Aalto University

MEC-E6006 Engineering Materials Laboratory L

Electron microscopy in engineering materials

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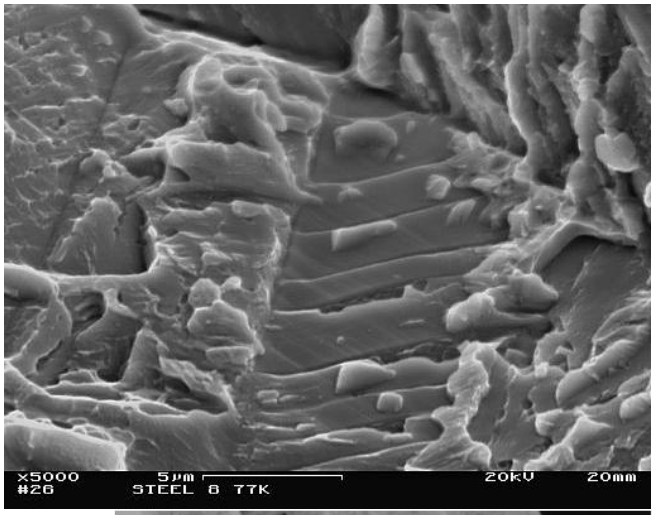
Basics of electron microscopy

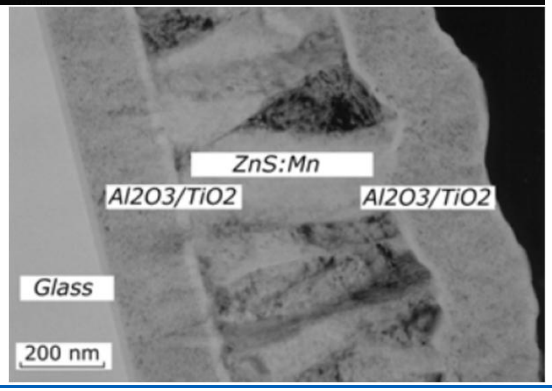
- Electrons are negatively charged particles within the atom
  - Electrons cannot be focused by glass lenses like light photons
  - Electrons can, however, be guided and focused using electromagnets instead of glass lens systems
- The resolution of light microscopy is limited by the wavelength of light
  - The shortest wavelength of visible light is λ = 400 nm (violet)

  Ø The best resolution possible is about 200 nm
- Two most common electron microscope types are transmission electron microscope (TEM) and scanning electron microscope (SEM)
  - SEM has a resolution of a few nm, modern high-resolution FESEMs can achieve resolutions smaller than 1 nm, and a modern aberration corrected HR-TEM can achieve resolutions smaller than 0.1 nm

Ø Electron microscopy provides significantly higher resolution than light microscopy

Scanning electron microscopy (SEM)





- Scanning electron microscopy (SEM) is nowadays the most widely used microscopy technique other than optical microscopy
- Compared to optical microscopy, SEM has significantly higher resolution and significantly higher depth of field
- Relatively easy specimen preparation
- In addition to imaging, SEM provides the capability of performing different types of analyses
  - Analysis of elemental composition (EDS/WDS)
  - Analysis of specimen crystallography (EBSD)

How SEM works

- Scanning electron microscope is an instrument for observing and analyzing the surface of a specimen using a finely focused beam of energetic electrons emitted from the electron gun
- An electron-optical lens system is used to form the electron probe which is then scanned across the surface of the specimen
- Various signals are generated through the interaction between the beam and the specimen and these signals are collected by different types of detectors
- The amplitude of the signal obtained at each position in the raster pattern is then assembled to form an image

