

Scanning Electron Microscopy (SEM)

Dr. Günter Bertsche
C10A29
Tel: 29 76351

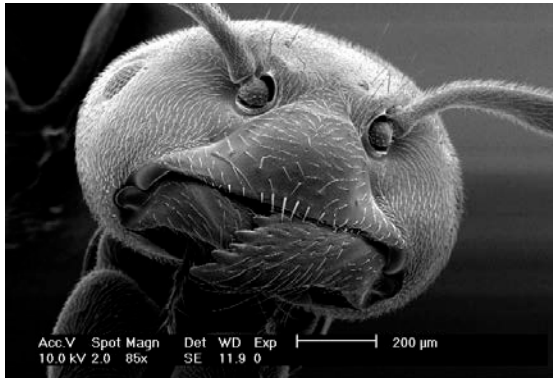
The following items serve as a guide to prepare for the experiment:

- ❖ Principle of the scanning electron microscope
 - Electron gun, electron optics, electron beam deflection, Everhart-Thornley-Detector
- ❖ Effects on the sample by bombardment with primary electrons (PE)
 - Electron-Specimen Interactions, Electron diffusion cloud (interaction volumes), interaction products, energy spectrum of emitted electrons
- ❖ Properties of the secondary electrons (SE)
 - Energy Distribution
 - Angular Distribution
 - Secondary Electron Yield δ
 - Dependence on the PE energy, on the specimen material (atomic number) and on the angle of incidence
 - Contribution of the Backscattered Electrons to the Secondary Electron signal
- ❖ Properties of the Backscattered Electrons (BSE)
 - Energy Distribution
 - Angular Distribution
 - Backscattering Coefficient η
 - Dependence on the PE energy, on the specimen material (atomic number) and on the angle of incidence
- ❖ Imaging with Secondary and Backscattered Electrons / Modes of Contrast in SEM
 - Topographic Contrast
 - Contrast due to different tilt angles of surface elements
 - Shadow Contrast
 - Contrast on edges / rough surfaces
 - Material Contrast
 - Voltage Contrast
 - Magnetic Contrast
 - Cristal Orientation Contrast
- ❖ Resolution of the SEM
 - Limit of Resolution and Electron Probe Size due to Lens Aberrations
- ❖ Depth of Focus
- ❖ Requirements to be fulfilled by the sample
- ❖ Elemental Analysis with X-Rays in SEM (EDX)

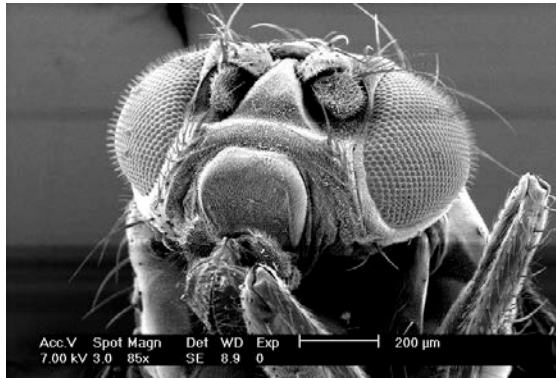
The individual topics are discussed briefly in the following, with presentation being limited only to the essentials. For further study reference is made to the literature (see last page).

Introduction

The SEM is an important device for surface structure analysis of solid samples, which is widely used in research and development in areas such as semiconductor physics, nanotechnology and biology. In this microscopy method a very finely focused electron beam is scanned line by line over the object surface. The interaction products thus generated by the primary electrons in the object (secondary electrons, X-rays, etc.) are collected and used for image display. Using the SEM, structures can be studied with a lateral resolution of only a few nanometers and with a depth of focus 1000 times larger compared to an optical light microscope.

