

Machine Learning in STEM 2023 Lecture 02

Lecture 2: Introduction to TEM

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- Literature
- Software– Analysis
- Software–Acquisition

Introduction

- Electron Meets Matter
- Diffraction and Imaging
- Basics
- EELS

Electron Optics

- Overview of TEM
- Ray Diagrams

Diffraction-Basics

- Basics

- ▶ Williams and Carter
Transmission Electron Microscopy
Plenum Press, 2009 (2nd Edition).
available online at the Library
- ▶ Reimer and Kohl
Transmission Electron Microscopy
Springer, Berlin, 2007 (5th Edition).
available online at the Library
- ▶ Zuo and Spence
Advanced Transmission Electron Microscopy: Imaging and Diffraction
in Nanoscience; Springer, New York, 2017
available online at the Library
- ▶ Kirkland
Advanced Computing in Electron Microscopy
Springer, New York, 2010 (2nd Edition).
available online at the Library



- ▶ R.F. Egerton.
Electron Energy–Loss Spectroscopy in the Electron Microscope.
Plenum Press, 2011 (3rd Edition).
available online at the Library
- ▶ R. Brydson.
Electron Energy–Loss Spectroscopy,
BIOS Scientific Publisher Ltd., 2001



Software–Image Analysis

There are three programs commonly used to analyze TEM data offline:

- ▶ Digital Micrograph (DM) is the most used image acquisition and analysis software at TEMs. The offline version is free.
- ▶ NIH Image for (old) Macs or Scion Image for Windows.
- ▶ ImageJ is a free software package that runs on any system thanks to Java. With the right plugin, ImageJ can read "Digital Micrograph" images. The image analysis is very powerfull but a little less intuitive than in Digital Micrograph.

We will use **Digital Micrograph** in this course. The download link is given on blackboard.

Please install this program till next lecture.



There are three programs commonly used to analyze EELS spectra:

- ▶ Digital Micrograph (DM) by Gatan is the most used image acquisition and analysis software at TEMs. The offline version is free but does not include the EELS capabilities normally.
- ▶ Velox by Thermo Fisher.
- ▶ Nion Swift - python based acquisition and analysis software

1925	Louis de Broglie	electron has a wavelike character with a wavelength less than light
1927	Davisson and Germer Thompson and Reed	classic electron diffraction experiments
1932	Knoll and Ruska	first electron lenses and first image (Noble Price 1986)
1936	Vickers	first commercial electron TEM
1939	Siemens and Halske	first usable and profitable TEM
1949	Heidenreich	first transparent metal foil (first materials science result)
2000	Krivanek (STEM) Rose/Heider (TEM)	first prototypes for spherical aberration objective-lens correctors
2007	Krivanek (STEM) Rose/Heider (TEM)	first prototypes for fifth order aberration objective-lens correctors



TEM/STEMs and dedicated STEMs are now available from many sources.

- ▶ ThermoFisher (FEI)
- ▶ Nion
- ▶ Hitachi
- ▶ JEOL

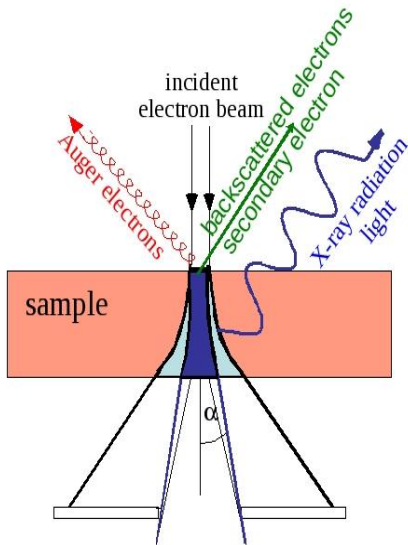
The dedicated STEMs and TEM/STEMs differ by electron source and whether they are equipped with a monochromator.

Monochromated dedicated STEMs by Nion reach energy resolution of about 7 meV, while monochromators from other manufacturer reach around 100 meV.