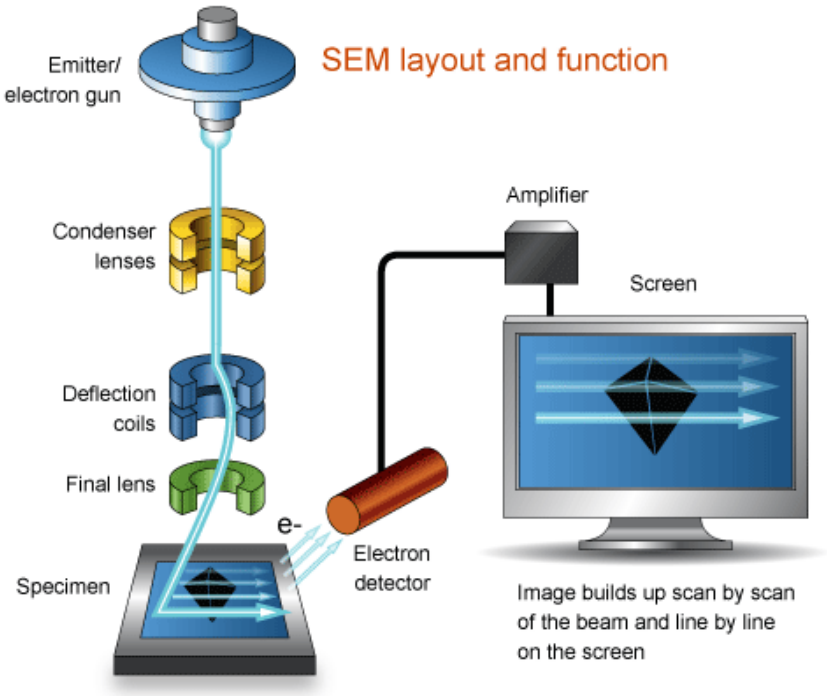


Image formation

- SEM images are formed from the electron intensities obtained at each separate position in the scanned raster pattern
- The scan of the electron beam and the corresponding screen raster are synchronized with intensity proportional to the collected signal
- Because the image is formed only from electron intensity information, SEM micrographs are monochromatic
  - No colors
- Magnification in SEM is defined as the ratio of the length of the line on display device to length scanned on the real specimen
  - For historical reasons, standard magnification is set for 12X10 cm screen
  - Changing the size of the image displayed changes the magnification

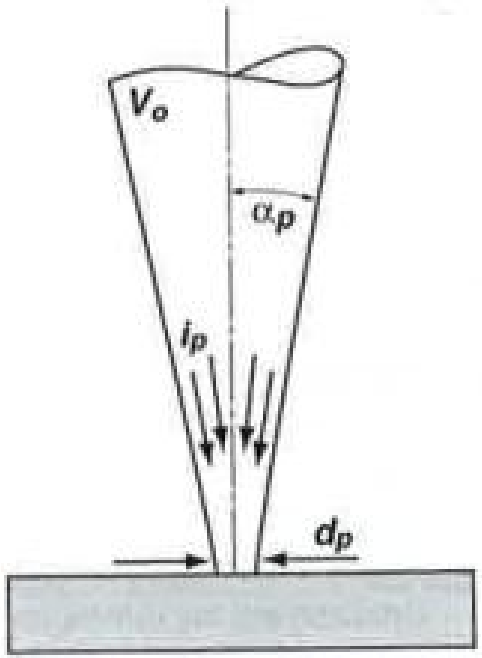
  Ø Always add a scale bar on the image

Specimen preparation

- SEM specimen preparation is relatively easy and straightforward
- The chamber of an SEM is in high vacuum and therefore the specimen must be prepared to withstand the vacuum conditions
  - No wet specimen
  - Specimen must be cleaned from oils, greases, etc.
- Metallographic specimen require a proper grinding and polishing (and possibly fine-polishing) procedures
- Specimen must be electrically conductive
  - Non-conductive specimen will result in specimen charging under the influence of the electron beam
  - Charging disturbs focusing, causes beam astigmatism, etc. making the imaging of the specimen hard/impossible
  - Non-conductivity of the specimen can be handled by different techniques, such as coating the specimen

Electron beam parameters

- Four electron beam parameters define the probe and determine resolution, contrast, and depth of field of SEM images
  - Probe diameter (d_p)
  - Probe current (I_p)
  - Convergence angle (α_p)
  - Accelerating voltage (V_0)
- The operator of an SEM must balance these parameters to optimize the
  - Resolution
  - Depth of field
  - Image quality
  - Analytical performance

