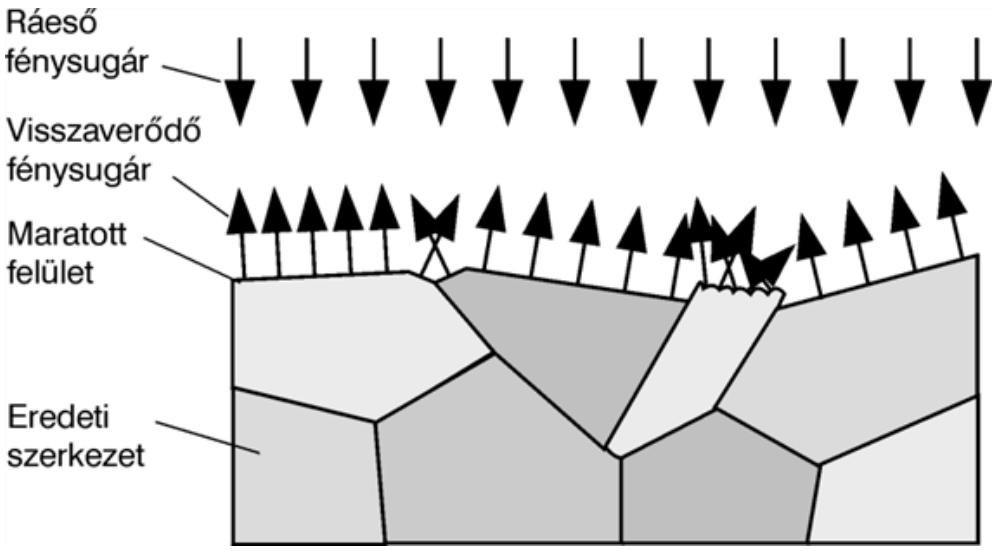
Non-destructive methods for materials testing

Non-classical material survey methods

- Sample preparation for microscopy methods
  - grinding (SiC polishing paper)
  - polishing (aluminum-oxide, diamond, textiles)
  - etching: 21% nitric-acid solution



Microscopy

- Optical microscopy
  - magnification: max. 1500x-2000x
  - resolution: 0.2µm
- Electron microscopy: magnification: 100 000x

$$\lambda = \frac{h}{mv} \qquad \frac{1}{2}mv^2 = eU \qquad \lambda = \sqrt{\frac{1.5}{U}}$$

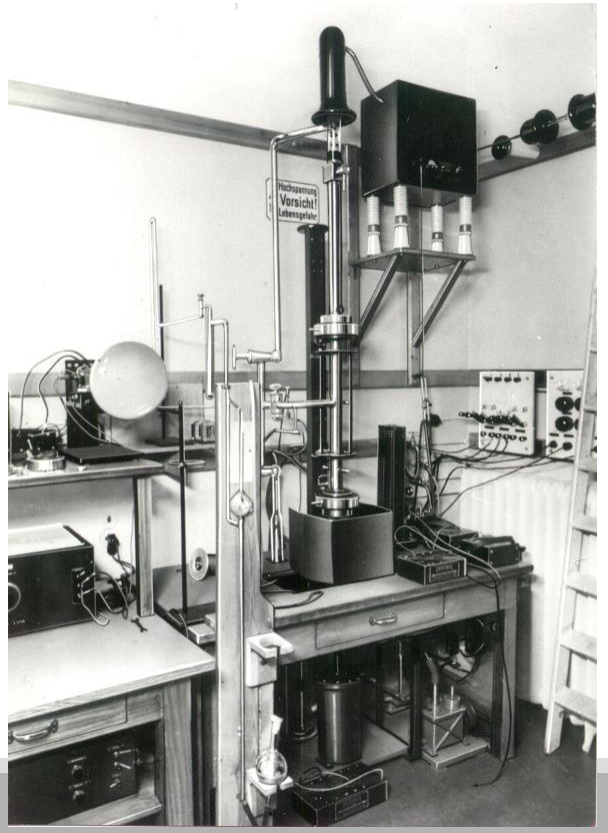
λ=0.01nm (15000V acceleration voltage)

Scanning electron microscopy (SEM)

The method of scanning electron microscopy - SEM

1935 - Max Knoll, german electrical engineer – first image taken with the aid of electrons

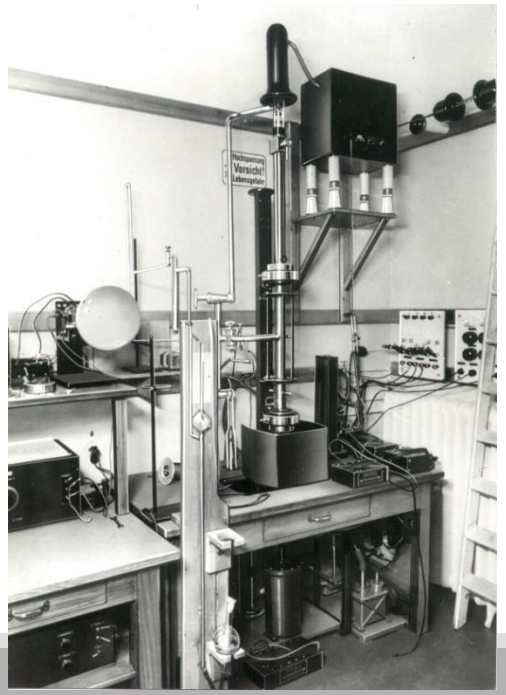
1937 - Manfred von Ardenne, first high resolution scanning electron microscope



The method of scanning electron microscopy - SEM

It is a method used for characterizing the structure of the matter. With the aid of the elecrons the scanned surface is magnified and visualized by **imaging**.

The imaging combined with spectroscopy opens the door to **elemental analysis**.



The method of scanning electron microscopy - SEM

	Optical microscopy	SEM
Wavelength	400 - 800nm	0,010 - 0,012nm
Resolution	200 - 300nm	1nm
Focal depth	several µm	2-3mm
Magnification	1000 – 1500x	10000 - 100000 x

Interaction of electrons with matter

Interaction of electrons with matter

Secondary electrons

Back scattered electrons

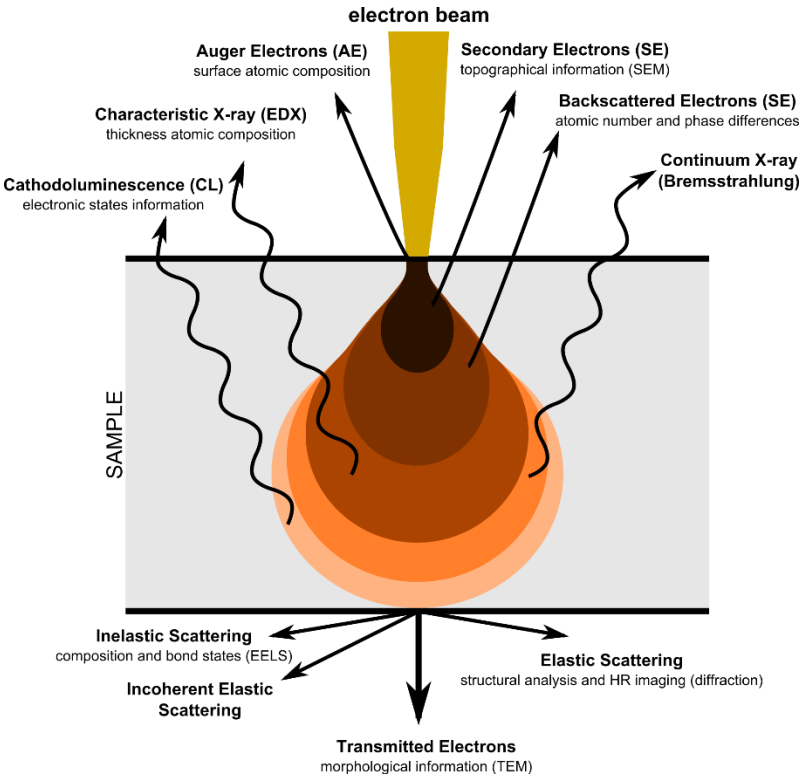
Auger electrons

Characteristic X-rays

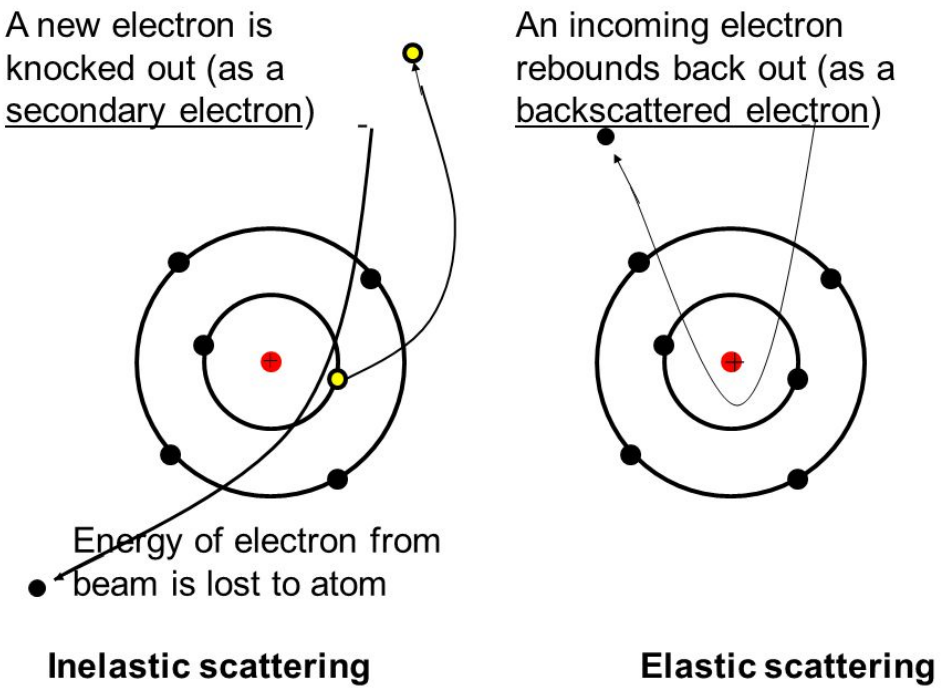
Bremsstrallung

Cathodluminescence light radiation

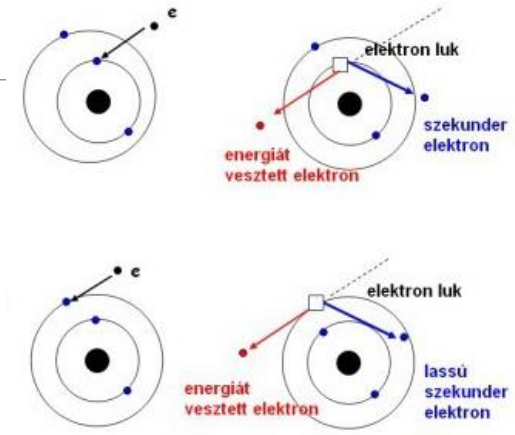
Thermal radiation

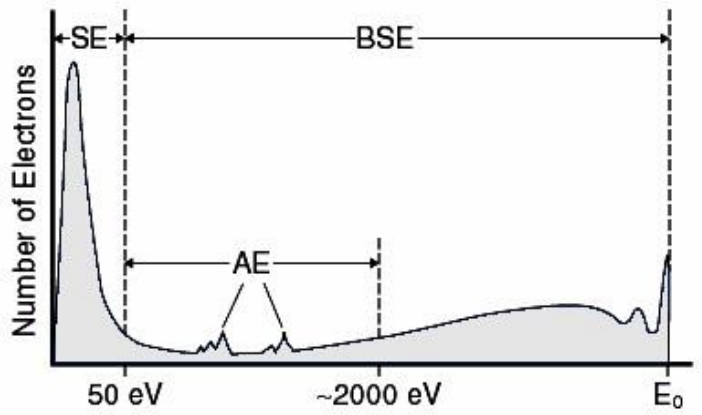


Backscattered and secondary electrons



Secondary electrons (SE)





Secondary electrons: the electrons kicked by the primary electron beam from the inner or outer electron shells

Energy < 50eV → gives an intense peak at the beginning of the spectra

Their low energy makes them **sensitive to the surface roughnesses**

Low energy → **able to get out only from the upper layer of the matter** (1-10nm). Electrons that penetrae more deeply cause an additional electric flow on the sample

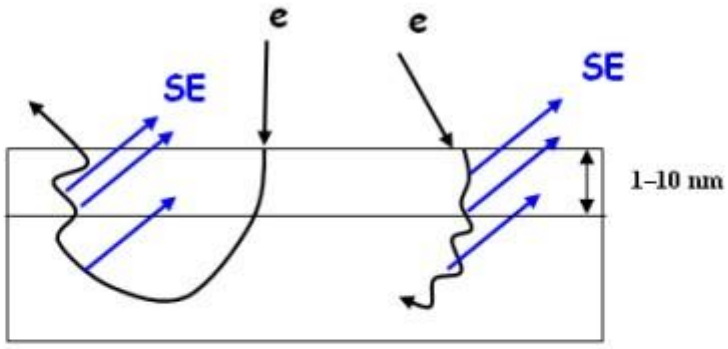
Their average free path in metals is10nm, in insulators 1nm.

