# Machine Learning in STEM 2023 Lecture 02

## Lecture 2: Introduction to TEM

Gerd Duscher

Materials Science and Engineering
The University of Tennessee, Knoxville

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# Outline

# Literature

- Williams and Carter
  Transmission Electron Microscopy
  Plenum Press, 2009 ($2^{nd}$ Edition).
  available online at the Library
- Reimer and Kohl
  Transmission Electron Microscopy
  Springer, Berlin, 2007 ($5^{th}$ 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 ($2^{nd}$ Edition).
  available online at the Library

# Literature

- R.F. Egerton.
  Electron Energy–Loss Spectroscopy in the Electron Microscope.
  Plenum Press, 2011 ($3^{rd}$ 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.

# Software–Image Analysis

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

# History

| | | |
|---|---|---|
| 1925 | Louis de Broglie | electron has a wavelike character with a wavelength less than light |
| 1927 | Davisson and Germer<br>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)<br>Rose/Heider (TEM) | first prototypes for spherical aberration objective-lens correctors |
| 2007 | Krivanek (STEM)<br>Rose/Heider (TEM) | first prototypes for fifth order aberration objective-lens correctors |

# Available TEM/STEMs

TEM/STEMs and dedicated STEMs are now available from many sources FEI, Hitachi, JEOL, NION).

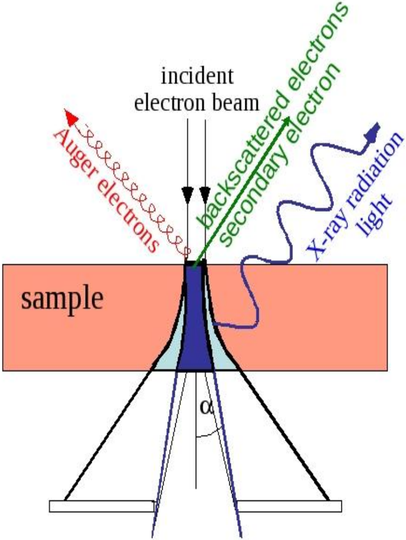
- ThermoFisher (FEI)
- Nion
- Hitachi
- JEOL

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.

# Electron Meets Matter

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).

# Electron Meets Matter



# Diffraction and Imaging

### 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.

# Modes of a TEM

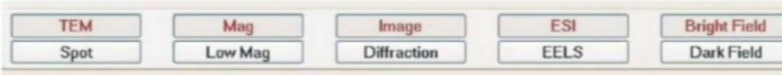
Some techniques:

| | |
|---|---|
| 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) |

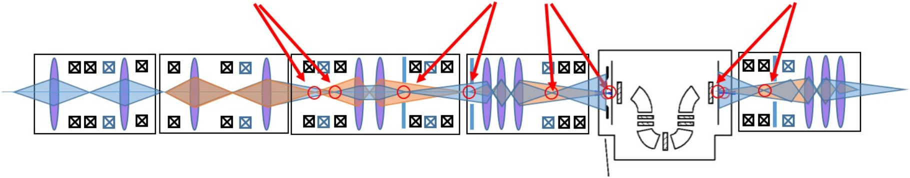
# Modes of a TEM/STEM

| | 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




| | illumination | objective | projective I | projective II |
|---|---|---|---|---|



| gun | condenser | objective | projector I | filter | projector II |
|---|---|---|---|---|---|
| | 24 settings each | 1 setting each | 15 settings each | | 15 settings each |