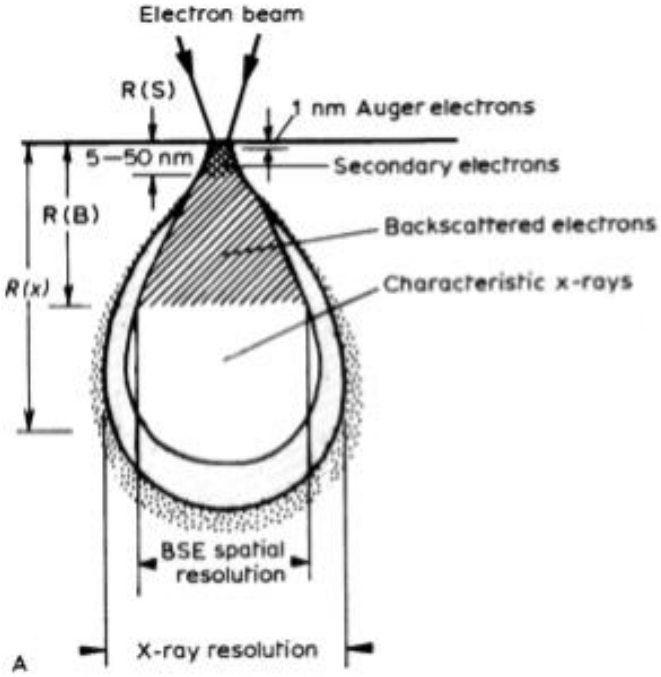


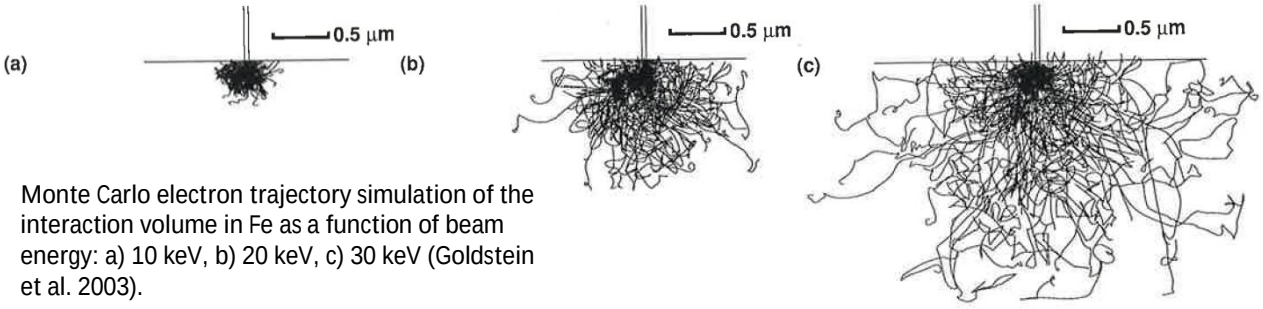
Four major electron beam parameters (Goldstein et al. 2003).

Resolution

- Theoretical resolution (Rayleigh criteria) $R = \frac{0.61\lambda}{NA} = \frac{0.61\lambda}{nsin\theta}$
- The wavelength of an electron accelerated at 10 kV is about 0.012 nm and 200 kV is about λ = 0.0025 nm

Ø The resolution of a scanning electron microscope is not limited by diffraction but by the diameter of the electron probe (spot size) and the interaction of electrons inside the sample (interaction volume)