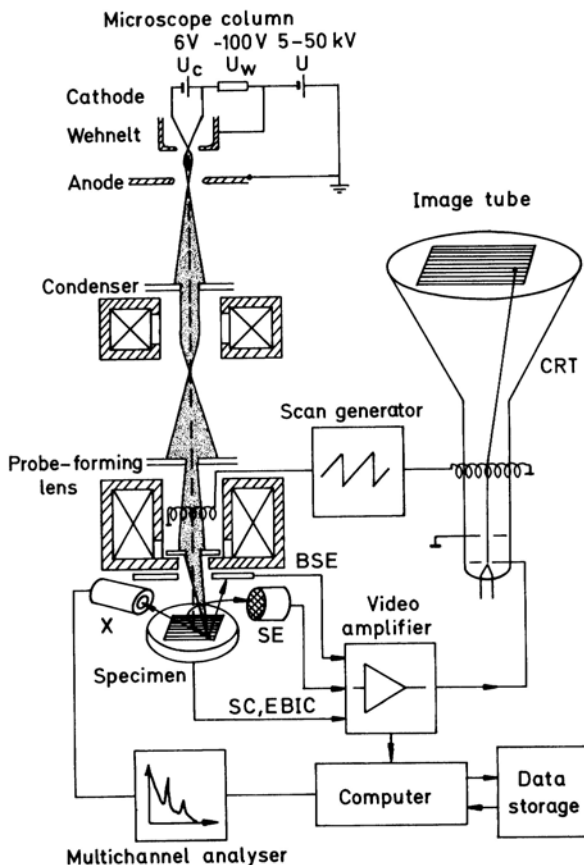


SEM image of an ant



SEM image of a drosophila fly

1. Principle structure of the Scanning Electron Microscope



The SEM consists of the following components, which are described individually in more detail below:

- **Electron Gun**
(e.g. thermionic electron gun: cathode, anode, Wehnelt cup)
- **Electron Lenses:**
Condenser lens, final probe forming objective lens for the demagnified imaging of the Crossover onto the samples surface.
- **Electron beam deflection:**
 - for electron beam adjustment and blanking.
 - for scanning the electron probe line by line across the sample.
 - Stigmator for compensating the astigmatism.
- **Detectors**
Everhart-Thornley-Detector, Semiconductordetector, etc.
- **Image Recording and Processing**

Figure 1: Schematic buildup and Principle of the SEM

(© L.Reimer: Scanning Electron Microscopy, 2nd ed. 1998, Fig. 1.1, mit freundlicher Genehmigung von Springer Science and Business Media)

1.1 Electron Guns

In principle, free electrons can be produced by thermionic emission, Schottky emission or field emission.

a.) Thermionic Electron Guns

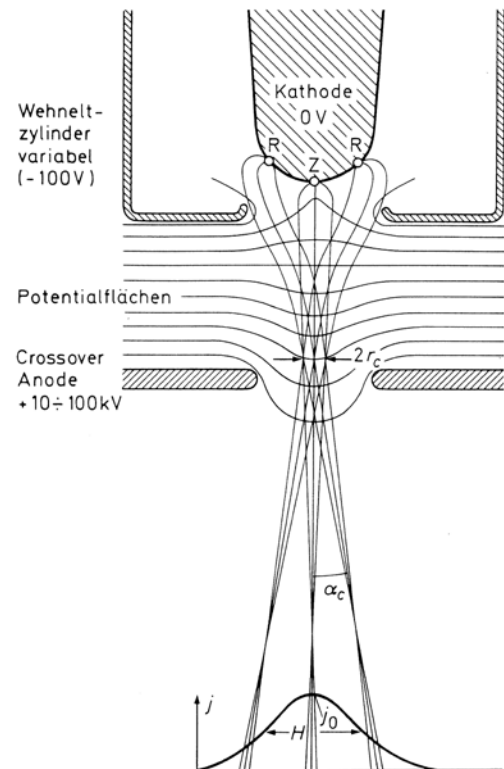
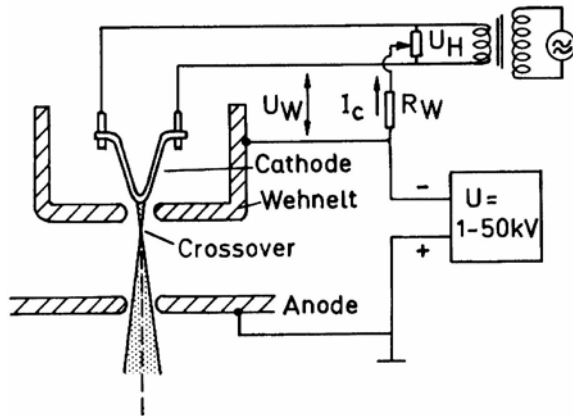


