 4.3 Contrasts in perfect (single) crystals
- 4.4 Contrasts in crystalline samples with lattice defects, moiré effect
- 4.5 Analysis of phase mixtures
- 4.6 Diffraction patterns (convergent electron diffraction)

### 5. Dynamic electron diffraction

### 6. Imaging of the crystal lattice/high-resolution electron microscopy (HRTEM).

- 6.1 Introduction and practical aspects of high-resolution transmission electron-. microscopy
- 6.2 Dynamic electron diffraction in the HRTEM
- 6.3 Image formation in the HRTEM

### 7. Scanning transmission electron microscopy

# 8. Electron holography

# 9. Transmission electron microscopy with phase plates

# **Electron Microscopy Lecture**







Electron Microscopy I: Transmission Electron Microscopy (2 SWS) (WS 23/24) and accompanying lab course with 4 exercises

Electron Microscopy II: Scanning electron microscopy and analyt. Methods (2 SWS) (SS 2024) and accompanying lab course with 4 exercises possible only in SS2024

**Elective subject in the Master Physics** 

Electron Microscopy I (4 ECTS) with lab course (4 ECTS) Electron Microscopy II (4 ECTS) with lab course (4 ECTS) EM for specialization and supplementary subjects in the field of "Condensed Matter" and "Nanophysics".

Elective for Master MatWerk: EM I and EM II oral exam (16 LP) Applied geosciences, business mathematics, chemistry....

<u>Attendance certificate for lecture</u> Attendance list (Eligible for certificate with a minimum of 2 absences)

Attendance certificates necessary for exam registration



### **Requirements**

### Optics Solid state physics/materials science Most important elements of quantum mechanics: Particle-wave duality



# Lab Course



ILIAS Course: Exercises in Electron Microscopy I WS23/24	
<ul> <li>Registration for the lab course until 07.11.2023.</li> </ul>	
PERSÖNLICHER SCHREIBTISCH - MAGAZIN -	
ACCIVITIZ – ODUITIGETT ZU LIEKCIONETTITIKI OSKOPIE I Mit der Transmissionselektronenmikroskopie (TEM) können Untersuchungen der Struktur im Probeninneren durchgeführt werden. Mit konventionellen Abbild Größenordnung von 1 nm auflösen. In der hochauflösenden TEM wird mit Geräten der neuesten Generation ein Auflösungsvermögen von besser als 0.1 nm elektronentransparenter Proben. Die maximaldurchstrahlbare Probendicke liegt in der Größenordnung von 1000 nm. Die TEM kann in Kombination mit der e Verlustspektroskopie und elektronenspezifischen Abbildungen zur Lösung folgender Fragestellungen eingesetzt werden: - Charakterisierung von Defekten (Ve und Dichte der Defekte - Verteilung und Größe von Ausscheidungen, Partikeln und Hohlräumen bis herab zu Größen von wenigen Nanometern - Analyse unter unterschiedlicher Elemente - Morphologie, Dicke und Grenzflächeneigenschaften von dünnen Schichten	
Inhalt         Info         Einstellungen         Mitglieder         Lernfortschritt         Metadaten         Export         Rechte         Voransicht als Mitglied aktivieren           Zeigen         Verwalten         Sortierung         Seite gestalten         Seite gestalten	
Neues Objekt hinzufügen -	
INHALT	
Anmeldung Übungen EM 1 Bitte melden Sie sich zu den Übungen bis zum 4.11.2021 über diesen Link an.	nmeldung.php
Versuch 1	4 exercises,
Versuch 2	Documents will be
Versuch 3	provided 2 weeks prior to the exercise
Versuch 4	

# Lab Course



### **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  $\rightarrow$  Electron Microscopy I  $\rightarrow$  Lab Course Registration

### Anmeldung Übungen EM 1

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

 $\leftarrow$  LINK to registration form

http://www.lem.kit.edu/praktikumsanmeldung.php

# Literature



# David B. Williams • C. Barry Carter **Transmission Electron** *Discrete Construction Discrete Construction Discrete*

Second Edition

D.B. Williams, C.B. Carter, Transmission Electron Microscopy, Plenum Press 2009.