Treshold energy in metals is lower → the surface should be coated

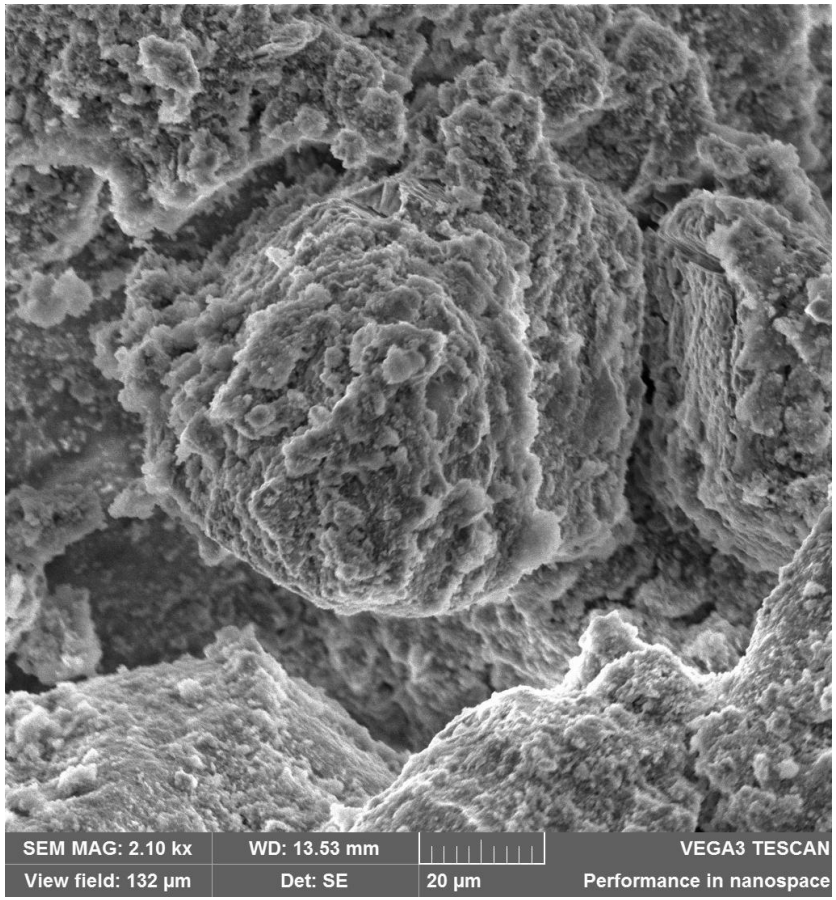
Secondary electrons (SE)

SE intensity is function of:

- **the angle of the surface anf electron beam**
- **electrons energy**
- With the decreasing of the primary electrons energy, decreases the secondary electron quantity that are able to get out from the surface



average free path: 1λ - for metals: 1nm
maximum path: 5 λ - for insulators: 10nm

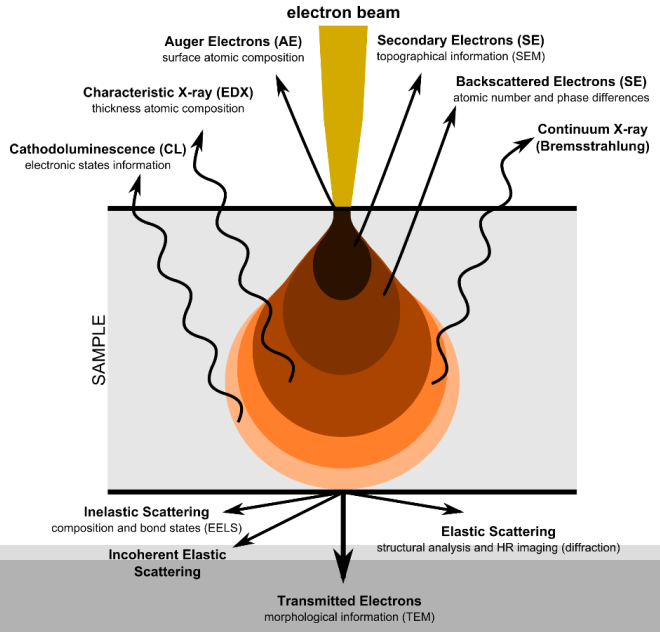

SEM MAG: 2.10 kx
WD: 13.53 mm
VEGA3 TESCAN
View field: 132 µm
Det: SE
20 µm
Performance in nanospace

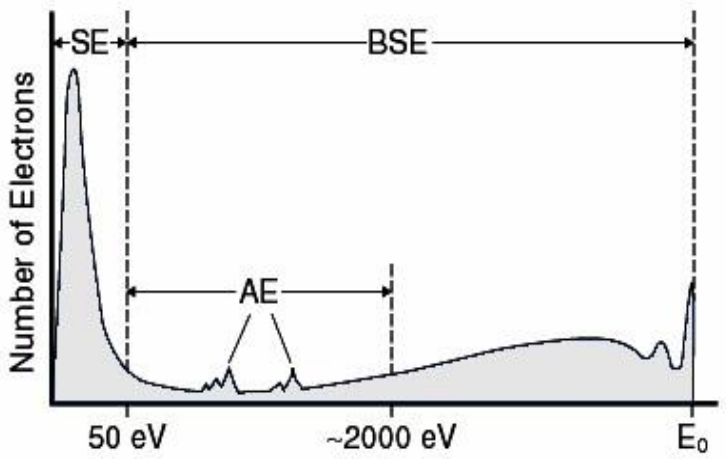
Back-scattered electrons (BSE)

Elastically scattered electrons.

The energy of BSE electrons is higher than that of the SE, therefore they can get out from a bigger volume of the sample. Only low resolution can be achieved.

BSE intensity is function of the atomic number.





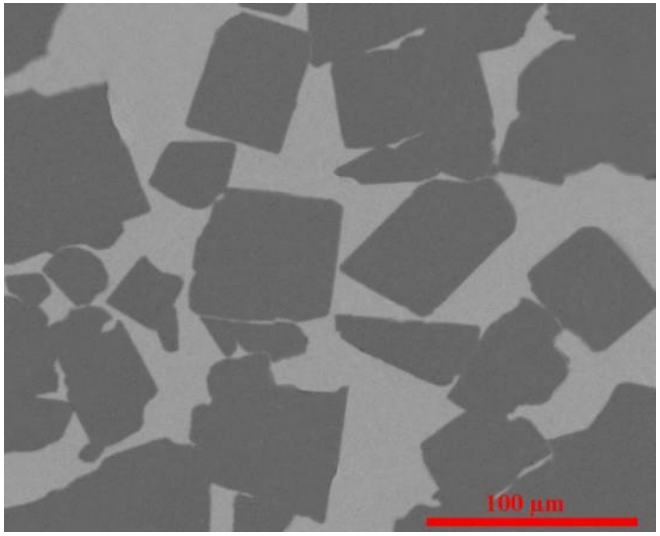
Back-scattered electrons (BSE)

pyrite - chalcopyrite

pyrite (FeS_2): $Z_{averaged} = \frac{Z_{Fe}+2Z_S}{3} = \frac{26+2x16}{3} = 19.33$

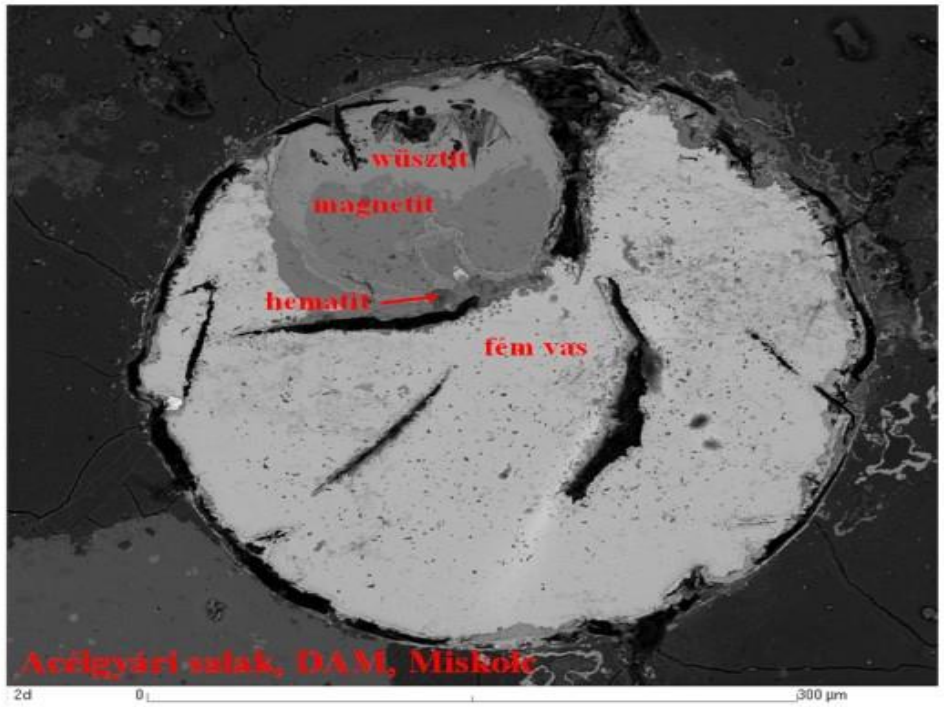
chalcopyrite ($CuFeS_2$):

$$Z_{averaged} = \frac{Z_{Cu} + Z_{Fe} + 2Z_S}{3} = \frac{29 + 26 + 2x16}{3} = 21.75$$



BSE

- Iron-oxides
  - würtzite (ZnS + Fe)
  - magnetite (Fe_3O_4)
  - hematite (Fe_2O_3)
  - metallic iron

