Chapter 1: Introduction to Transmission Electron Microscopy (TEM)

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Transmission electron microscopy and diffraction
Doctoral school (MSE-637)

1

Schedule of the course

		MSE-637 (b) 18.11.2019	**Transmission Electron Microscopy and Diffraction** 19.11.2019	20.11.2019
			ELD020	ELD020
08:15			6) HRTEM MC	7) Structure factor & crystals DA
09:15	09:00			8) Diffraction analysis & Excerises DA & EO
	10:00	DIA 005		
10:15		1) Introduction to TEM RZ	5) Specimen prep DL	9) Dynamical scattering DA
11:15	11:00	2) TEM components and operation RZ		10) Contrasts in TEM imaging DA
	12:00			11) CBED and NBED mapping DA
		BC02	CIME MXC building	
13:15		3) TEM imaging modes EO	DEMO	RZ Reza Zamani
	14:00		Osiris MXC-030	EO Emad Oveisi
14.15		4) TEM crystallography and diffraction DA	Talos MXC-014	DA Duncan Alexander
15:15	15:00		Specimen preparation MXC-116	MC Marco Cantoni
16:15	16:00			DL Danièle Laub
	17:00			

2

Outline

- What is electron microscopy?
- Why electrons?
- A brief history
- Concept of resolution
- TEM limitations
- Electron-matter interaction

3

What is electron microscopy?

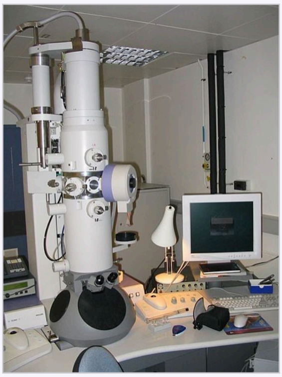
Electron microscope

From Wikipedia, the free encyclopedia

An **electron microscope** is a microscope that uses a beam of accelerated electrons as a source of illumination. As the wavelength of an electron can be up to 100,000 times shorter than that of visible light photons, electron microscopes have a higher resolving power than light microscopes and can reveal the structure of smaller objects. A scanning transmission electron microscope has achieved better than 50 pm resolution in annular dark-field imaging mode[1] and magnifications of up to about 10,000,000× whereas most light microscopes are limited by diffraction to about 200 nm resolution and useful magnifications below 2000×.

Electron microscopes use shaped magnetic fields to form electron optical lens systems that are analogous to the glass lenses of an optical light microscope.

Electron microscopes are used to investigate the ultrastructure of a wide range of biological and inorganic specimens including microorganisms, cells, large molecules, biopsy samples, metals, and crystals. Industrially, electron microscopes are often used for quality control and failure analysis. Modern electron microscopes produce electron micrographs using specialized digital cameras and frame grabbers to capture the images.



A modern transmission electron microscope

4

Electron microscopy

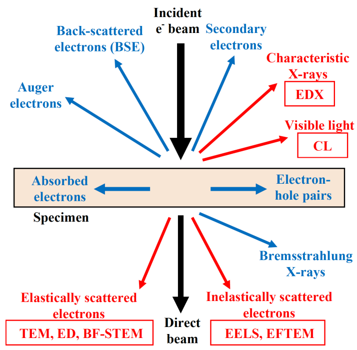
The electron microscope:

- is a very expensive piece of equipment.
- requires stringent environment, free of all sort of disturbance.
- demands expertise, knowledge and experience for data interpretation.
- has its limitations.
- provides information from very small objects with very high resolution.
- provides versatile information due to complex **electron-matter interaction**.

Why electrons?

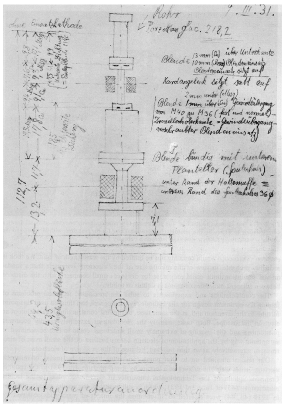
- Limited resolution of light microscopy:
  - (Rayleigh criterion)
- High quality electron probes:
  - Narrow energy distributions
  - Bright beams (high electron dose)
  - adjustable
- Interesting events happening in electron-matter interaction:
  - Elastic/inelastic coherent/incoherent scatterings, diffraction, etc.
- And more…

$$\delta = \frac{0.61\lambda}{\mu \sin \beta}$$



A brief history

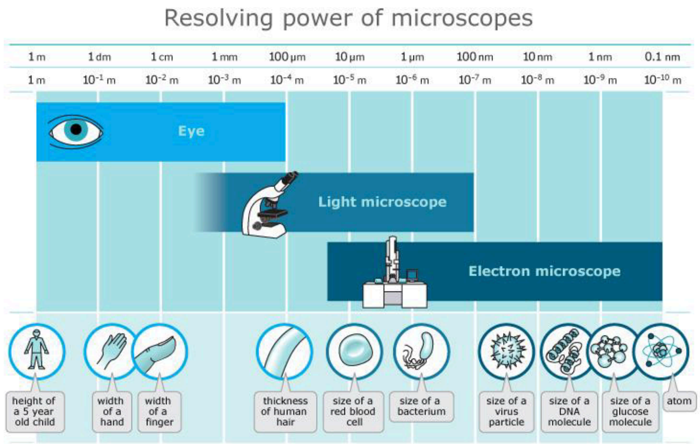
- 1925: Wave-like characteristics of electrons were theorized by Broglie
- 1926: electromagnetic lens was developed by Busch
- 1927: classic electron diffraction experiments were carried out
- 1932: Knoll and Ruska came up with the idea of using electrons for microscopy, built the first TEM and published the first electron images (Ruska won the Nobel Prize in 1966)
- 1936: first commercial TEM was released
- After the WW II: TEMs were widely available