# Diffraction and Imaging

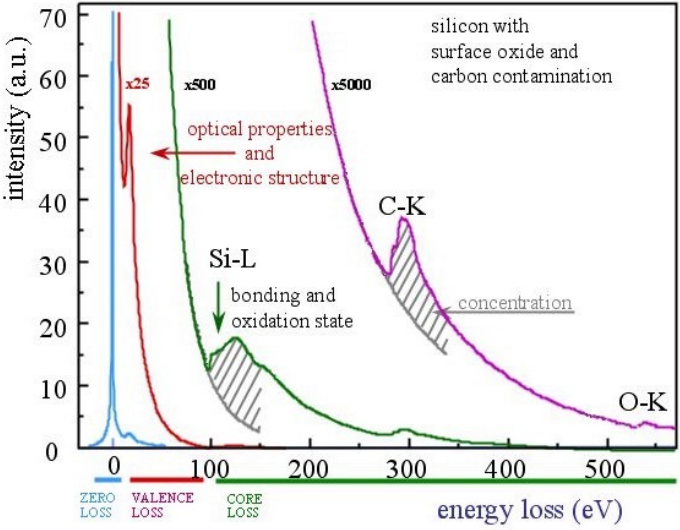
# Basics of Diffraction

- 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

# Diffraction and Imaging

- 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



# Electron Energy-Loss Spectroscopy

### No Energy Transfer

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

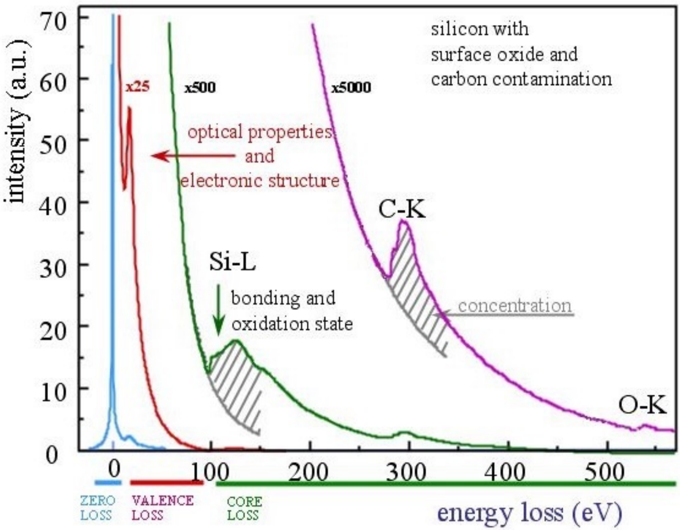
### Little Energy Transfer: 1-70 eV

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.

# Electron Energy-Loss Spectroscopy



# 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

# Important Questions

**Why do we use electron microscopes**

# Important Questions

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Spatial resolution

# Important Questions

**What are the drawbacks of a transmission electron microscope?**

# Important Questions

**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.

# Important Questions

**What electron microscopes are there?**

# Important Questions

**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

# Important Questions

**What techniques are there?**

# Important Questions

**What techniques are there?**

- Diffraction
- Imaging
- Analytic TEM

# Important Questions

**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

# Summary

- 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**

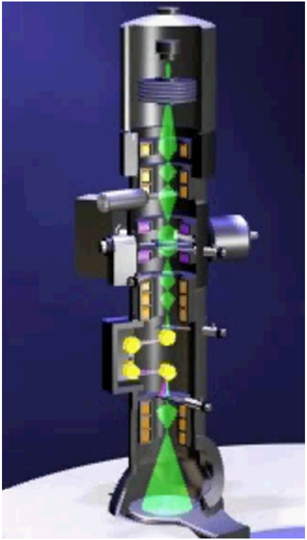
Carter and Williams: Chapter 6

# Electron Microscopy

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:

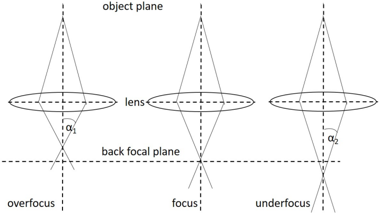
- electron source
- electrostatic lens
- accelerator
- magnetic lens
- magnetic and electrostatic deflectors
- magnetic multipoles
- apertures
- detectors (viewing screen, CCD)
- sample holder

# 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.

# Electron Optics - Ray Diagrams

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.

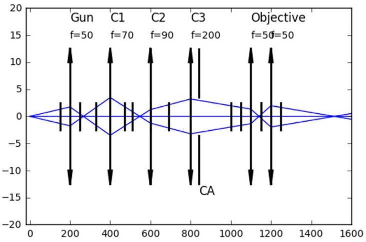
Condensor Ray Diagram

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]



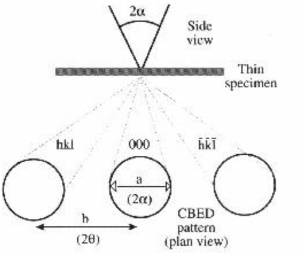
# Electron Optics in Python

## 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.

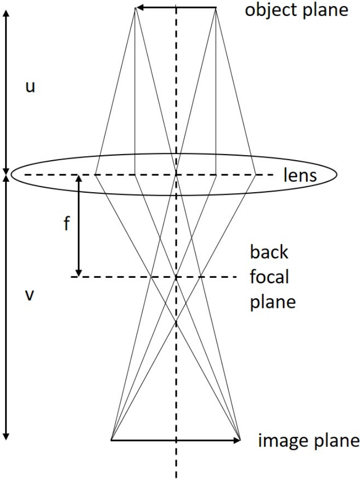
# Electron Optics - Ray Diagrams

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

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

The magnification $M$ is defined as

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



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# Dynamic and Kinematic Theory

- 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.

# Kinematic Diffraction Theory Buzz Words

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

# Diffraction Theory Buzz Words

| | | |
|---|---|---|
| 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 |
| $\sigma$ | cross-section | scattering probability expressed as an effective area |
| $\frac{\partial\sigma}{\partial\Omega}$ | partial cross-section | scattering probability in a solid angle $\Omega$ |
| $\lambda$ | mean free path | scattering probability expressed as a path length between two scattering events |

# Dynamic Diffraction Theory Buzz Words

Bethe Theory
Bloch Wave
Rocking Curve
Extinction Distance
Howie–Wheeland Equation
Two Beam Condition
Dynamically. Activated Reflection
Multi-Slice Theory
Fourier Coefficients

# Dynamic Diffraction Theory Buzz Words I

| | |
|---|---|
| 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 *Intensities* for Bloch wave contributions. The result of a Bethe theory calculation. |