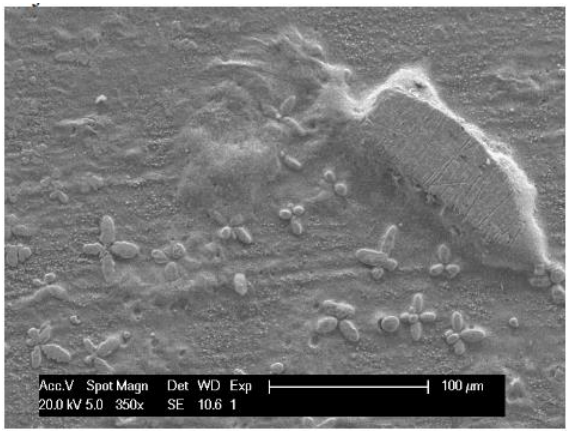


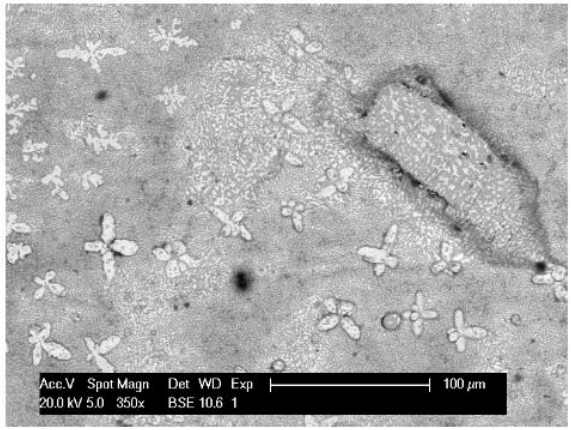
SE and BSE

SE and BSE images taken in the same position, from the same sample

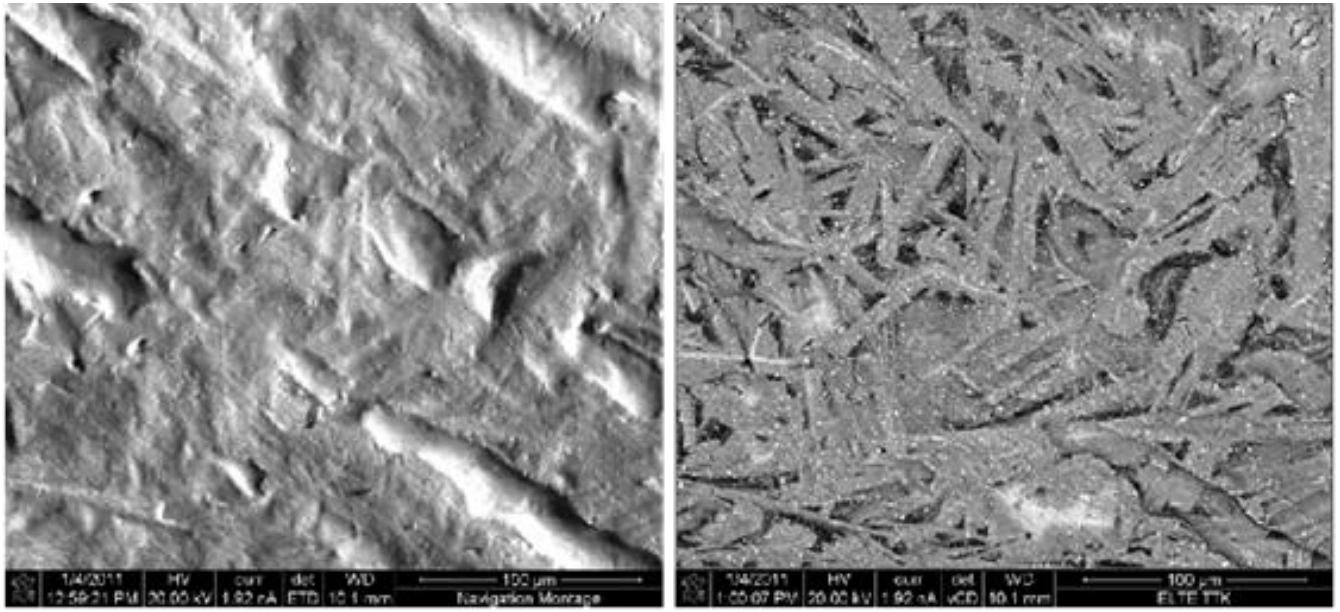
- SE: surface
- BSE: composition, smaller and bigger atomic numbers give lighter and darker iimages on the white-grey-black scale (smaller Z darker, bigger Z lighter) (tin: Z=50 and lead: Z=82).

Tin-lead welding SE and BSE images





SE AND BSE



(a) (b)

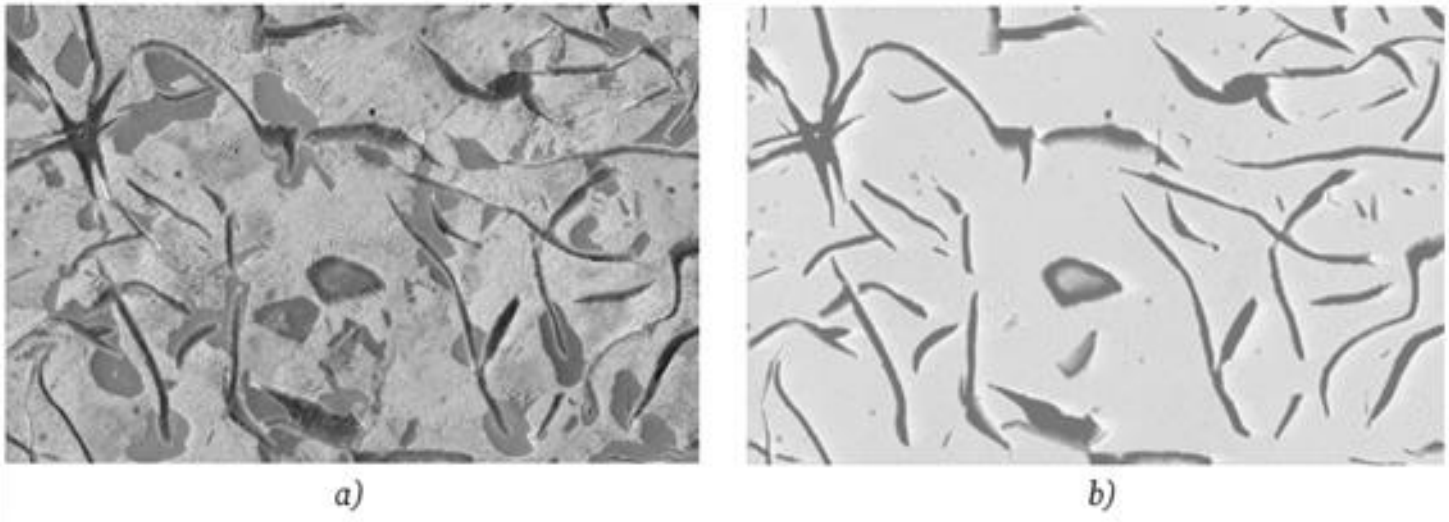
MgCuNiY alloy a) SE, b) BSE images

SE AND BSE

- Cast iron (500X)

a) SE

b) BSE



X-ray radiation

Secondary electrons

Back scattered electrons

Auger electrons

Characteristic X-rays ←

Bremsstrallung ←

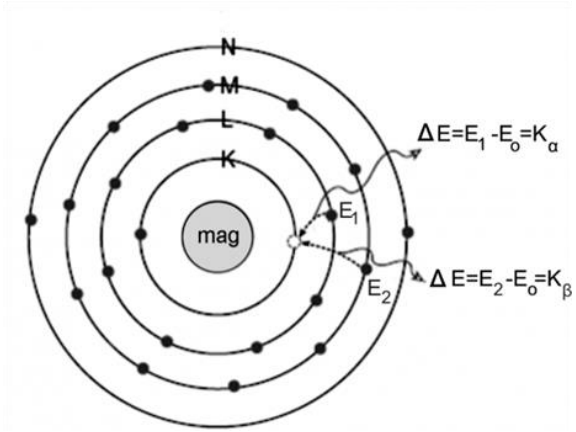
Cathodluminescence light radiation

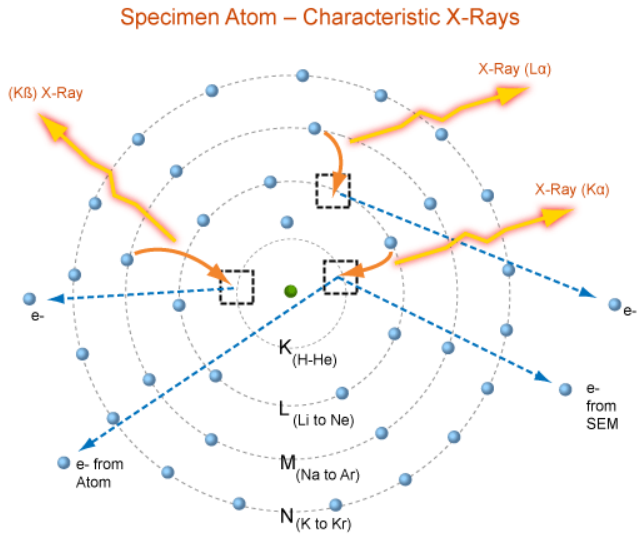
Thermal radiation

Production of characteristic x-rays

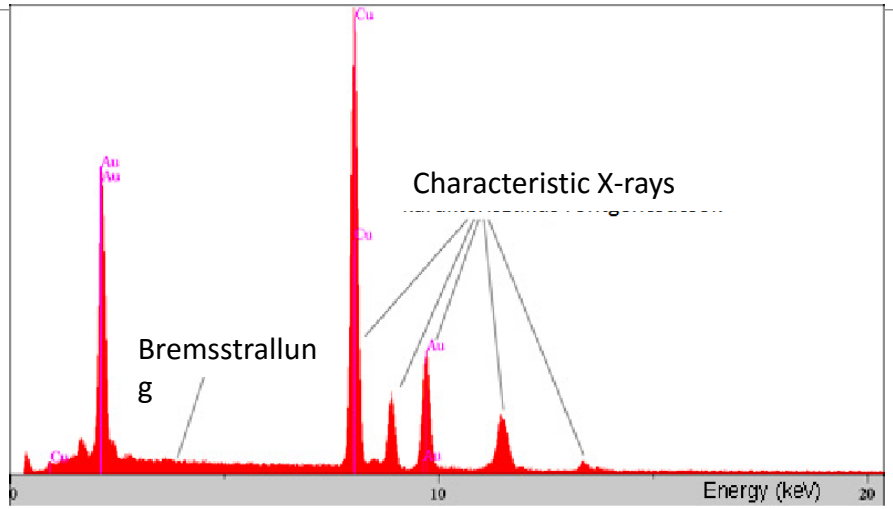
The production of SE is followed by characteristic X-ray production

It is always followed by the so called bremstrallung radiation, which is a continuous radiaton.





Production of characteristic x-rays



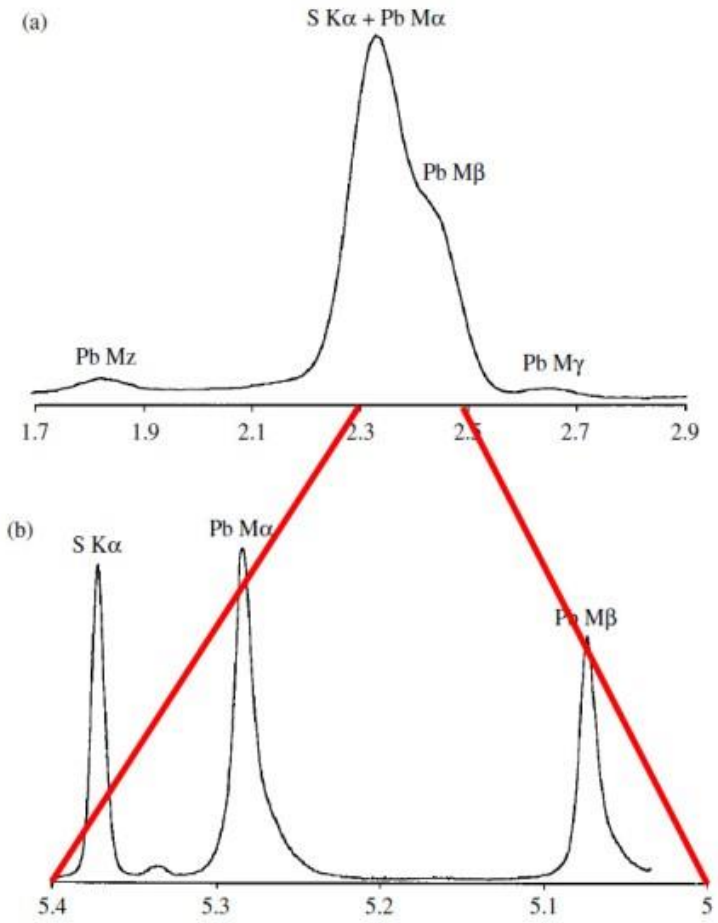
The electrons kicked out from the inner shells produce ionization.