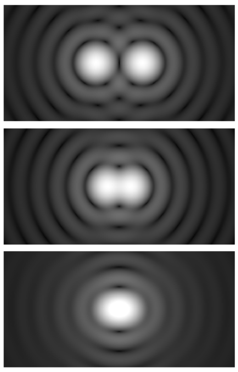


7

Concept of resolution

- Resolution or the resolving power (of a device): smallest distance between to points that can be resolved





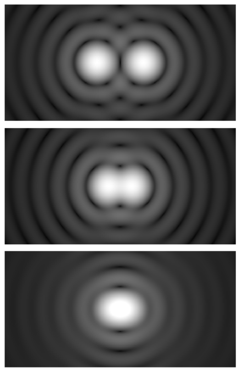
8

Concept of resolution

- Resolution or the resolving power (of a device): smallest distance between to points that can be resolved
- Rayleigh's resolution limit for light microscopy:

$$\delta = \frac{0.61\lambda}{\mu \sin\beta}$$

  - μ: refractive index of the medium
  - β: collection semi-angle
  - λ: wavelength of visible light (let's say 400nm for violet)
  - $\rightarrow \delta \geq 200\ nm$



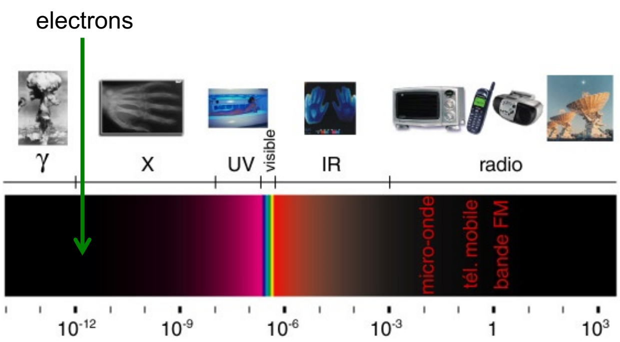
The shorter the wavelength, the higher the resolution

9

Concept of resolution

Electron wavelength according to de Broglie equation: $\lambda = \hbar/p$

with $p = m_o v = (2m_o eV)^{1/2}$



non relativistic (<50keV):

$$\lambda = \frac{h}{(2m_0 eV)^{1/2}}$$

Relativistic correction (>50keV):

$$\lambda = \frac{h}{\left[2m_0 eV\left(1 + \frac{eV}{2m_0 c^2}\right)\right]^{1/2}}$$

10

Concept of resolution

TABLE 1.1 Fundamental Constants and Definitions

Charge (e)	(–) 1.602 × 10^{-19} C
1 eV	1.602 × 10^{-19} J
Rest mass (m_0)	9.109 × 10^{-31} kg
Rest energy (m_0c^2)	511 keV
Kinetic energy (charge × voltage)	1.602 × 10^{-19} N m (for 1 volt potential) = J
Planck's constant (h)	6.626 × 10^{-34} N m s
1 A	1 C/s
Speed of light in vacuum (c)	2.998 × 10^8 m/s

Accelerating voltage [KV]	Nonrelativistic λ [nm]	Relativistic λ [nm]	Mass [x m_0]	Velocity [x 10^8 m/s]
1	0.03879	0.03878	1.002	0.13
10	0.01227	0.01221	1.02	0.42
80	0.00434	0.00418	1.157	1.1
200	0.00274	0.00251	1.391	1.59
300	0.00224	0.00197	1.587	1.82
1000	0.00123	0.00087	2.957	2.44

non relativistic (<50keV):

$$\lambda = \frac{h}{(2m_0eV)^{1/2}}$$

Relativistic correction (>50keV):

$$\lambda = \frac{h}{\left[2m_0eV\left(1+\frac{eV}{2m_0c^2}\right)\right]^{1/2}}$$

Concept of resolution

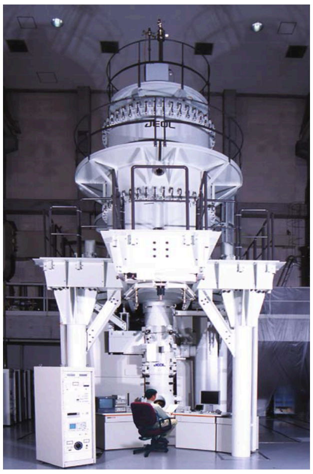
- In TEM we have something similar for resolution:

$$\delta \approx \frac{1.22\lambda}{\beta} \qquad \lambda = \frac{1.22}{\sqrt{E}}$$