EELS and EDS spectrometer are attachments, that are available on most TEM/STEMS



To gather information about a sample the electron has to interact with this sample, otherwise it would be invisible. There is a whole zoo of interactions (see schematic in next figure). The primary and most important interaction for TEM imaging is scattering. All the other processes are secondary (for example: X-ray emission).





No Interaction

Most electrons do not interact with a thin specimen at all.

Interaction without Energy Transfer

Elastic scattering is the basis for diffraction and imaging.

Interaction with Energy Transfer

Inelastic scattering causes a diffuse background in images and diffraction pattern, but can be used for analytical TEM.



Some techniques:

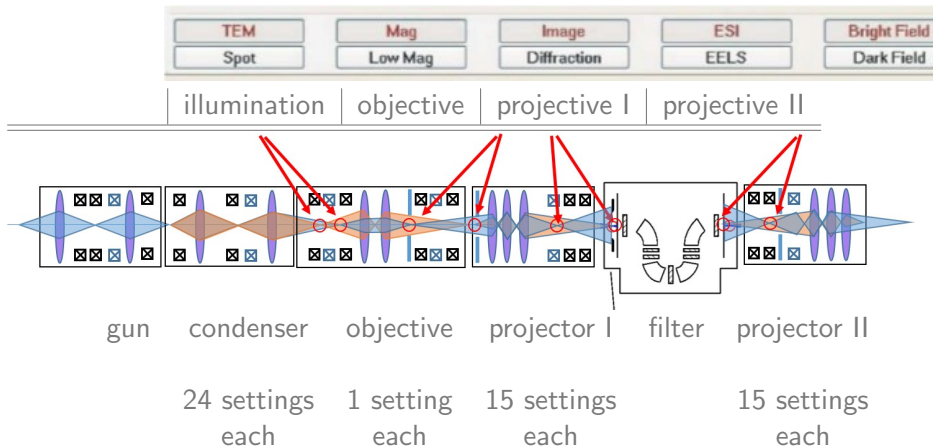
SAED	selected area electron diffraction
CBED	convergent beam electron diffraction
Kikuchi	Kikuchi diffraction
Fresnel	Fresnel diffraction
CTEM	conventional transmission electron microscopy
BF	bright field imaging
DF	dark field imaging
HRTEM	high resolution (phase contrast) transmission electron microscopy
SE	secondary electron imaging
BE	backscattered electron imaging
Lorentz	Lorentz microscopy
HAADF	high angle annular dark field imaging (Z-contrast)

Modes of a TEM/STEM



	illumination	objective	projective I	projective II
TEM imaging	TEM	Mag	Image	ESI
Nanoprobe	Spot	Spot	Image	ESI
LowMag imaging	TEM	Mag	LowMag	ESI
Microprobe	Spot	LowMag	Image	ESI
SAED	TEM	Mag	Diffr	ESI
Low angle diffraction	TEM	LowMag	Diffr	ESI
CBED	Spot	Mag	Diffr	ESI
LACBED	Spot	LowMag	Diffr	EELS
Spectroscopy	TEM	Mag	Image	EELS
Spectroscopy	Spot	Mag	Diffr	EELS
STEM	Spot	Mag	Image	ESI
STEM-LM	Spot	LowMag	Diffr	ESI
STEM-SI	Spot	Mag	Diffr	EELS