The minimum energy that is needed for the ionization is called ionization potential **(E_k)**.

The grade of ionization is controlled by the high voltage/ionization potential rate.

SEM

- EDS – energy dispersive spectroscopy
- WDS – wavelength dispersive spectroscopy

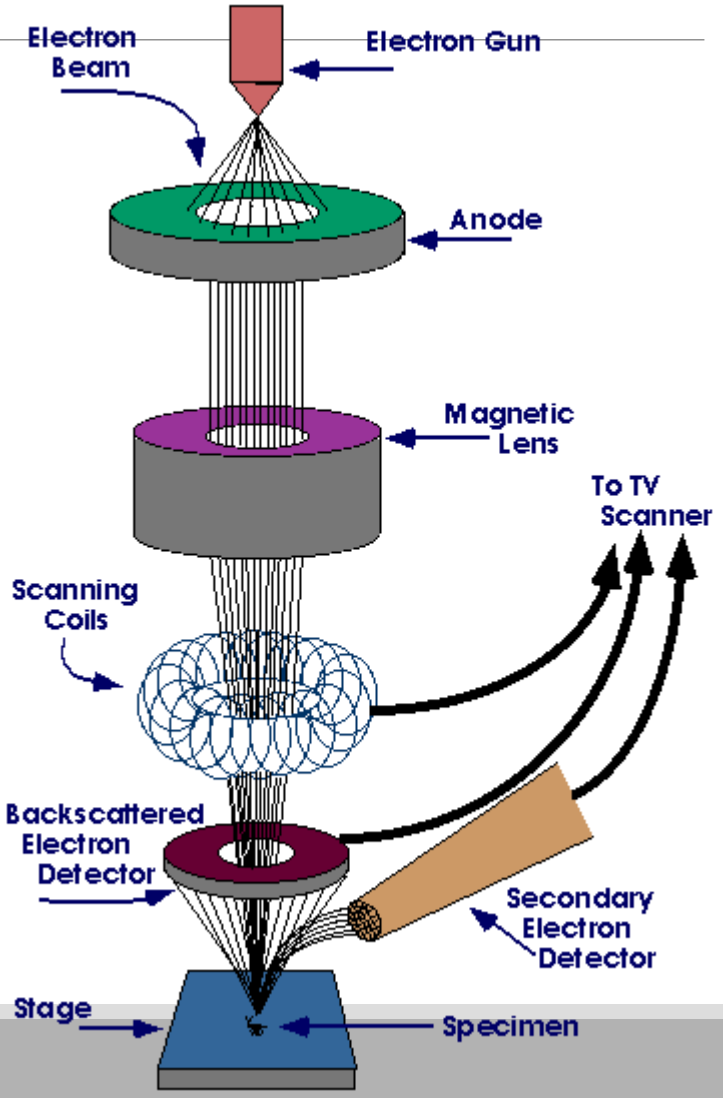


Functional parts of the microscope

SEM FUNCTIONAL PARTS

- Electron column (electron source, slits and lenses)
  - **produces the electron beam**
  - determines the electron intensity
  - forms the beam (orientation, diameter)
- Scanning coils
- Detectors
- Sample chamber
- Electron optics and light optics analogy.

2 types:
- only imaging
- for elemental analysis



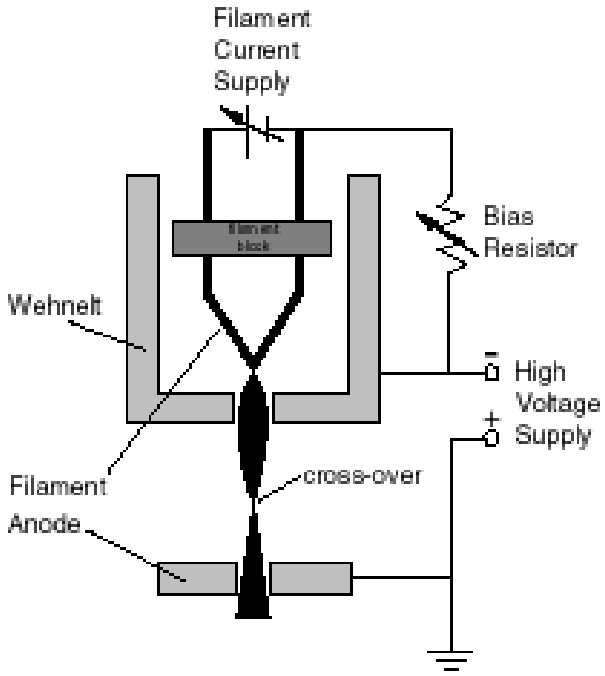
Electron gun with a tungsten filament cathode

Three electrodes:

- tungsten cathode
- anode
- Wehnelt cylinder

The electrons stepping out from the cathode will be accelerated towards the anode

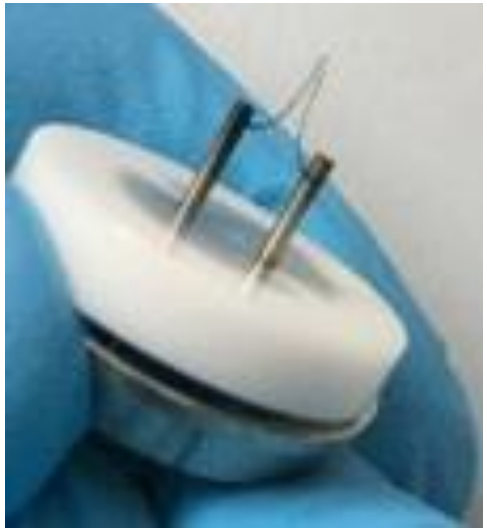
The Whenelt cylinder focuses the electrons in one point. This will be the electron source.



Electron gun with a tungsten filament cathode

W-cathode: V form, thin (0.1mm) tungsten vire, from its top will the electrons step out

The cathode is heated up to 2700K. At this temperature the energy of the electrons is 0.23eV.



MAGNETIC LENSES

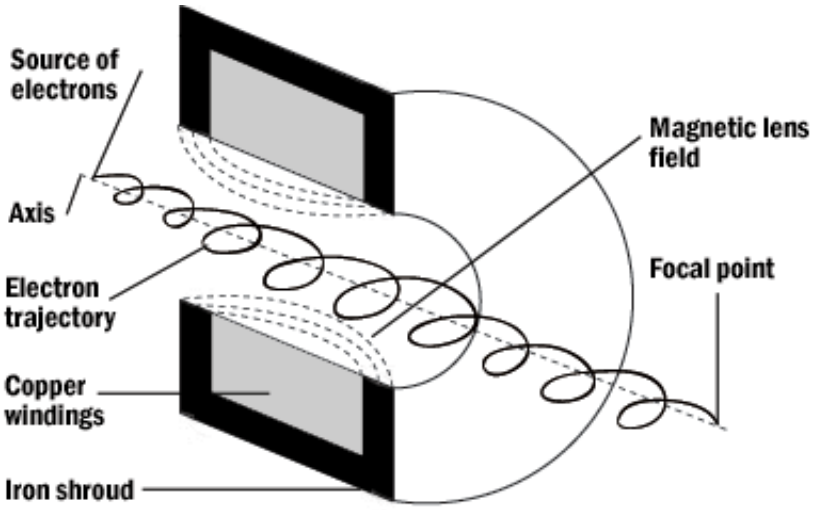
Used for focusing the divergent beam.

They work similarly to the optical lenses.

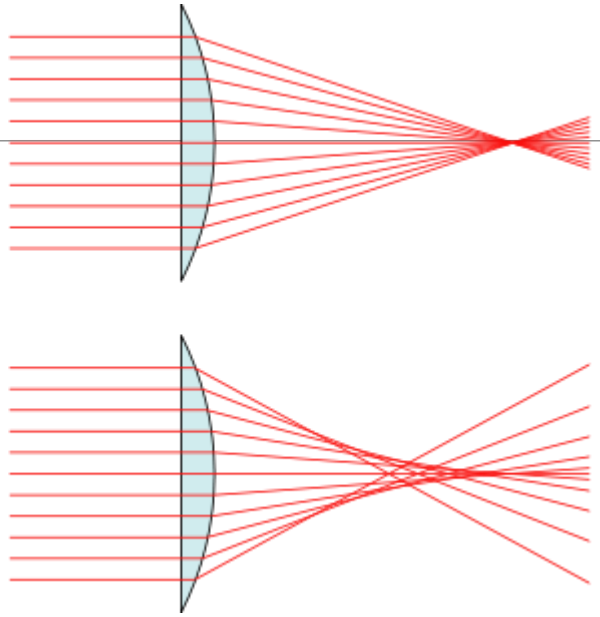
They are made out of two copper rings with circular symmetry with an iron shroud.

The F force is acting upon the electron. F will be proportional to the vectorial product of the velocity of the electron (v) and the magnetic field vector (B):

$$\vec{F} = -e(\vec{v} \times \vec{B})$$



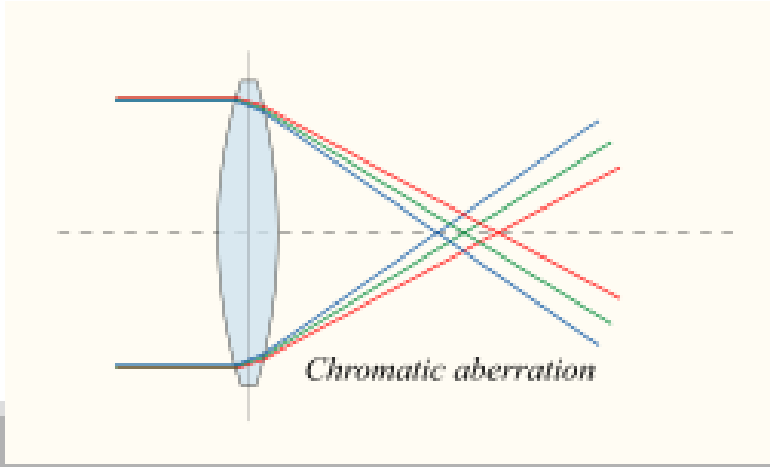
Optical aberrations



Spherical aberrations: if the magnetic field is inhomogeneous, the focal path will be shorter for along the axis then at the sides

Chromatic aberrations: caused by the energy dispersion of the electrons. The focal point for different wavelengths will be at different places.

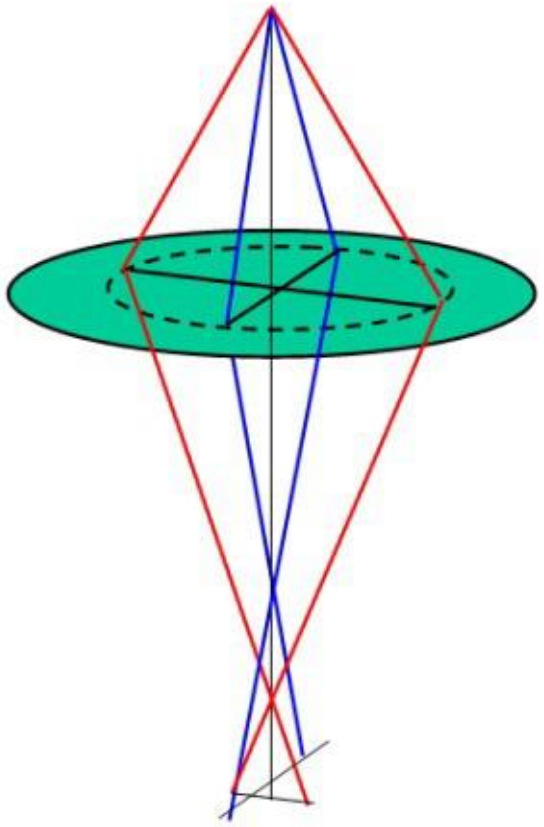
Cannot be corrected.



Optical aberrations

Astigmatism: imperfection of the lenses: the focusing is orientation dependent, the image of a point will give an ellipse.