  - E: electron beam energy
  - Let's say an optimum β of 20 mrad
  - $\rightarrow \delta \approx 4\ pm\ for\ a\ 100\ keV\ beam$
  - $\rightarrow \delta \approx 1.3\ pm\ for\ a\ 1\ MeV\ beam$

But, lenses are not perfect…

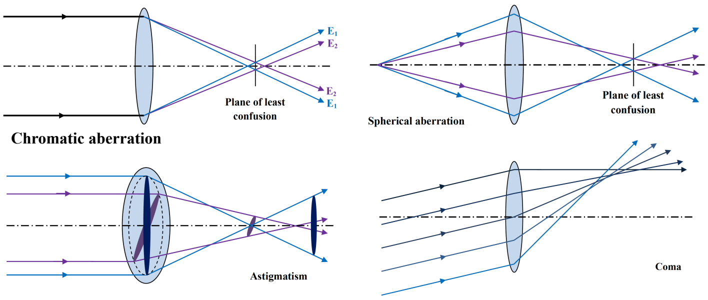
- Does increasing the acceleration voltage improve the resolution?

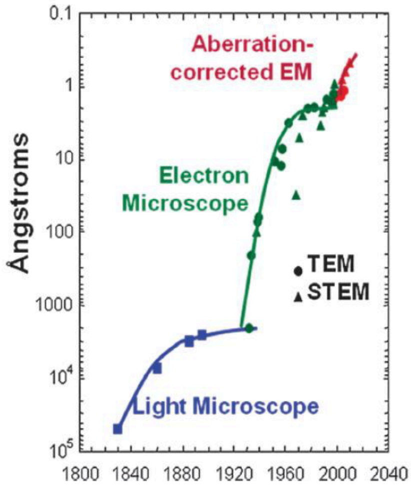


Concept of resolution

- Resolution in the early microscopes wasn't so high due to imperfections in the electron sources and lenses

Aberrations

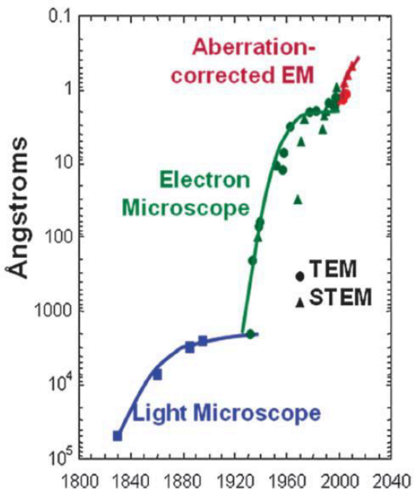




We'll get back to this in the next chapter.

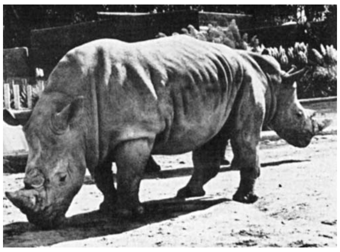
Concept of resolution

- Improvements in electron sources
  (FEG, C-FEG, monochromator, …)
- Improvements in lenses, detectors, vacuum technology, mechanics and electronics, software, and so on…
- Aberration correctors
  (spherical and chromatic)
- Spatial resolution below 0.5 Å is available nowadays!



Limitations of TEM

- Can we see everything with TEM?!
  - Not exactly…
- Some limitations:
  - Data interpretation: We're projecting a 3D object into a 2D image.
  - Knock-on damage: such strong electron beam can modify the material
  - Sample preparation: We need very thin samples (electron transparent)
  - Beamtime is (relatively) expensive!
  - Experience and knowledge is required!