L. Reimer, H. Kohl, Transmission Electron Microscopy, Springer Series in Optical Sciences vol. 36, Springer Verlag 2008, 5th edition



Edington, Jeffrey William

Note: This is not the actual book cover

J.W. Edington, Practical electron microscopy in material science, 1991

ISBN 13: 9780442222307

# Literature



### Optic

E. Hecht, A. Zajac, Optics, Addison-Wesley Publishing Company

### Transmissionelectronmicroscopy and electron diffraction

H. Alexander, Physikalische Grundlagen der Elektronenmikroskopie Teubner 1997

### D.B. Williams, C.B. Carter, Transmission Electron Microscopy, Plenum Press 2009

P.J. Goodhew, F.J. Humphreys, P. Beanland, Electron Microscopy and Analysis, 3rd edition, Taylor and Francis ca. 40 EU (vergriffen)

L. Reimer, H. Kohl, Transmission Electron Microscopy, Springer Series in Optical Sciences vol. 36, Springer Verlag 2008, 5th edition

B. Fultz, J.M. Howe, Transmission Electron Microscopy and Diffractometry of Materials (Advanced Texts in Physics) Springer Verlag 3rd edition

P. Hirsch, A. Howie, R.B. Nicholson, D.W. Pashley, J. Wheelan, Electron Microscopy of Thin Crystals, Robert E. Krieger Publishing Company, 1977 (

M. De Graef, Introduction to Conventional Transmission Electron Microscopy, Cambridge University Press

John C. H. Spence, Experimental High-Resolution Electron Microscopy, Oxford University Press, 4<sup>th</sup> edition, 2013

E. Fuchs, H. Oppolzer, H. Rehme, Particle Beam Microanalysis, VCH Verlag 1990

# Literature



R. Erni, Aberration-Corrected Imaging in Transmission Electron Microscopy: An Introduction, Imperial College Press 2010

X. Zou, S. Hovmoller, P. Oleynikov, Electron Crystallography: Electron Microscopy and Electron Diffraction, Oxford University Press 2011

E.J. Kirkland, Advanced Computing in Electron Microscopy, Springer 2nd edition

### Applications in solid state physics/materials research/materials science.

E. Hornbogen, B. Skrotzki, Werkstoffmikroskopie, Serie Werkstoff-Forschung und -Technik, Band 11, Herausgeber B. Ilschner, 2. Auflage Springer Verlag 1993

G. Thomas, M. J. Goringe, Transmission Electron Microscopy of Materials, John Wiley & Sons Inc 1987

### **Additional Textbooks:**

M.H. Loretto, Electron Beam Analysis of Materials, Chapman and Hall

J.H.C. Spence, J.M. Zuo, Electron Microdiffraction, Plenum Press

A. Tonomura, Electron Holography, Springer Series in Optical Sciences 70, 1993



- Examination of objects with a *resolution of 0.05 nm in* comparison to conventional light microscopy (few 100 nm) due to low electron wavelength
- Transillumination of thin specimens View inside the specimen
- Combination of mapping (structure in real space), diffraction (reciprocal space), chemical analysis and investigation of electronic properties on sub-nm (sub-Å) scale
- In particular, study of defects, interfaces, non-periodic structures.
- in-situ observation of plastic deformation, chemical reactions, ...
- Tomography (three-dimensional object reconstruction)
- Electron holography (reconstruction of amplitude and phase), imaging of electric and magnetic fields, ...

### **Application in:**

Solid state physics, materials research Chemistry, catalysis research Nanotechnology Geology/Mineralogy Biology/Zoology/Medicine Archaeology Art Nobel Prize for Ernst Ruska in 1986 for design of first electron microscope

Nobel Prize for Dubochet, Frank and Henderson in 2017 for Cryo-EM



Platinum clusters and single platinum atoms on a thin carbon film







Figure 8. Experimental images of two different edge dislocations in SrTiO<sub>3</sub> [110] with a spherical aberration of  $-40~\mu$ m and a defocus of around 8 nm.

C.-L. Jia et al, Microsc. Microanal. 10, 174 (2004)



Figure 1. An atomic-resolution ADF-STEM image of a  $\langle 110 \rangle$  projection of <u>Si doped with Sb</u> by low-temperature MBE showing contrast from single atoms. The right-hand side is the Si substrate, in which there is no Sb and all the atomic columns are the same intensity (atomic columns are white in this imaging mode). The left-hand side is the doped material. Most of the brightest atomic columns on the left contain a single Sb atom. A few (~15%) contain two Sb atoms. The image was acquired at room temperature in a 200-kV JEOL 2010F-ARP STEM with 10-mrad incident convergence angle and 50-mrad detector inner angle. We estimate this region of the sample is ~50 Å thick. The image was smoothed and background corrected.