Can be corrected with the aid of potentiometers.



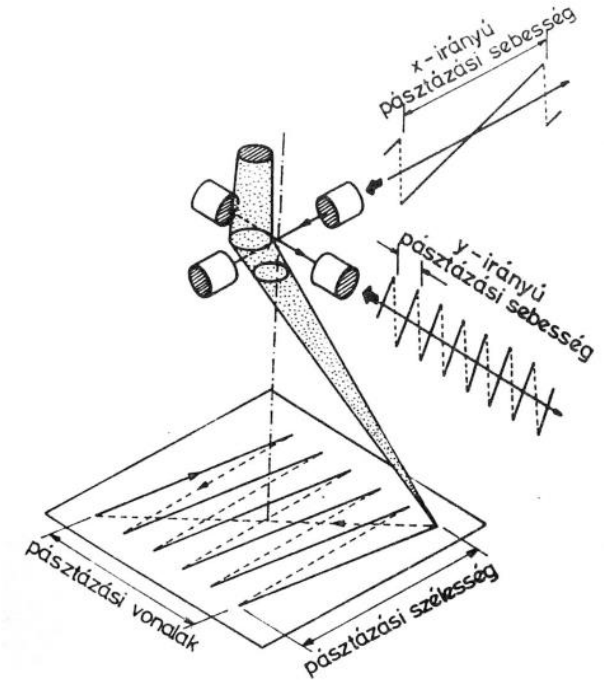
Scanning unit

Deflects the electron beam in two directions, making possible the scan over the surface being magnified.

Each point of the sample has its point-like image on the screen. The relation between the scanned surface and the screen will give the magnification N.

$$N = \frac{l_{screen}}{l_{scanned\ surface}}$$

With the decreasing or increasing of the scanned territory, the magnification can also be increased or decreased.



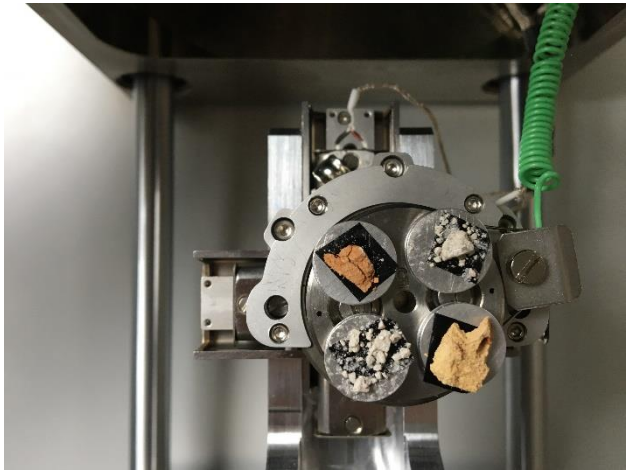
Sample chamber

Sample table with several sample positions.

Need of a conducting tape for fixing the sample.

Vacuum inside the chamber.

Some SEM types with low vacuum system, used for BSE detectors.



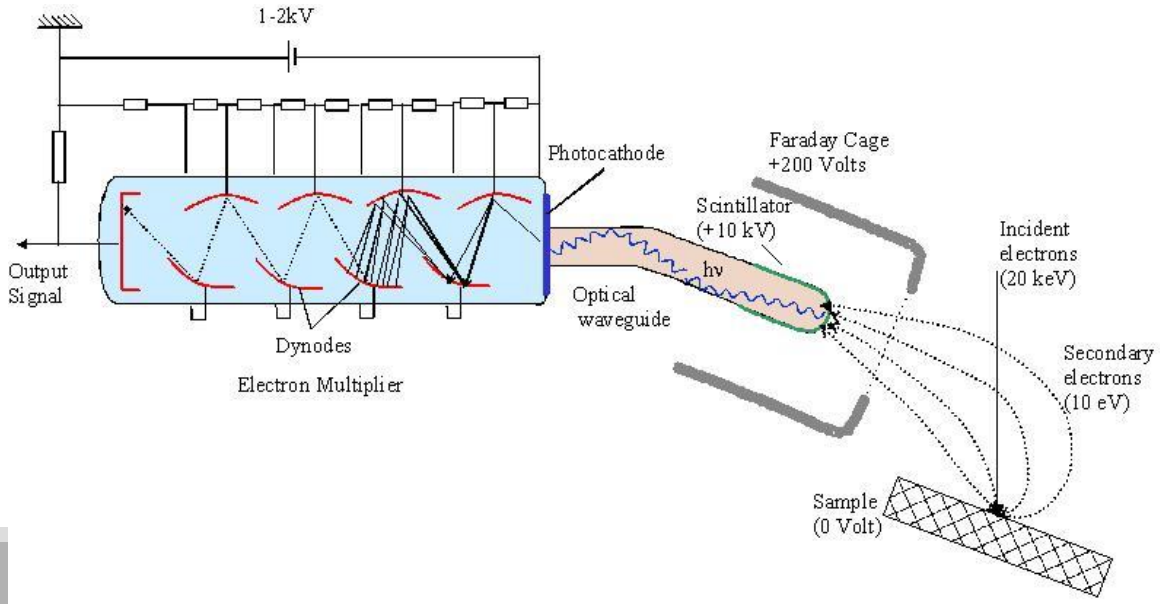
Detection of electrons

Setection of SE electrons

„Everhart-Thornley” detector: scintillator screen with a photomultiplayer

Faraday-cage: metallic plate, with +300V potential. Small energy SE electrons are collected from all directions.

Scintillator: ring plastic disc, coated with phosphorous. The electrons cause production of visible light in the phosphorous (400nm wavelength). They are converted with a photocathode and electron multiplyer to electric signal.



Sample preparation

If the surface of the sample is not conductor, the surface has to be coated with a conducting metallic layer. Smaller magnifications can be achieved also without coating.

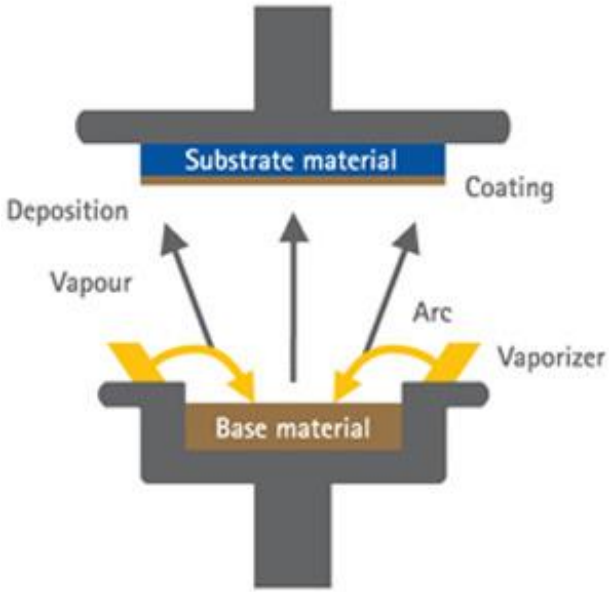
Vapour deposition

- decreases the work needed for getting out of the electrons
- conducts the electrons from accumulated on the surface

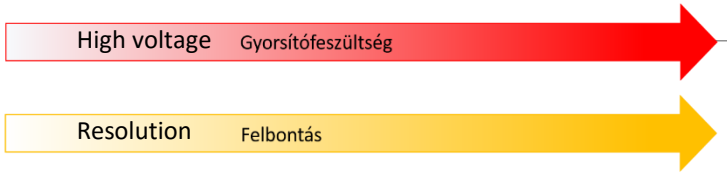
Conducting layer:

- graphite (small electron absorption) – chemical analysis
- gold, chromium, iridium – topological images

For BSE images the surface has to be polished. (for example with aluminium-oxide paste or diamond powder)

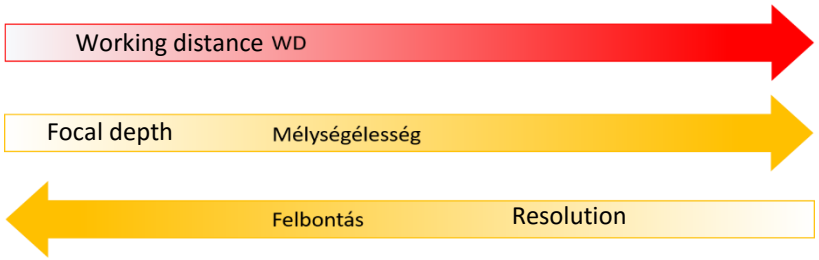


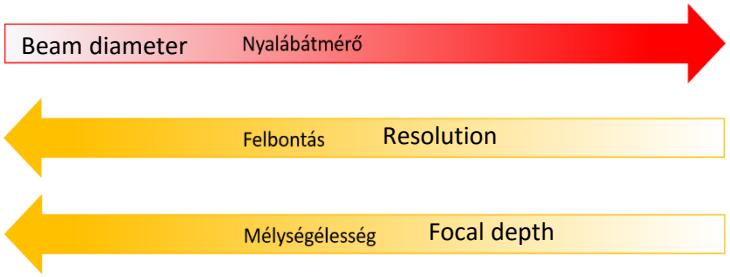
Measurements



Clear surface structure
Smaller damage of the sample
Smaller e accumulation
Smaller edge effect

Obscure surface structure
Higher damage of the sample
Higher e accumulation
Higher edge effect





Grainy image

High beam intensity

Examples

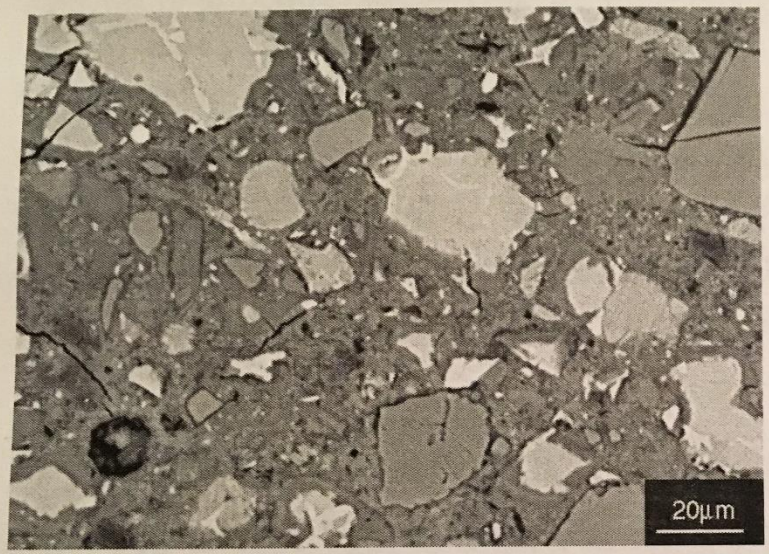


Figure 1.3 Example of backscattered electron image of a polished section of concrete showing: unhydrated cement grains (bright); silica sand (grey) and cement hydration product (grey matrix).

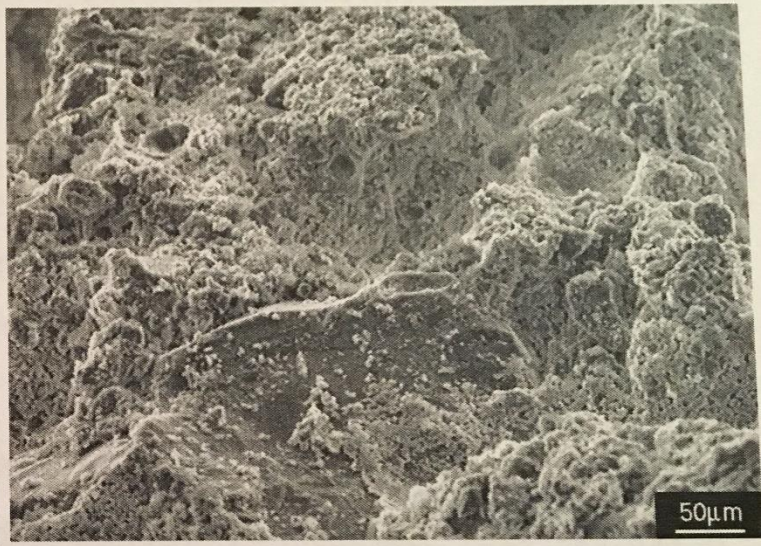


Figure 1.4 Example of secondary electron image of a fracture surface of mortar: the rough surface is cement hydration product and the smoother particle at lower centre is a sand particle.