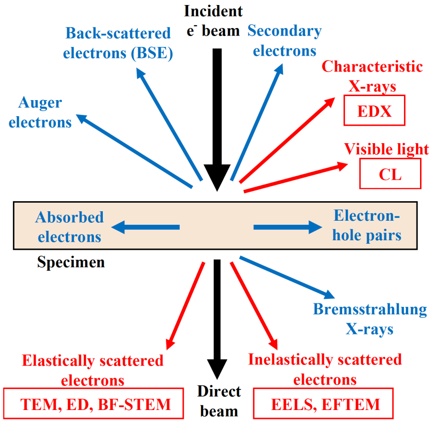


So, plan your experiment wisely…

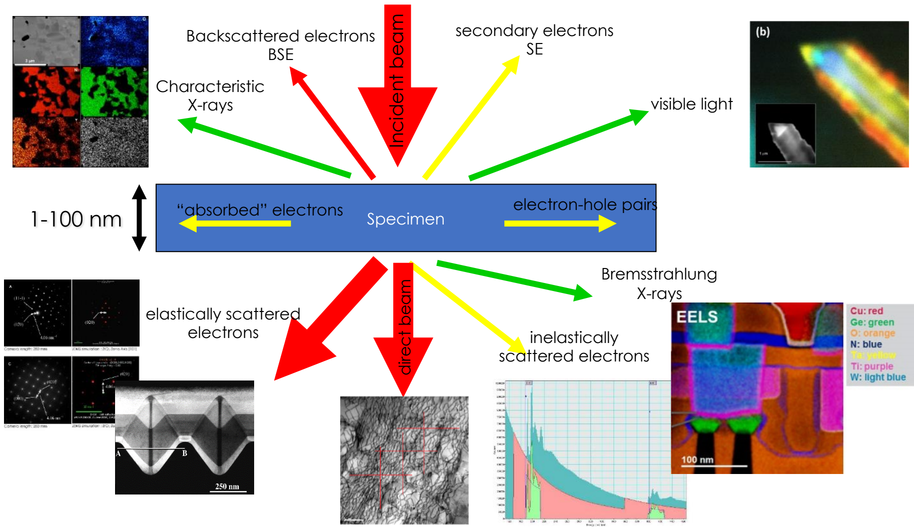
So why using electrons?

- The electrons interact with matter strongly!
- Several events happen, each one produces some unique signals.
- Each signal carries some specific information that can be detected by special detectors and cameras.

Plan your experiment wisely, and you'll get awesome results!!



Electron-matter interaction



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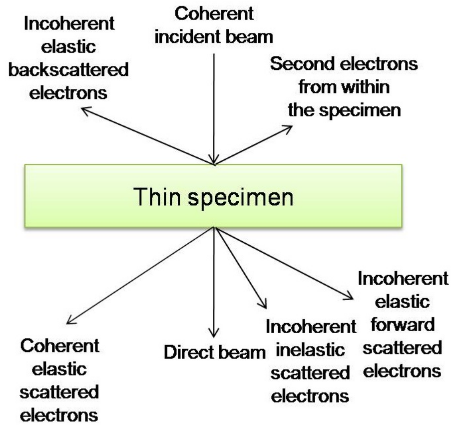
17

Electron-matter interaction

Two Categories of electrons:

1. **Elastically scattered**
   - *Coherent*
     - Bragg diffracted electrons (selected area electron diffraction, bright-field, dark-field, weak beam)
     - Phase Contrast imaging (HRTEM)
   - *Incoherent*
     - Mass-thickness contrast imaging
     - Z-Contrast imaging (HAADF STEM) ***(Under debate!!)***
     - Backscattered electrons
2. **Inelastically scattered**
   - *Secondary signals*
     - Characteristic X-rays and Bremsstrahlung
     - Visible light (Cathodoluminescence)
     - Auger electrons
   - *Incoherent*
     - Secondary Electrons
     - Electron Energy Loss Spectroscopy (EELS)



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18

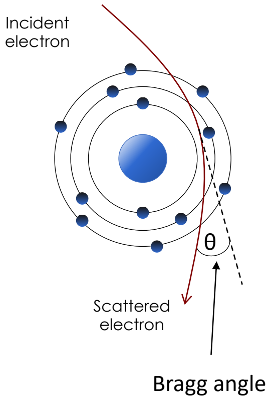
Electron-matter interaction: Elastic scattering

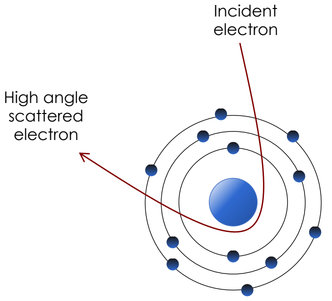
Elastic scattering

No energy transfer

- ❑ Low angle scattering: Coulomb interaction with the electron cloud.
- ❑ High angle scattering (or back scattering): Coulomb interaction with nucleus.
- ❑ Atoms are not ionized.

Example: Bragg diffraction





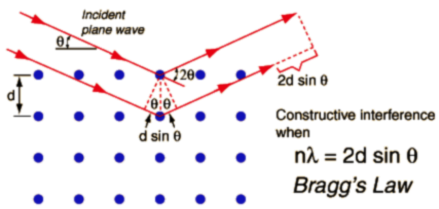
Example: Rutherford scattering, so-called Z-contrast

Electron-matter interaction: Elastic diffraction

Bragg's law

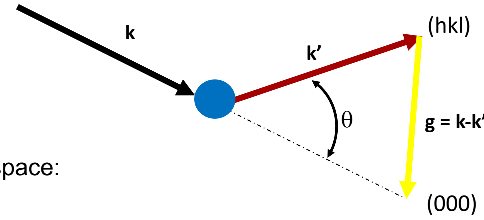
Bragg diffraction occurs when radiation, with a wavelength comparable to the atomic spacing, is scattered by the atom centers and undergoes constructive interference. The path difference, $\boldsymbol{d_{hkl}}$, associated to the scattering angle, θ, is given by

$$d_{hkl} = n\,\lambda/2\,\sin\theta$$



Elastic diffraction

$$|\mathbf{k}| = |\mathbf{k'}|$$



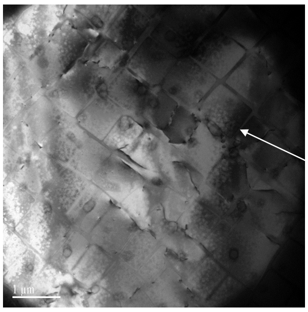
Periodic arrangement of atoms in the real space:
g : vector in the reciprocal space