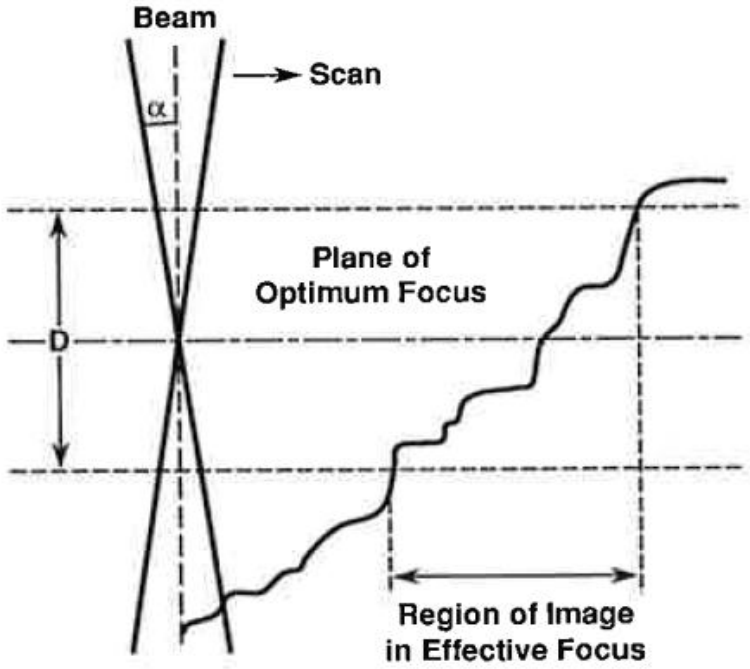




Monte Carlo electron trajectory simulation of the interaction volume in Fe as a function of beam energy: a) 10 keV, b) 20 keV, c) 30 keV (Goldstein et al. 2003).

Depth of field

- One of SEMs significant advantages is significantly high depth of field compared to light microscopy
- Distance above and below the plane of focus in which the beam becomes so broadened that the image does not stay in focus
- The depth of field is a function of convergence angle (α) and magnification
  - Smaller convergence angle and smaller magnification result in higher depth of field

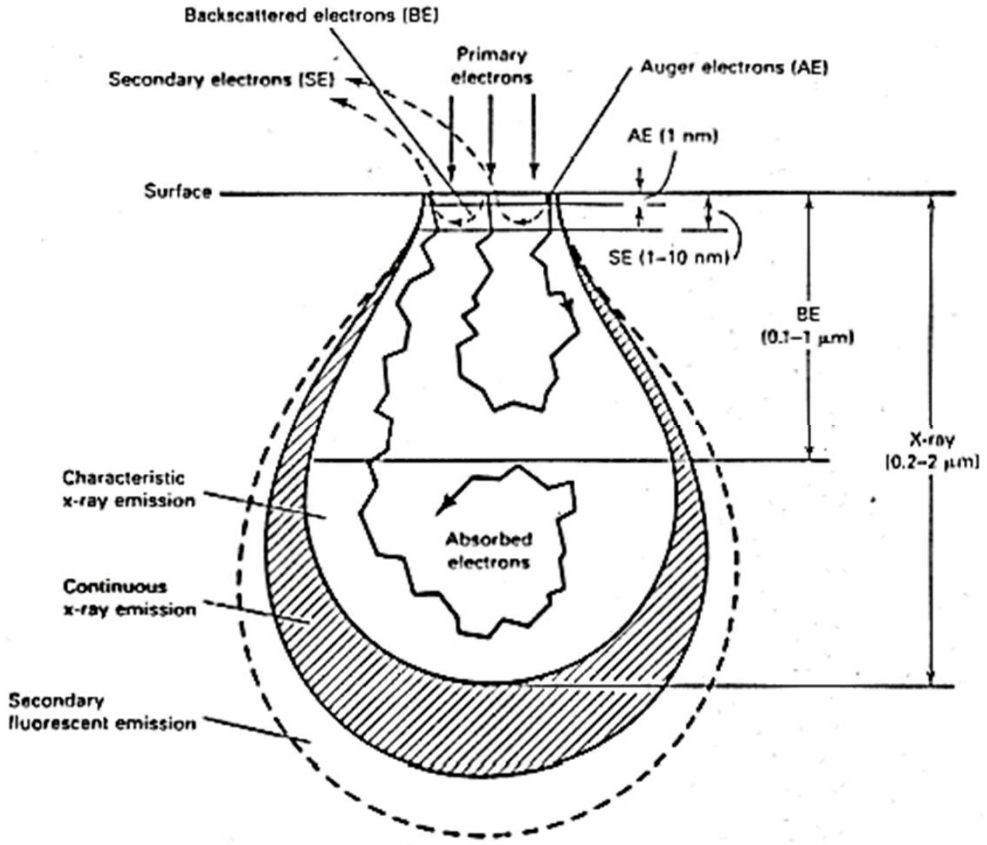


Schematic illustration of the depth of focus (field) in an SEM image (Goldstein et al. 2003).