Modes of a TEM: Example Zeiss Libra





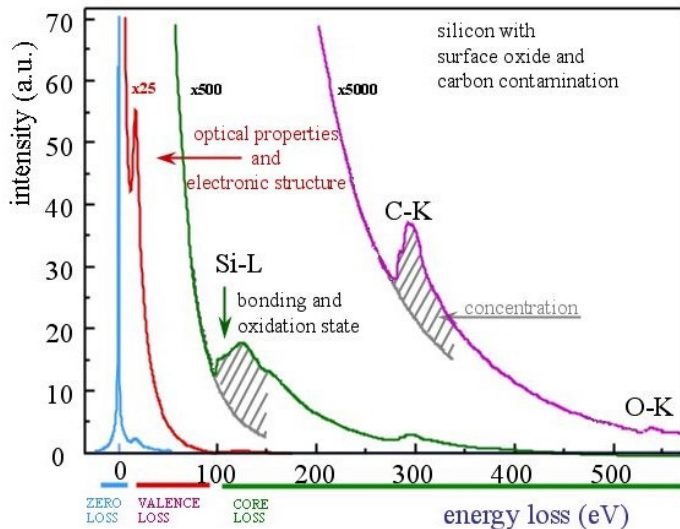


- ▶ Diffraction is the direct result of the interaction (without energy transfer) of electrons and matter.
- ▶ Kinematic diffraction theory describes only the Bragg angles (the position) but not the intensity in a real diffraction pattern.
- ▶ Dynamic theory is responsible for the intensity distribution



- ▶ To form an image from a diffraction pattern only a Fourier transformation of parts of the diffraction pattern is needed.
- ▶ Any image in a TEM can be described as Fourier filtering, because we select beams. The knowledge of which and how many diffracted beams contribute to the image formation is crucial.
- ▶ Because the intensity of selected diffracted beams is necessary to calculate image intensities, dynamic theory is necessary.
- ▶ Understanding Diffraction theory of electrons is the core of the analysis of TEM data.

Electron Energy-Loss Spectroscopy





No Energy Transfer

The zero-loss peak is caused by electrons of the acceleration energy which apparently did not lose any energy.

Little Energy Transfer: 1-70 eV

The valence-loss region shows intraband, interband, and plasmon transitions.

High Energy Transfer: above 70eV

The core-loss region contains excitation from the atom core levels into the conduction band appear as saw tooth like edges.

Secondary Processes

After excitation through the incident electrons, the atoms will go back to their ground state and emit the gained energies as photons (in the light and X-ray region) or (Auger-) electrons. These secondary processes are also used for analytical analysis such as:

- ▶ Energy Dispersive X-Ray Spectroscopy (EDS)
- ▶ Auger-Spectroscopy
- ▶ Cathodoluminescence



Why do we use electron microscopes



Why do we use electron microscopes

Spatial resolution



What are the drawbacks of a transmission electron microscope?



What are the drawbacks of a transmission electron microscope?

- ▶ Sample preparation is tedious and can induce artifacts.
- ▶ Sampling: Only a small area is getting investigated.
- ▶ Electron beam damage
- ▶ Sample contamination
- ▶ Image/data interpretation is not easy:
- ▶ A micrograph is a projection only, and high resolution images must be simulated.
- ▶ The instruments are under vacuum and are generally fragile, which results in long experimental times.



What electron microscopes are there?



What electron microscopes are there?

- ▶ Scanning electron microscopes: SEM,
- ▶ Transmission electron microscopes: TEM (high [1MV], medium [60-400kV] and low voltage [5-60 keV]),
- ▶ Scanning transmission electron microscopes: STEM



What techniques are there?



Important Questions

What techniques are there?

- ▶ Diffraction
- ▶ Imaging
- ▶ Analytic TEM



When do we use what technique?



Important Questions

When do we use what technique?

Material and question dependent
– the big thing to learn in this course



- ▶ The TEM enables many powerful techniques.
- ▶ The TEM is only useful to solve problems needing spatially resolved information
- ▶ The TEM is most powerful with complementary (less spatially resolved) techniques
- ▶ Outlook
 - ▶ The interpretation of selective area diffraction.
 - ▶ Next: Geometric Ray Optics.