**Dr. Duncan Alexander & Dr. Emad Oveisi will discuss this topic in detail in their lectures.

Electron-matter interaction: Diffraction contrast imaging

Using Bragg diffracted electrons, you can generate

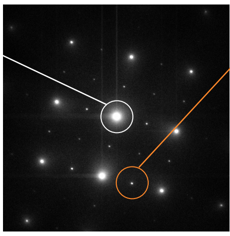
1. Diffraction patterns, which have crystallographic information, i.e., crystal symmetry, lattice parameters, atom postions, etc…
2. Bright-field images that hold information about morphology, grain size, defect structures, etc…
3. Dark-field images, which can be used to correlate crystalline phase and orientation to grain morphologies, distribution with the microstructure, defect structures, etc.

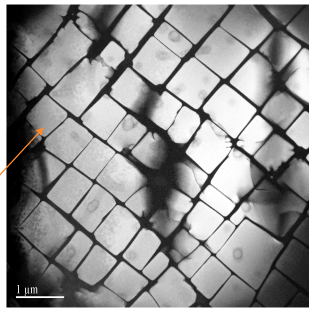


Bright-field TEM image

Nickel based superalloys

Contrast γ/γ'

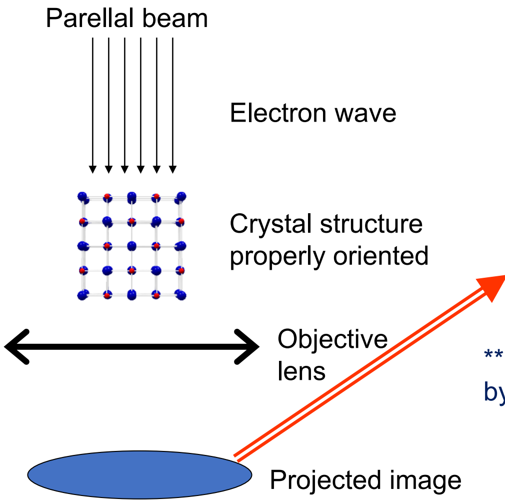


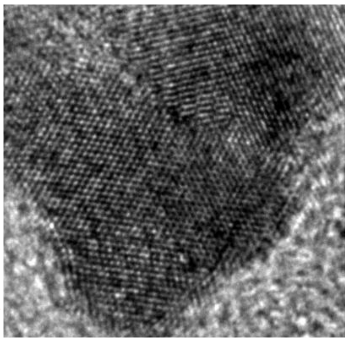


Dark-field TEM image

Electron-matter interaction: Phase contrast & HRTEM

Phase contrast for crystalline specimen





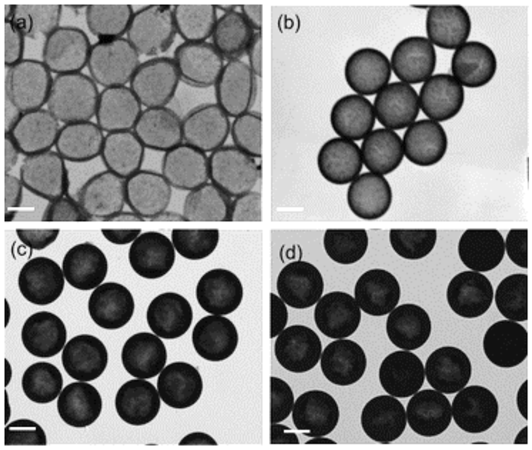
**HRTEM will be discussed in more detail by Dr. Marco Cantoni on Tuesday morning

Electron-matter interaction: Incoherent 'elastic' scattering, mass-thickness contrast

Mass thickness Contrast examples: Hollow polymer spheres with different wall thicknesses

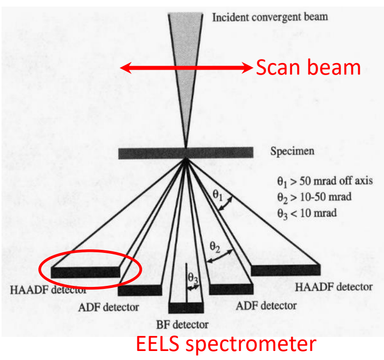
Mass Thickness Contrast:

- Incoherent elastic scattering that results form the difference in the atomic number (Z) and/or thickness (t)
- Scattering is proportional to Z^2 t
- Higher-Z or thicker areas will appear darker in bright-field TEM mode
- Being incoherent, this contrast mechanism applies to both crystalline and amorphous materials



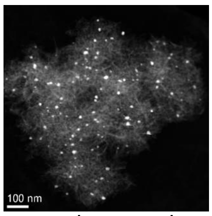
Electron-matter interaction: Incoherent 'elastic' scattering, Z-contrast contrast in STEM

Focused e^- probe scanned on sample; disc and annular detectors in back focal (diffraction) plane