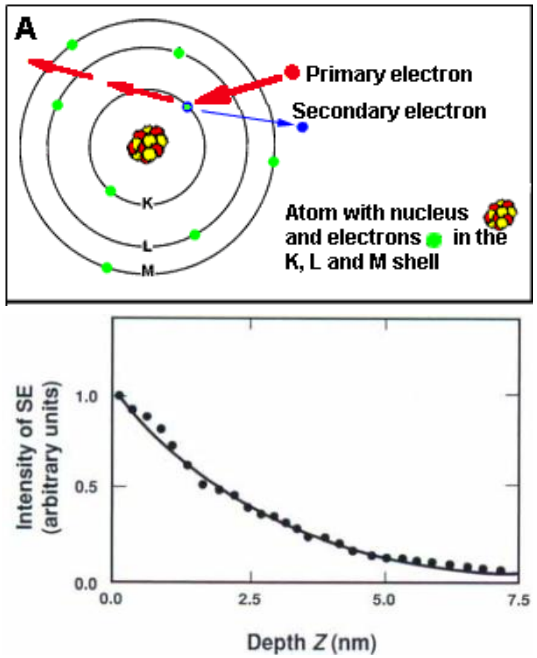
Electron-beam - specimen interactions

The electrons of the electron beam (primary electrons) interact with the atoms in the specimen producing various signals containing different information

- Auger electrons (AE)
- *Secondary electrons (SE)*
- *Backscattered electrons (BSE)*
- *Characteristic X-rays*
- Continuous X-rays (Bremsstrahlung)
- Cathodoluminescence

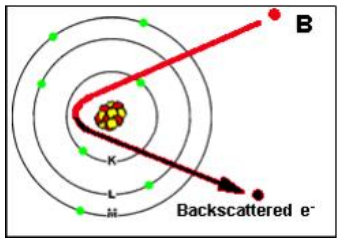


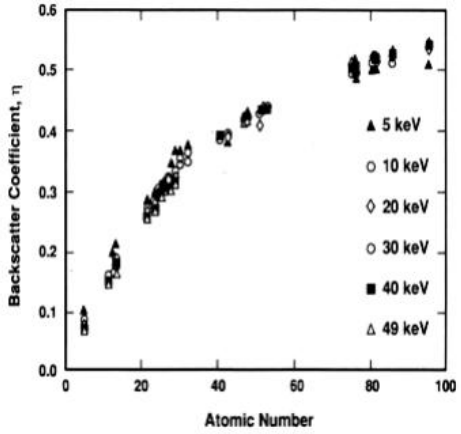
Secondary electrons (SE)

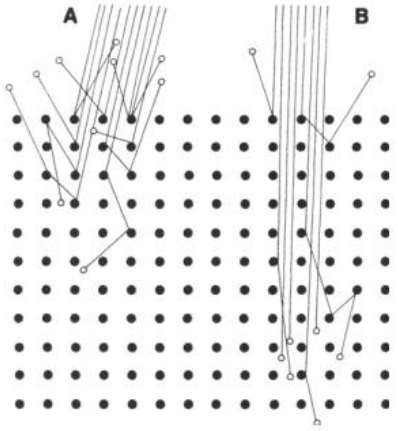


- Secondary electron (SE) is ejected from an atom as a result of primary electron (PE) scattering inelastically from the electron cloud of an atom and ionizing the atom
- Detected using Everhart-Thornley detector consisting primarily of a scintillator inside a Faraday cage in which a low positive voltage is applied to attract relatively low energy electrons
- SEs have quite low energy (~2-50 eV) and only SEs generated near the surface of the specimen can exit and reach the detector (SEs generated deeper inside the specimen recombine)
  - Escape probability for SEs as a function of generation depth à image resolution for SE imaging closer to the size of the probe
- SEs generally give topography contrast in SEM imaging and SEs are the most commonly used source of signal in SEM imaging
- SE yield is higher for heavier elements à SEs also give composition contrast

Backscattered electrons (BSE)