Read your Assignment

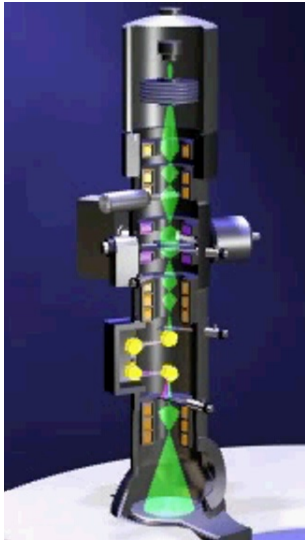
Carter and Williams: Chapter 6



Why do microscopy with electrons?

- ▶ small wavelength
 - ▶ You cannot resolve anything much smaller than your wavelength
- ▶ lenses
 - ▶ The reason one does not have X-ray or neutron microscopes is that there are no lenses for those particles

Atomic scale resolution is possible with electron and ion microscopy



A TEM is a stack of electro-optical elements:

- ▶ electron source
- ▶ electrostatic lens
- ▶ accelerator
- ▶ magnetic lens
- ▶ magnetic and electrostatic deflectors
- ▶ magnetic multipoles
- ▶ apertures
- ▶ detectors (viewing screen, CCD)
- ▶ sample holder

Electron Optics - Overview of the Whole System



We start on the top.

The Electron Gun produces the electrons, focuses them and accelerates them.

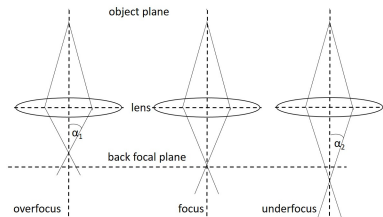
The Condenser Lens System varies the beam size, the illumination area and the convergence angle.

The Objective Lens does the maximum magnification in imaging mode.

The Intermediate Lens System switches between imaging and diffraction mode. The objective lens does not magnify anything in diffraction mode, because the back focal plane of the objective lens is object plane of the intermediate plane. The intermediate lens does the maximum magnification.

The Projector Lens System magnifies everything roughly to the magnification indicated on the display/chosen on the console.

The microscope consists of a stack of lenses in which the image plane of one lens is the object plane of the next one. The focal length f of a magnetic lens can be changed by its electric current. If the field is too weak, we call it underfocus, is it too strong we speak of overfocus.



Electron Optics in Python



This is part of the optics notebook to visualize condenser–lens settings.

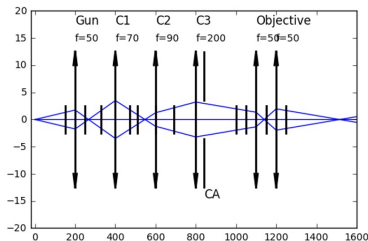
Condensor Ray Diagram

```
In [7]: # Define lenses here
lenses =      ['Gun', 'C1', 'C2', 'C3', 'Objective', '']
zl = np.array([200.0, 400.0, 600.0, 800.0, 1100, 1200    ]) # lens positions
ff = np.array([50.0, 70.0, 90.0, 200.0 , 50, 50]) # lens focal lengths for Spot Mode in TEM
#ff = np.array([50.0, 70.0, 90.0, 142.0 , 50, 50]) # Koehler Illumination

propagate_beam((0,),1, 3, zl, ff, 'DM', 'b')
#      add the lenses
for i in range(np.size(zl)): add_lens(zl[i], ff[i], 25, lenses[i])
add_aperture(840, 25, 7, 'CA')

#      plot optical axis and title
plt.axis([-20,zmax, -20, 20])
```

Out[7]: [-20, 1600.0, -20, 20]



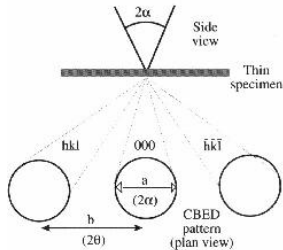


Ray Optics of TEM

- ▶ Lenses provide illumination and magnification
- ▶ Lenses switch modes in a TEM
- ▶ Lenses allow to select the angles within a TEM
- ▶ Apertures help to select the information projected in a TEM

A detailed understanding of the angles within the TEM allows for a project oriented experimental setup.