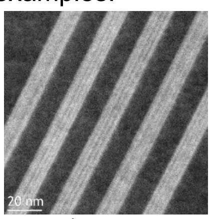


High-angle annular dark-field => compositional contrast: intensity ∝ $Z^2 t$ (thickness t, atomic number Z)

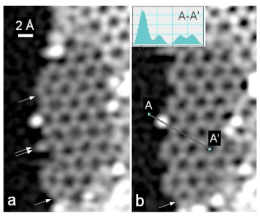
Z-contrast examples:



Pt catalyst on Al_2O_3



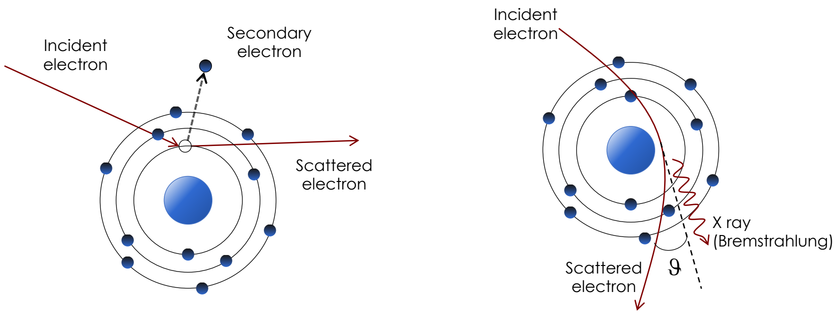
Si-Ge/Si multilayer



Cs-orcrected - graphene with dopant atoms (Krivanek et al., Nion)

Electron-matter interaction: Inelastic scattering

- The incident electron can be scattered by Coulomb interaction with the nucleus, and lose momentum in the form of background X-ray
- An incident electron ejects a bound electron and scatters with an energy lowered by the electron bound energy.
- The ejected electrons having low energies (5-50 eV) are called secondary electrons (SE) and carry information about the surface topography
- In the case of inelastic interaction, there is **energy transfer**, and the target atom can be ionized



25

Electron-matter interaction: Inelastic scattering

Energy absorption

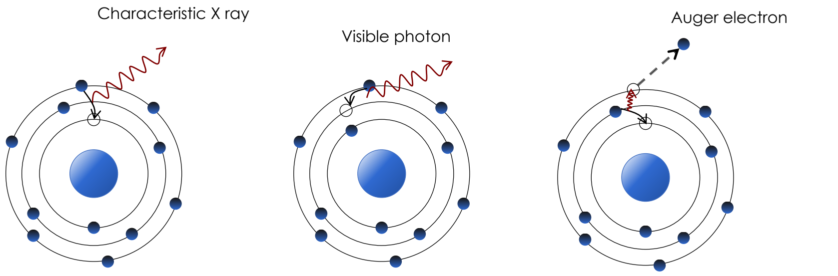
- atom displacement ("knock on")
  - Radiation damage
- chemical bound breaking
  - Radiolysis
- lattice atom vibrations (phonons)
  - Sample heating
- charge collective oscillation excitation (plasmons)
- excitation of surface electronic level (transition valence/conduction,...)
- core atomic level excitation (ionization)
- Bremsstrahlung radiation

The relative impact of these various interaction mechanisms varies with the type of material (no simple modelling of absorption)

26

Electron-matter interaction: Inelastic scattering

Relaxation processes of the excited state



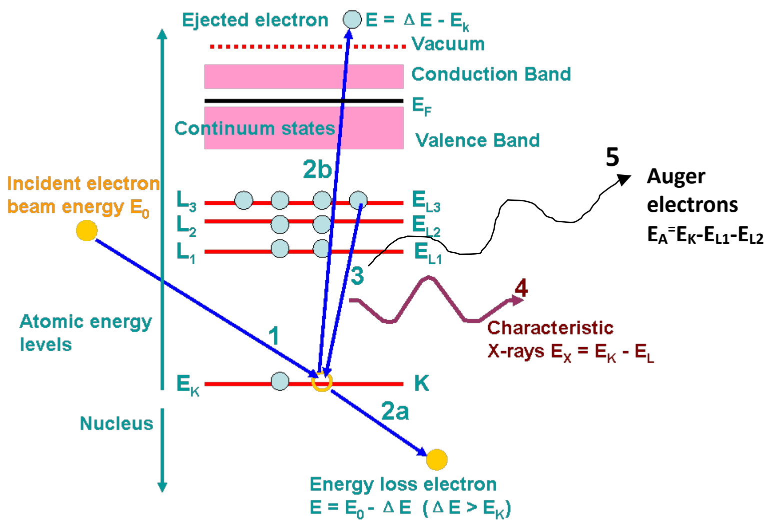
X-ray generation
X ray energy characteristic interorbital electron transitions and thus of the element

Fluorescence
Low transition energy, visible or UV photon is emitted

Emission Auger
The relaxing process interacts with an electron with a characteristic energy