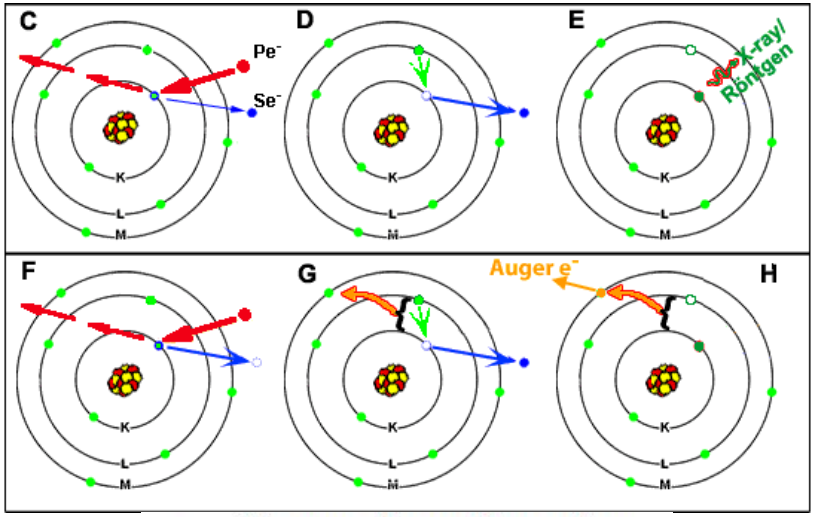
- Backscattering occurs when a primary electron collides with a nucleus of an atom and is elastically scattered to a large angle
- BSEs are primary electrons and, thus, have a high energy
- Scattering cross-section is proportional to the size of the nucleus
- Backscattered electron yield is strongly dependent on mean atomic number of the specimen
  - Atoms of heavier elements cause higher backscatter electron yield

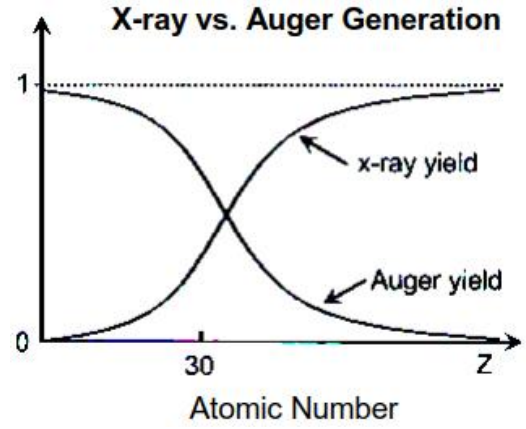
  Ø BSE shows composition contrast
- In crystalline materials, the gaps or channels between the atomic centers provide a path for the beam to penetrate more deeply into the crystal before scattering

  Ø BSE also shows orientation contrast in crystalline materials

Characteristic X-rays

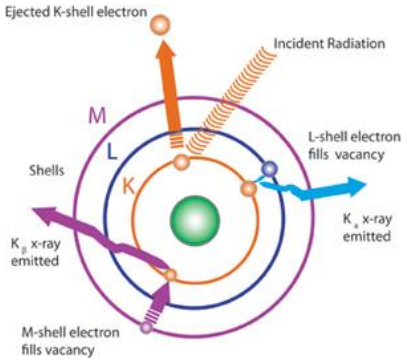
- A scattering event kicks out an electron from an inner shell creating an electron vacancy in the inner shell of an atom
- An electron from an outer shell falls to fill in the vacancy
- Energy difference results in a release of an X-ray of characteristic energy/wavelength or an Auger electron
- Energy/wavelength of the X-ray photon or the yield of the Auger electrons can be measured
- An X-ray emitted by an element has a characteristic energy/wavelength typical to that element
- Characteristic X-rays are generated in a large volume compared to the volume of the SEs or the BSEs

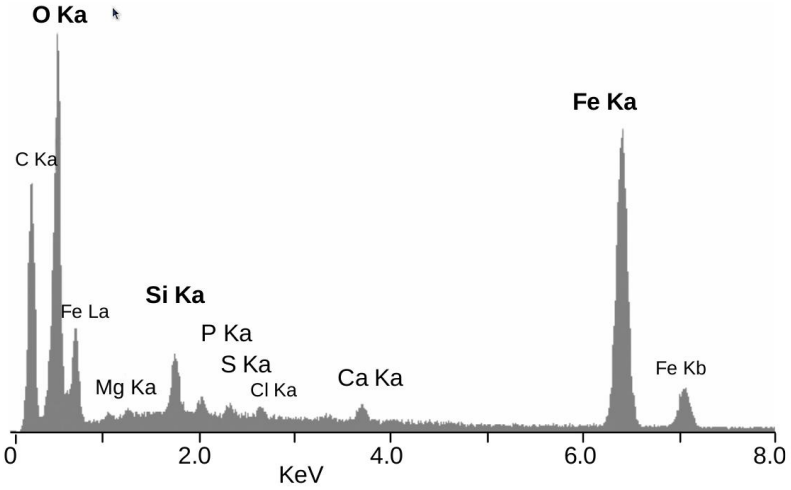




Energy-dispersive X-ray spectroscopy (EDX/EDS)