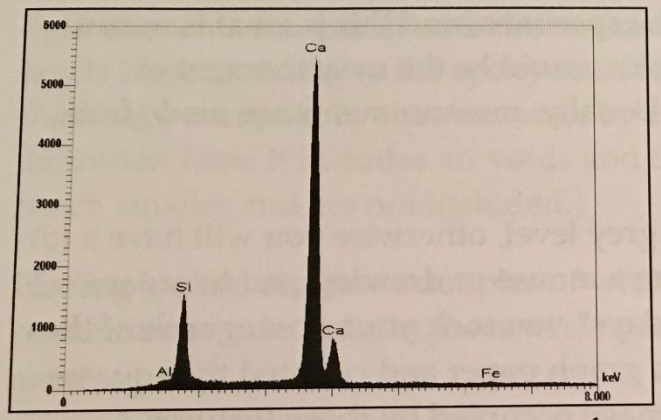


Figure 1.7 Example of X-ray spectrum of alite, showing strong peaks due to calcium and silicon. Expanding the vertical scale would show weak peaks due to iron, aluminium and magnesium.

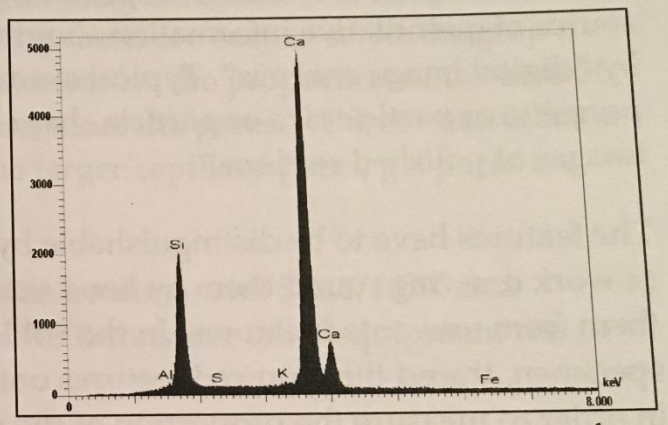
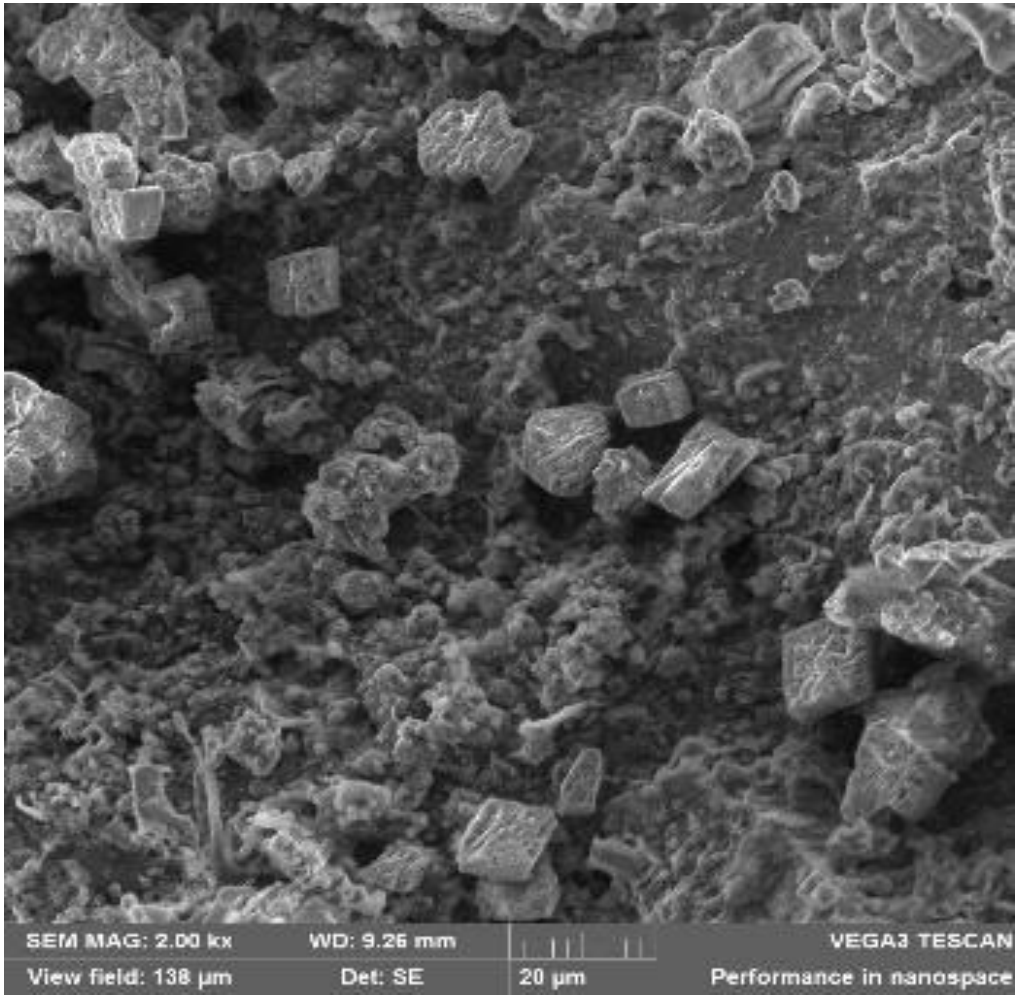


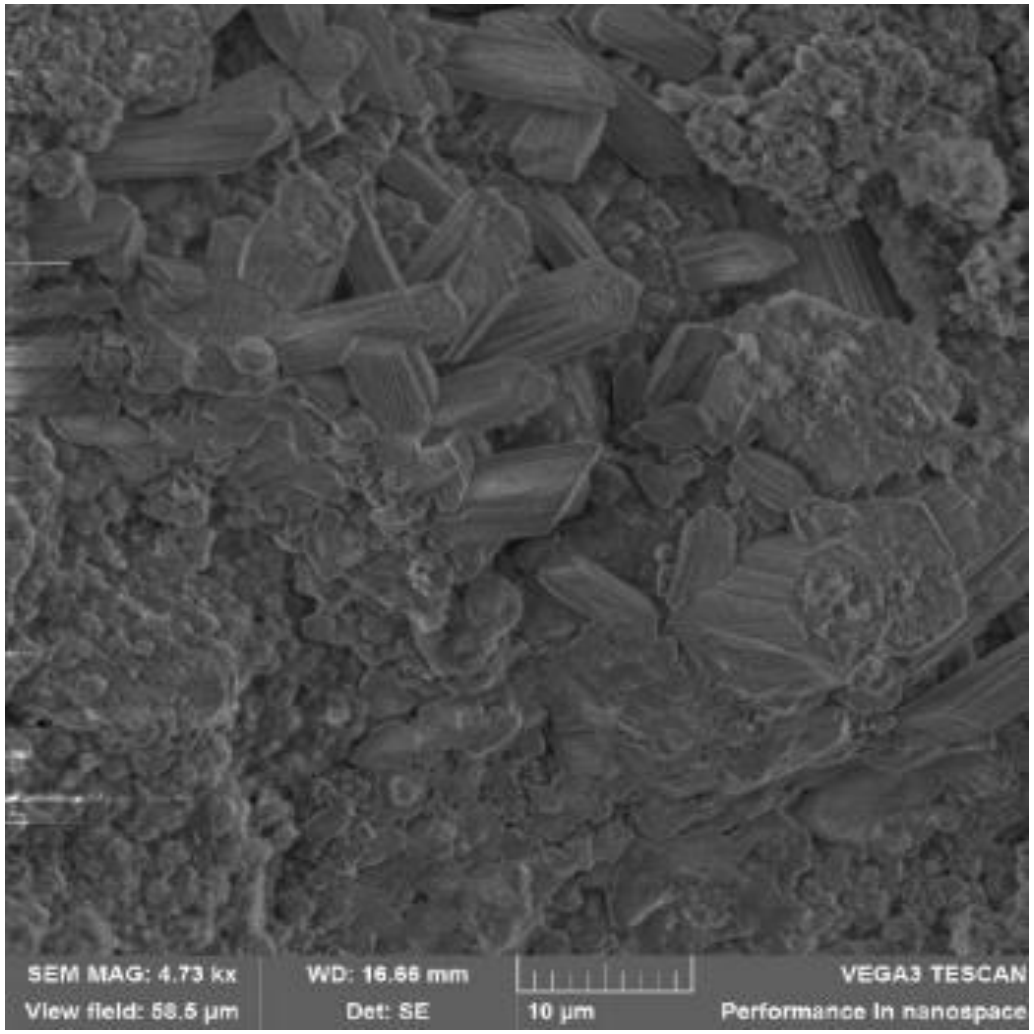
Figure 1.8 Example of X-ray spectrum of belite, showing strong peaks due to calcium and silicon, with weak peaks due to iron, sulfur, aluminium and potassium also visible, but better seen on an expanded vertical scale (not shown here).

Table 1.1 Quantitative analyses (as oxide weight percent.) of the alite and belite spectra in Figures 1.7 and 1.8.

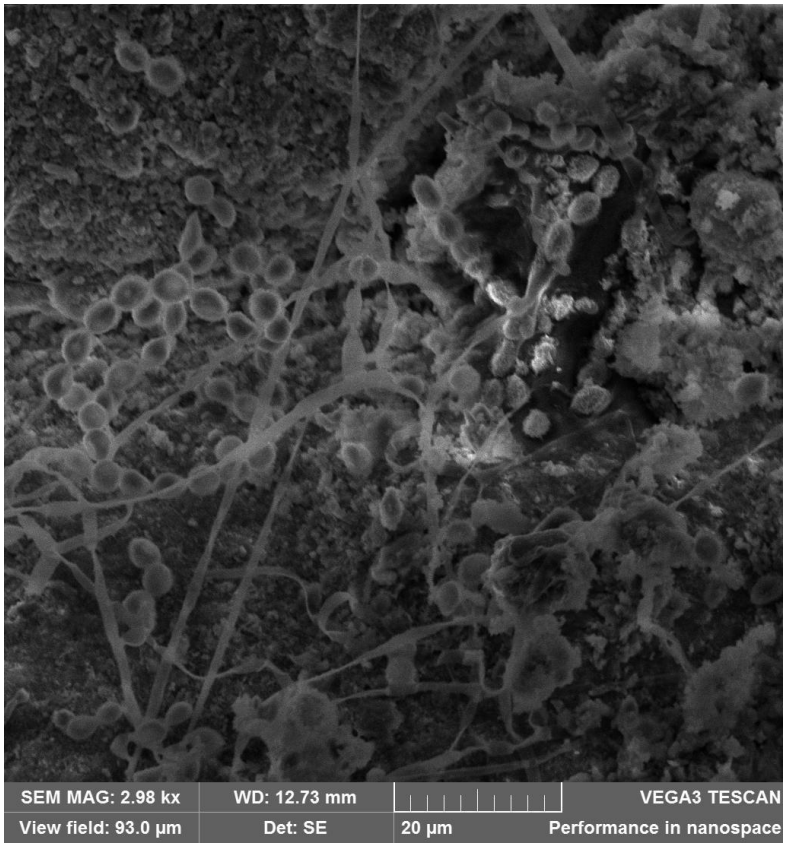
	Na_2O	MgO	Al_2O_3	SiO_2	SO_3	K_2O	CaO	TiO_2	Mn_2O_3	Fe_2O_3
Alite	0.1	0.3	1.3	24.8	0.1	0.1	72.6	0.0	0.0	0.7
Belite	0.1	0.1	0.7	33.6	0.2	0.5	64.4	0.1	0.0	0.3