27

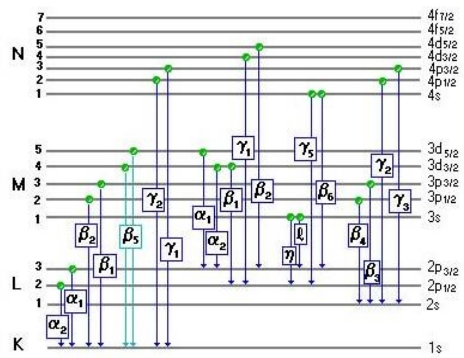
Electron-matter interaction: Inelastic scattering



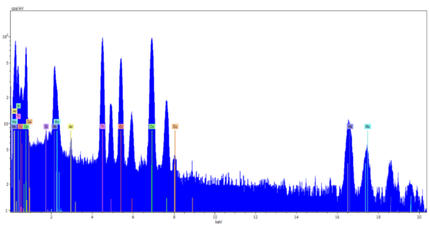
28

Electron-matter interaction: Inelastic scattering

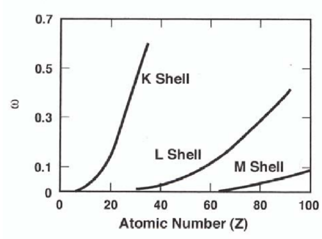
Characteristic X-rays



K- edges L- edges

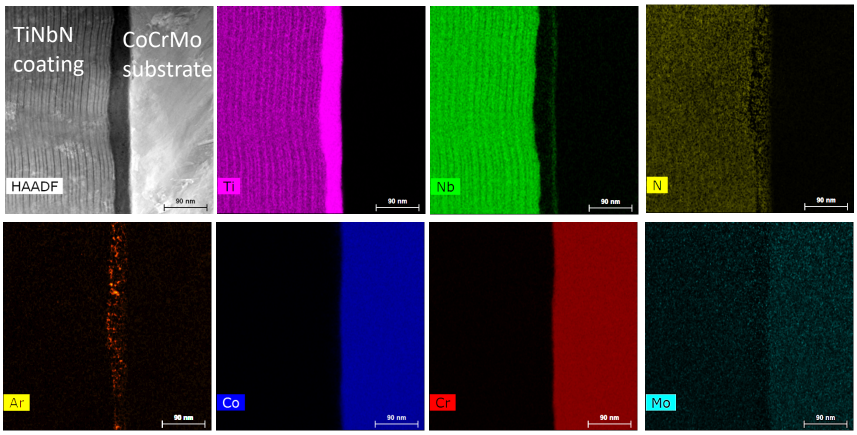


Energy Dispersive Spectroscopy



Electron-matter interaction: Inelastic scattering

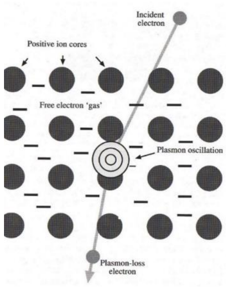
STEM EDS mapping

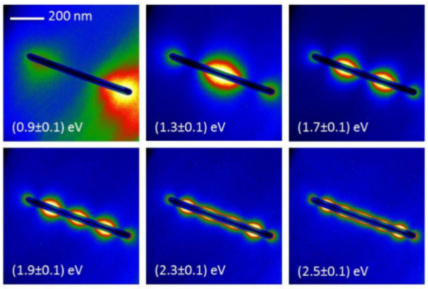


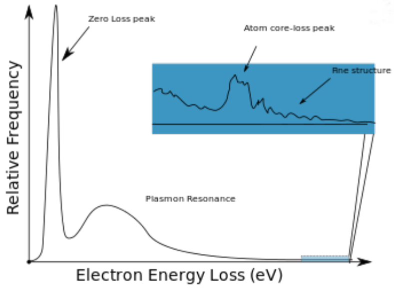
Electron-matter interaction: Inelastic scattering

Interaction with plasmons and core losses

- ❑ Plasmons are collective oscillations of weakly bound electrons – plasmon losses dominate in materials with "free electron" bonding (can be used to calculate thickness)
- ❑ Core losses (atom ionization) depends on atomic species and thus carries some chemical information about the sample as well as bonding, valance states, density of states (pre-edge ELNES), near neighbour coordination (post edge, EXELFS), etc.