Condenser Lenses: Convergence Angle



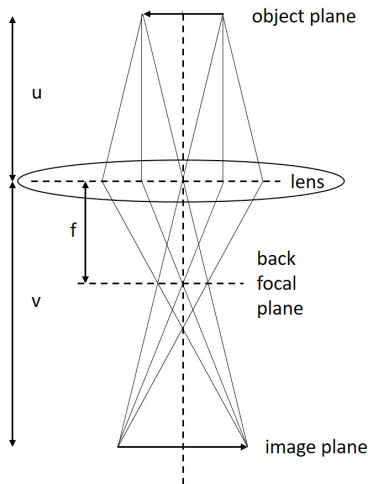
The convergence angle is set by the condenser lens system and can be measured with a convergent beam electron diffraction (CBED) pattern. Under plane wave illumination the diffraction pattern consists of points. If you imagine a second incident plane wave with some angle to the first one, there will be two slightly shifted diffraction patterns made out of points. A lot of plane waves with a conical angle distribution will result in small circles instead of a point. The radius of the circle corresponds to the convergence angle. One can easily calculate the convergence angle from a CBED pattern of a known sample.

The magnification of a lens is given by Newton's law:

$$\frac{1}{u} + \frac{1}{v} = \frac{1}{f} \quad (1)$$

The magnification M is defined as

$$M = \frac{v}{u} \quad (2)$$





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- ▶ Any image in a TEM can be described as Fourier Filtering, because we select the beams that form the images. The knowledge of which and how many diffracted beams contribute to the image formation is crucial.
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- ▶ Understanding Diffraction theory of electrons is the core of the analysis of TEM data.



- ▶ Kinematic Theory is based on a single scattering event per electron
- ▶ Kinematic theory is used in neutron and X-ray diffraction almost exclusively.
- ▶ Dynamic theory incorporates multi scattering events.
- ▶ Dynamic theory results in *Rocking Curves* of intensities of diffracted beams with sample thickness.
- ▶ Dynamic theory can analytically be solved only for the two beam case, strangely the basis for conventional TEM.



f	scattering factor
F	form factor
	forbidden reflection
σ	cross-section
$\frac{\partial \sigma}{\partial \Omega}$	partial cross-section
λ	mean free path



f	scattering factor	scattering Strength of an atom
F	form factor	combination of symmetry and atomic scattering factor
	forbidden reflection	is a direct result of the form factor
σ	cross-section	scattering probability expressed as an effective area
$\frac{\partial \sigma}{\partial \Omega}$	partial cross-section	scattering probability in a solid angle Ω
λ	mean free path	scattering probability expressed as a path length between two scattering events



Bethe Theory

Bloch Wave

Rocking Curve

Extinction Distance

Howie–Wheeland Equation

Two Beam Condition

Dynamically. Activated Reflection

Multi-Slice Theory

Fourier Coefficients



Bethe Theory

Bethe calculated first the dynamic theory of electron diffraction

Bloch Wave

A plane wave that has the same spatial frequency as the crystal potential in the direction of this beam. A set of Bloch waves are the solution to the electron diffraction Schrödinger equation.

Rocking Curve

The intensity variation of a single diffracted beam with thickness

Extinction Distance

The distance within the sample a diffracted beam has to travel which is equivalent to one period in the rocking curve. Usually defined by where a reflected beam has its first minimum.

Dynamic Diffraction Theory Buzz Words II



Howie–Wheeland Equation	Describes the dynamic theory but are not based on Quantum Mechanics.
Two Beam Condition	Only two beams are strongly active within the sample, all others are ignored.
Dynamically Activated Reflection	Some forbidden reflections can be activated by double (dynamic) diffraction and become visible.
Multi-Slice Theory	The sample will be divided in a set of slices and there will be a interaction and a propagation operator for each of these slices
Fourier Coefficients	The <i>Intensities</i> for Bloch wave contributions. The result of a Bethe theory calculation.