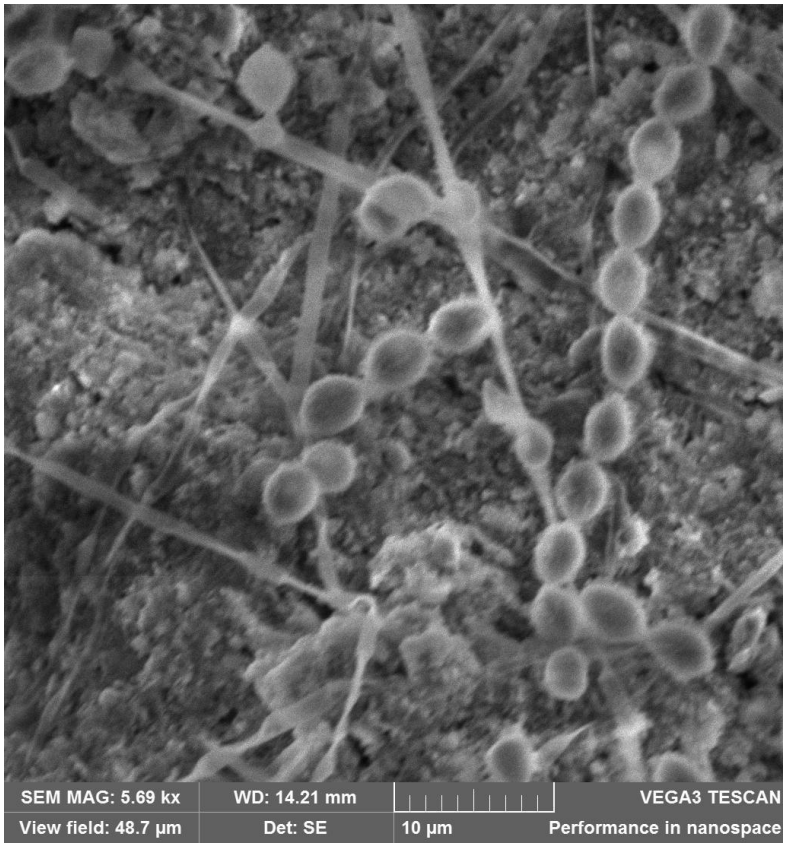


Cement mortar – 28% NaCl solution

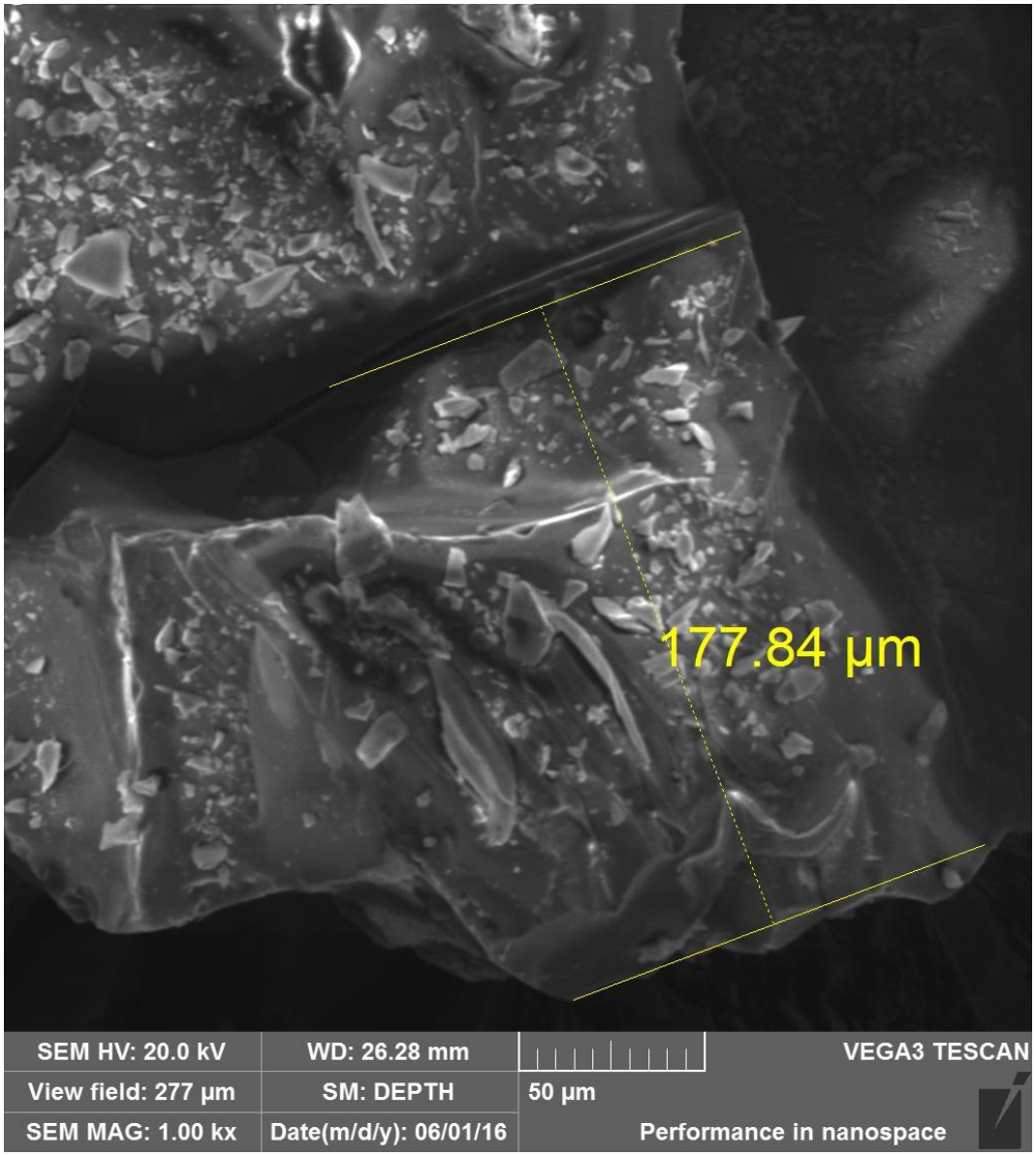


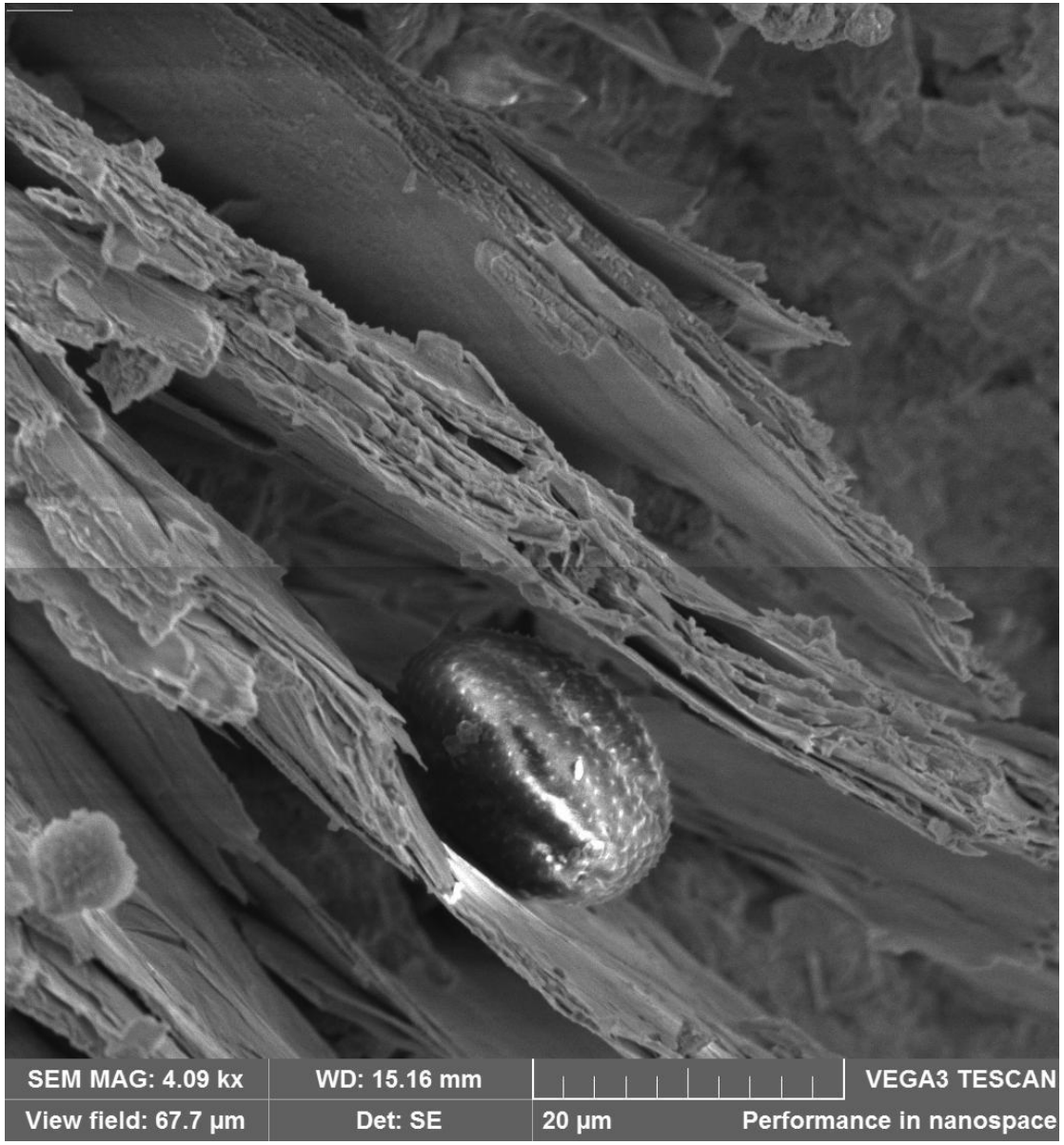
Cement mortar – needle like salt crystals