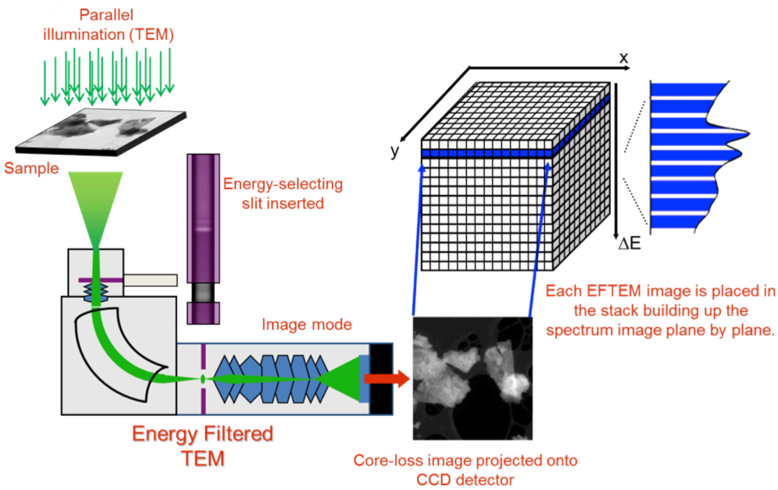




31

Electron-matter interaction: Inelastic scattering

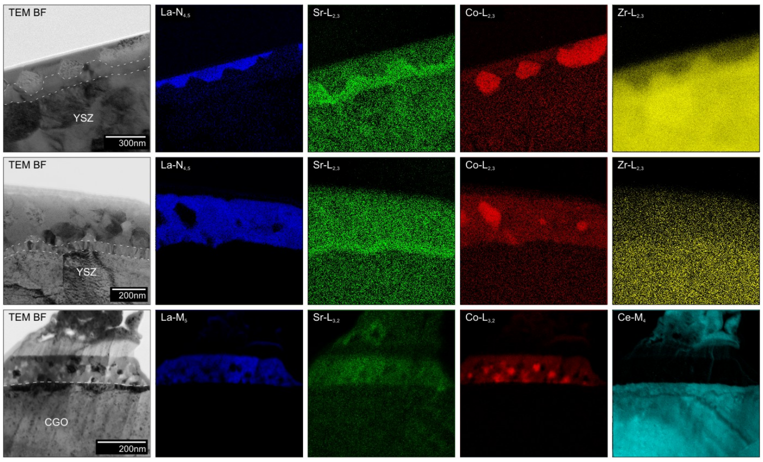
Energy filtered TEM with core loss electrons



32

Electron-matter interaction: Inelastic scattering

Energy filtered TEM with core loss electrons



Example taken from KIT - LEM - Research - Solid oxide fuel cells

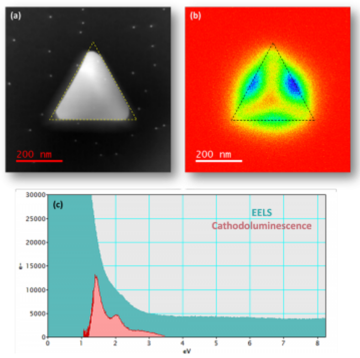
Electron-matter interaction: Inelastic scattering

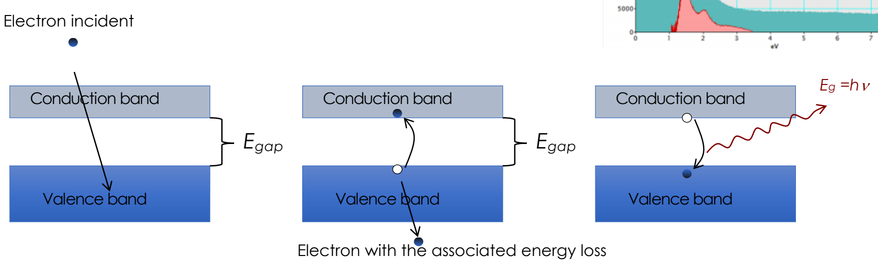
Electron-holes creation:

An incident electron onto a semiconductor can excite a valence electron to the conduction band, creating an electron-hole pair.

Cathodoluminescence:

The excited electron recombines with its hole, emitting a photon with energy equal to the band gap, that is usually in visible range. This technique can be used to understand how defects modify the band gap in nanostructures.





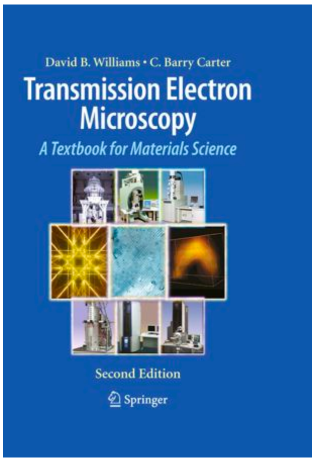
Summary

Why do we use electrons as probe?

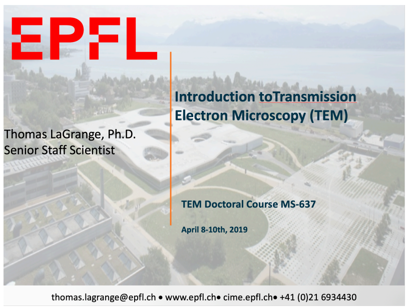
1. Easy to produce high brightness electron beams
   - High coherence beams allow us to generate diffraction patterns and high spatial resolution images
2. Easily manipulated
   - Electron lenses and deflectors can used to easily change focal lengths and beam directions which is a necessary operating condition for flexible imaging devices
3. High energy electrons have a short wavelength
   - Shorter wavelengths means higher spatial resolution (Rayleigh Criterion)
4. Electrons interact strongly with matter
   - Secondary signals have information specific to the material
   - Bragg diffracted electrons –structure, orientation, phase distribution, defect content and structures, etc.

35

References



Williams & Carter TEM textbook



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36