- In energy-dispersive X-ray spectroscopy (EDS), a solid state detector simultaneously measures full energy range of collected X-ray photons resulting in an EDS spectrum
- Elements are identified from the spectrum based on energies of their characteristic X-rays
- Elements heavier than Be can be qualitatively identified
  - A detection limit of EDS generally around 0,1 wt. %, depending on matrix and composition
  - Overlapping peaks must be taken into account
- Also quantitative analysis is possible using different types of quantification methods



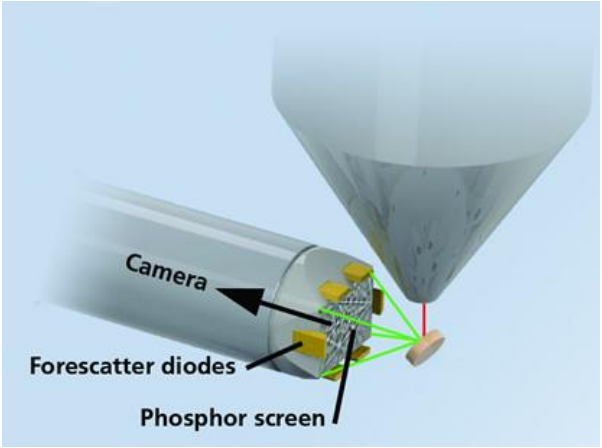


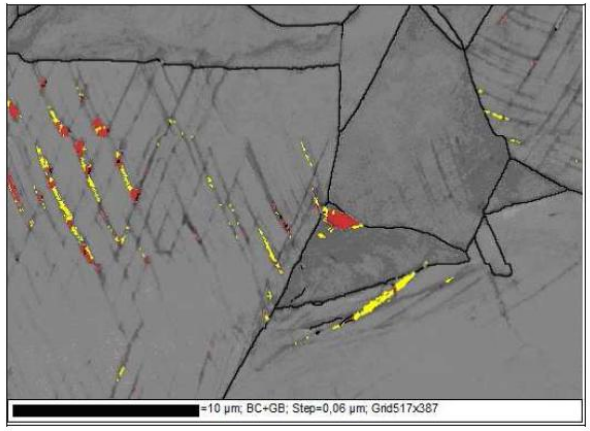
Wavelength-dispersive X-ray spectroscopy (WDS)

- The principle of wavelength-dispersive X-ray spectroscopy (WDS) is based on detecting the same signal than in the EDS, but with a different technique and instrumentation
- In WDS, a number of X-rays of a specific wavelength diffracted by a crystal in the WDS detector are counted
  - Ø WDS has significantly better energy resolution and is more sensitive than EDS
- WDS analysis is performed to the X-rays of a single wavelength at a time
  - Ø WDS is significantly slower than EDS

Electron backscatter diffraction (EBSD)

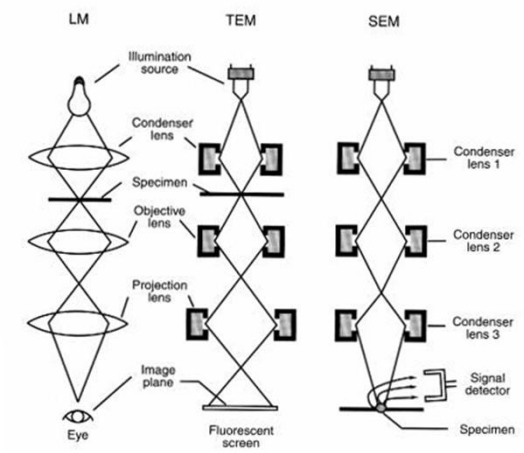
- Electron backscatter diffraction (EBSD) is used to examine crystallography of a specimen in an SEM
- In EBSD, the electron beam interacts with a tilted crystalline specimen and the diffracted backscattered electrons form a diffraction pattern that can be detected
- The diffraction pattern is characteristic of the crystal structure and orientation and can therefore be used to
  - Determine crystal orientation
  - Discriminate between crystallographically different phases
  - Characterize grain boundaries
  - Gather information about local crystalline imperfection