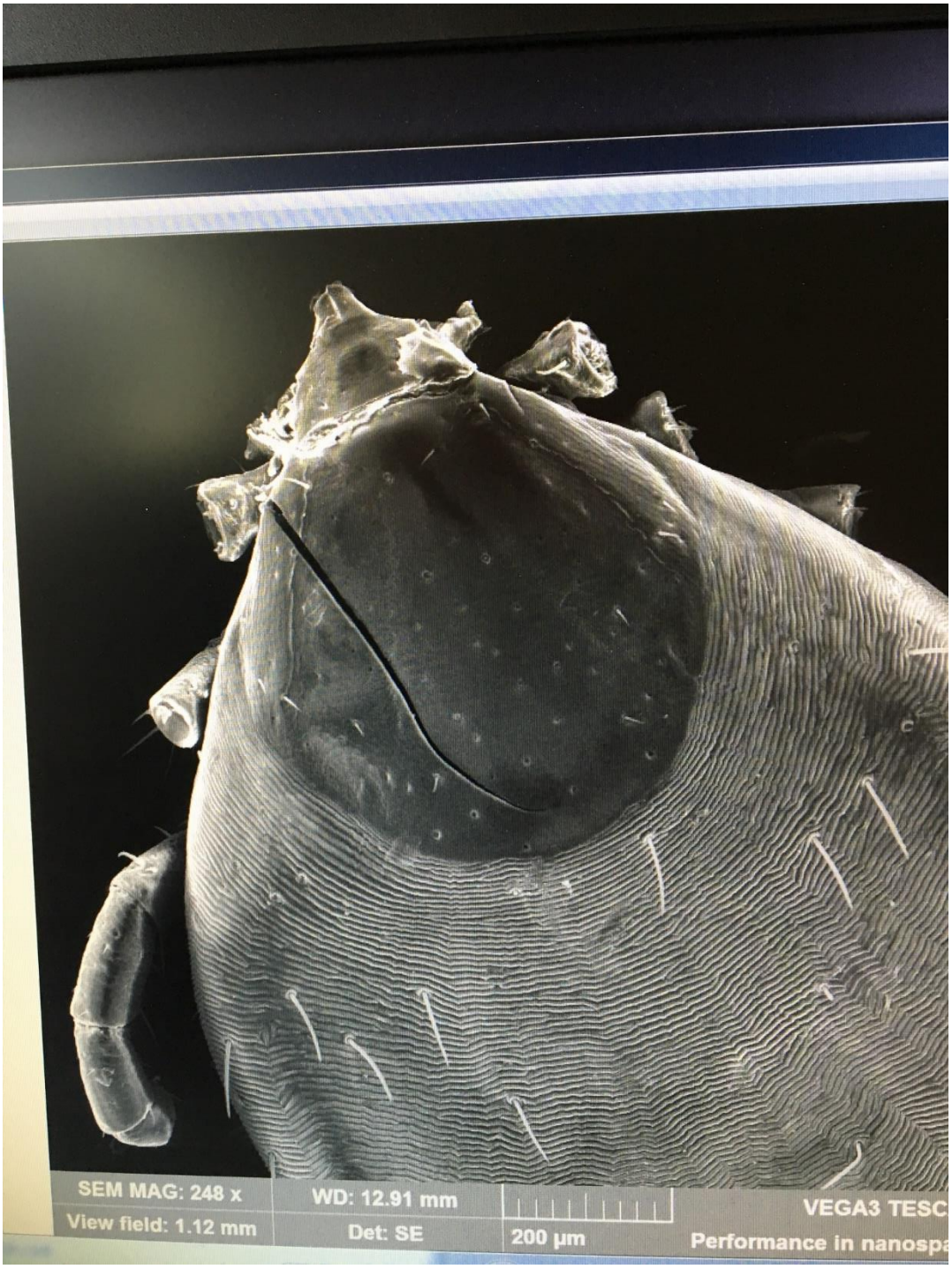




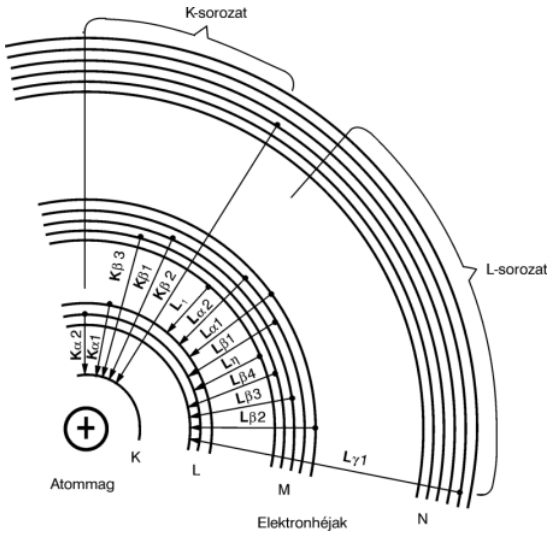
Cement mortar - bacteria

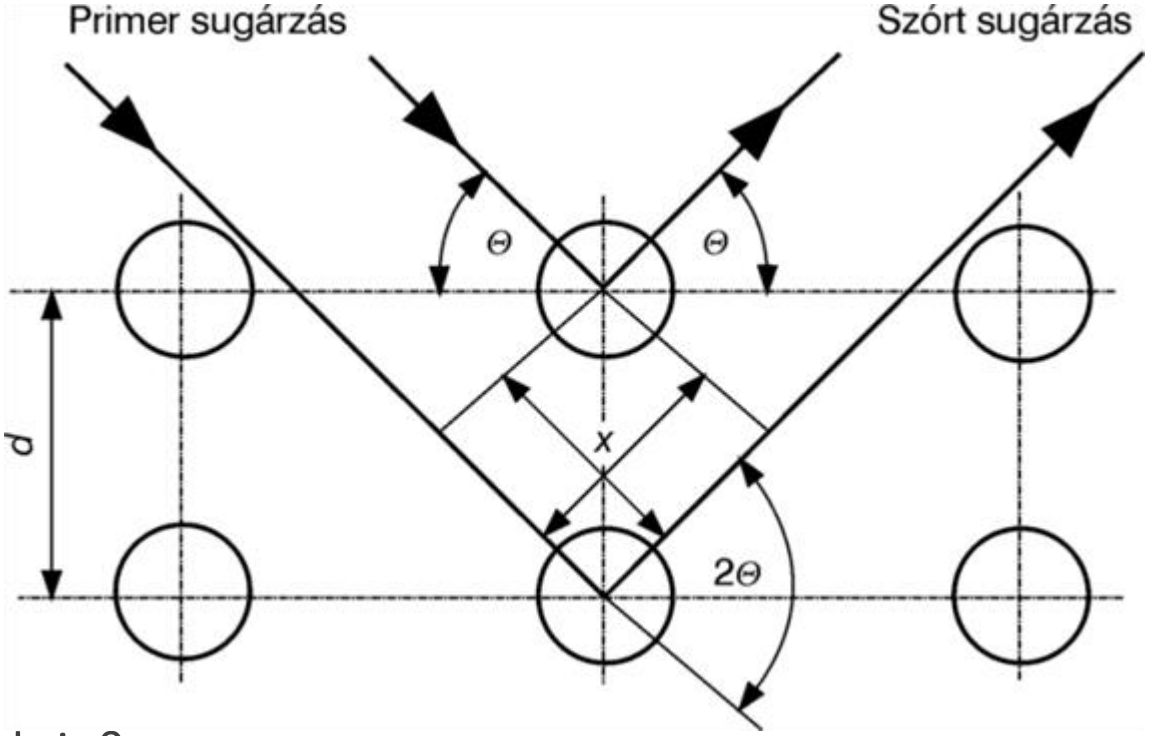

177.84 µm
SEM HV: 20.0 kV
WD: 26.28 mm
VEGA3 TESCAN
View field: 277 µm
SM: DEPTH
50 µm
SEM MAG: 1.00 kx
Date(m/d/y): 06/01/16
Performance in nanospace


SEM MAG: 4.09 kx
WD: 15.16 mm
VEGA3 TESCAN
View field: 67.7 µm
Det: SE
20 µm
Performance in nanospace


SEM MAG: 248 x
View field: 1.12 mm
WD: 12.91 mm
Det: SE
200 µm
VEGA3 TESC
Performance in nanospa

X-ray diffraction



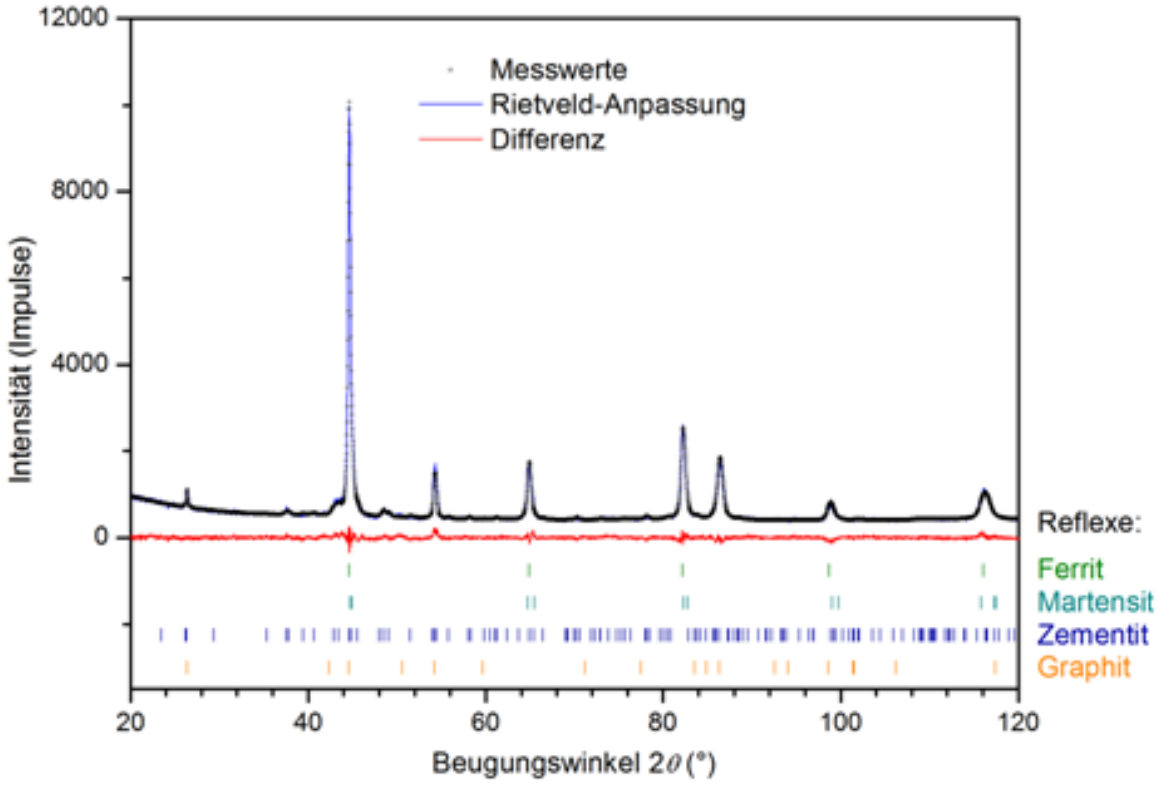


Bragg equation: $\lambda = n\, 2d \sin\theta$

X-ray diffraction

- Reveals:
  - dislocation density
  - changes in the lattice parametres
  - anisotropy
  - presence of different phases and, qualitative identification of them
  - chrystalline phases quantitative determination
  - residual stress determination

X-ray diffraction



Other methods

- TEM – transmission electron microscopy
- Radiography (X-rays, neutrons)
- Pigment penetration tests
- Elemental analysis tests
- ^{60}Co radioisotope tests (radiation transmission)
- Acustic emission tests

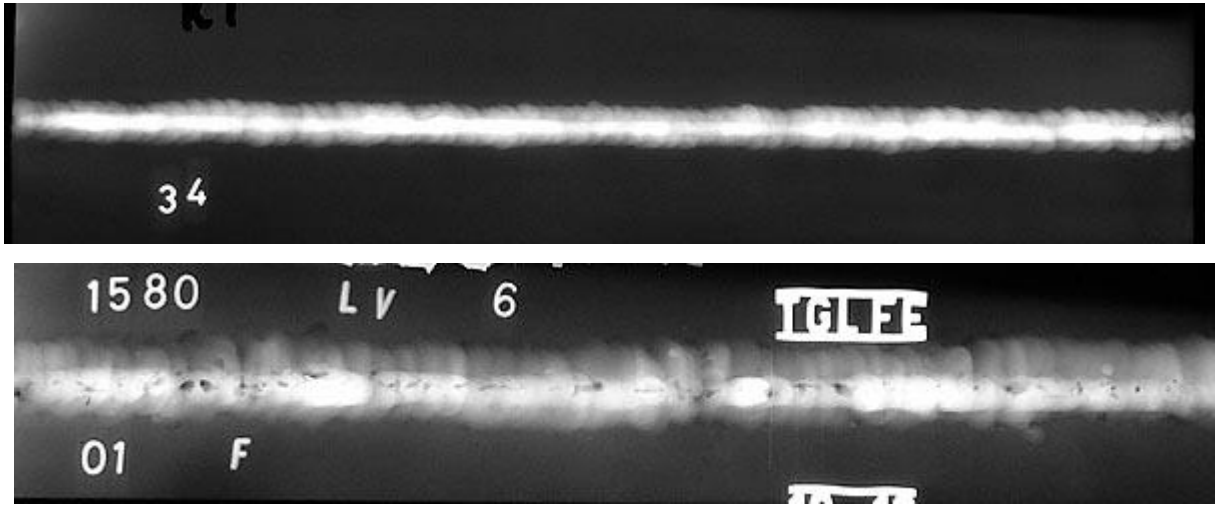
etc.

Other methods

X-ray radiography

of a perfect an of

a damaged welding



Cracks on a steel surface – paint penetration test