P.M. Voyles, Microsc. Microanal. 10, 291 (2004)



# Wide range of applications at medium magnifications

Electron holography





Fig. 4. Amplitude and phase image of  $0.3 \,\mu\text{m}$  NMOS (left) and PMOS (right) transistors. The source and drain areas are revealed in the phase images. Sharp black-white contrast lines are due to phase changes larger than  $2\pi$ 

Dislocation network/small angle grain boundary in aluminum R. Wittmann et al. (LEM) Mat. Sci. Engn. A 266, 183 (1999)

Rau et al, phys. Stat. sol. (b) 222, 213 (2000)





Philips CM200 point resolution 0.24 nm @200kV



FEI Titan<sup>3</sup> 80-300 point resolution 0.07 nm @ 300kV



Upper part of the microscope column

# 1. From light microscopy to electron microscopy



# 1.1 Light and matter waves

Basis of resolution improvement: wave-particle duality (de Broglie, 1924)

$$\substack{\lambda: \text{ Wavelength} \\ \text{p: Impulse}} =$$

h: Planck's constant 1.05-10<sup>-34</sup> Nms (6.5-10<sup>-16</sup> eVs)

Impulse of an electron accelerated in vacuum with voltage U:

$$eU = \frac{p^2}{2m} \longrightarrow p = \sqrt{2eUm} \longrightarrow \lambda = \frac{h}{\sqrt{2eUm}}$$

h

p

For U = 100000 V (100 kV):  $v \cong 0.5 c$  — relativistic calculation p = mv

$$\lambda = \frac{h}{\sqrt{2m_0 eU\left(1+\frac{eU}{2m_0c^2}\right)}}$$
 c: Speed of light



Wavelength in pm (10 <sup>-12</sup> m)	Electron energy in keV
3.5	100
3.348	120
2.507	200
1.968	300
1.643	400
0.8715	1000

Discrepancy between best possible (50 pm) and diffractionlimited resolution due to lens aberration!



Light optics: glass/plastic lenses Electron optics: electrostatic or **magnetic** lenses (refraction by Lorentz force)



Variable focal length through change of the coil current and the magnetic field strength







Pictures: Levin Dieterle (LEM)







### Construction of an image through ray diagrams comparable to light optics **Rays: Trajectories of electrons**



Figure 6.1. Image formation by a convex lens. A point object is imaged as a point and the collection semiangle of the lens is defined relative to the object ( $\beta$ ) or the image ( $\alpha$ ).

D.B. Williams, C.B. Carter, Transmission Electron Microscopy, Fig.6.1

or CCD (charge-coupled device) camera Real image, convex lens necessary

Optical axis through center of lens

Object side opening semi-angle  $\beta$ Image side convergence semi-angle  $\alpha$ 

 $\alpha$ ,  $\beta$  in the TEM very small (few degrees)





**Figure 6.3.** A complete ray diagram for a finite object, symmetrically positioned around the optic axis. All rays emerging from a point in the object (distance u from the lens) that are gathered by the lens converge to a point in the image (distance v from the lens) and all parallel rays are focused in the focal plane (distance f from the lens).

D.B. Williams, C.B. Carter, Transmission Electron Microscopy, Fig.6.3

I: Image size O: Object size M: Magnification u: Object width v: Image width f: Focal length



### Construction of an image by imaging with a thin lens



**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 v increases, but the object distance is unchanged. D.B. Williams, C.B. Carter, Transmission Electron Microscopy, Fig.6.4

In the TEM

- For a <u>fixed</u> object width *u*, a stronger lens has shorter focal length *f2* and produces a lower magnification than a weaker lense with a longer focal length *f1*.
- 4 to 6-stage lens systems, in order to reach magnifications of up to 1 million
- Total magnification: product of the magnifications of every single lense



General description of a wave by location and time dependent amplitude with wavenumber vector  $\vec{k}$ , wavelength  $\lambda$  and frequency  $\omega$   $\vec{E}(\vec{r},t) = \vec{E_0} \exp\left(i(\vec{k}\vec{r}-\omega t)\right)$  Electric wave  $|\vec{k}| = \frac{1}{\lambda}$  $\psi(\vec{r},t) = \psi_0 \exp\left(i(\vec{k}\vec{r}-\omega t)\right)$  Electron wave function

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

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