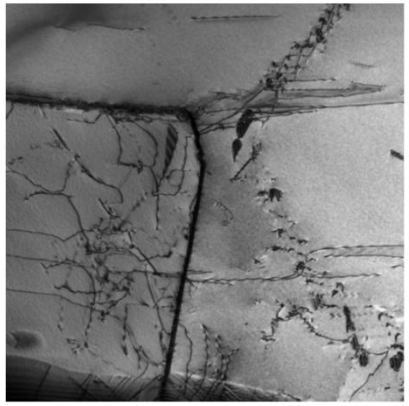


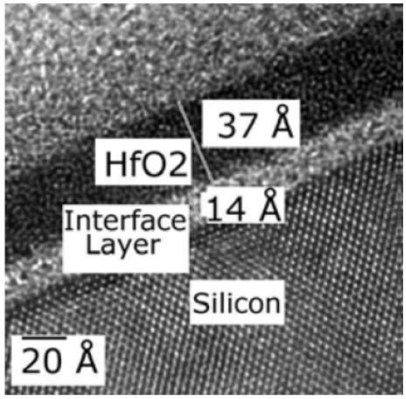


Transmission electron microscopy (TEM)

- Another common electron microscopy technique is called transmission electron microscopy (TEM)
- In TEM, an electron beam is transmitted through a very thin specimen (in case of metals usually some tens of nm) and the image is formed from the transmitted electrons
- Modern TEMs can achieve very high resolution (up to less than 1 Å)
- TEMs are quite expensive compared to SEMs
- The specimen preparation for TEM is way more complicated than for SEM and the studied areas are smaller in TEM





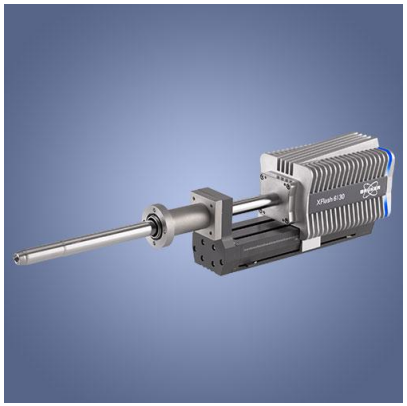


Summary

- SEM provides significantly higher resolution and depth of field than light microscopy
- The resolution of SEM is determined by the interaction volume of electrons in a specimen
- Signals produced in a beam-specimen interaction:
  - Secondary electrons
  - Backscattered electrons
  - Characteristic X-rays
- In addition to imaging using SEs and BSEs, SEM provides a capability of performing elemental (EDS/WDS) and crystallographical (EBSD) analysis
- In general, the major advantage of electron microscopy is that it is a technique which allows imaging, spectroscopy, and diffraction to be performed at the same time with the same machine in real-time

Zeiss Merlin VP Compact FESEM

- Installed at 2014
- Field emission scanning electron microscope equipped with Schottky field emitter
- Acceleration voltage up to 30 kV with a resolution up to 0.8 nm at 30 kV (STEM mode)
- Detectors
  - Secondary electron (SE)
  - In-lens duo (in-lens SE + EsB)
  - 4-Channel-Angular selective backscatter (AsB)
  - Variable pressure SE (VPSE)
- Variable pressure (VP) mode with in the specimen chamber up to 60 Pa
- Nitrogen charge-compensation
- Bruker Quantax EDS (XFlash 6|30) and EBSD (e-Flash HD) system for elemental analysis and crystallographic characterization



Zeiss Ultra 55 FESEM

- Installed at 2005
- Field emission scanning electron microscope equipped with Schottky field emitter
- Acceleration voltage up to 30 kV with a resolution up to 0.8 nm at 30 kV (STEM mode)
- Detectors
  - Secondary electron detector (SE)
  - In-lens SE detector
  - Energy selective backscatter detector (EsB)
  - Backscatter electron detector (QBSD)
- Bruker XFlash EDS detector for elemental analysis
- Oxford HKL Nordlys EBSD detector for crystallographic characterization