Figure 2: Thermionic electron gun consisting of cathode, Wehnelt cup and anode. The bias of the Wehnelt cup is about 100 V more negative with respect to the cathode. This leads to the formation of a space charge cloud in front of the cathode, whereby a more stable emission current is reached. Furthermore, the potential distribution of the Wehnelt cup causes a concentration/focusing of the electrons to the crossover with a diameter of $2r_c \approx 50 \mu\text{m}$.

b.) Schottky Emission Gun:

Application of an external electric field to the cathode results in a reduction of the work function of about $\Delta\Phi_W$ (see Fig. 3). This decrease of the effective work function is called Schottky effect, it is negligible for thermal cathodes. At a field strength of $E=10^6 \text{ V/cm}$ it is $\Delta\Phi_W \approx 0.4 \text{ eV}$. The cathode is heated, so that the electrons can overcome the reduced work function $\Phi_{W,\text{eff}}$.

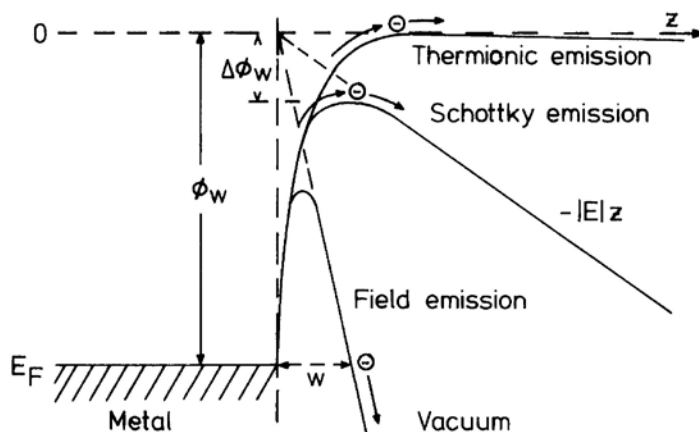


Figure 3: Potential diagram for electrons at the metal-vacuum-boundary.

c.) Field-emission Gun

The field emission is based on the quantum mechanical tunnel effect.

At a very sharp metal tip (e.g. tungsten) with radius of curvature $r < 100$ nm an applied voltage generates an electrical field strength, which reduces the width of the potential well in front of the tip to a few nanometers. Electrons near the Fermi energy can tunnel through this potential barrier and escape into vacuum, where they are then accelerated by a second anode to their final kinetic energy.

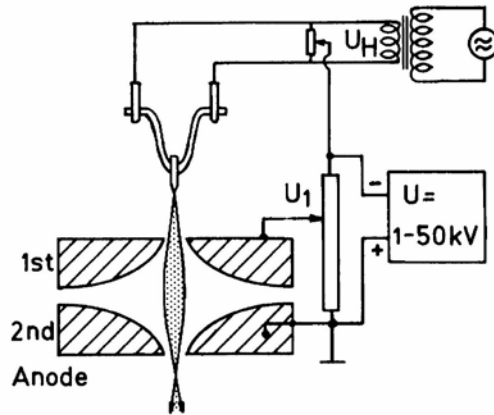


Figure 4: Schematic of a field-emission gun.

(© L.Reimer: Scanning Electron Microscopy, 2nd ed. 1998, Fig. 2.2, mit freundlicher Genehmigung von Springer Science and Business Media)

The main features of the various emitters are summarized in the following table:

	Thermionic Emitter	Schottky-Emitter	Field-Emitter
Cathode	tungsten, $\Phi=4,5$ eV $T=2500-3000$ K	ZrOW, $\Phi=2,7$ eV, $T=1800$ K, $r < 1\mu\text{m}$	tungsten, $\Phi=4,5$ eV $r < 0.1 \mu\text{m}$
Electron current density	Richardson-Equation $j \propto T^2 \cdot e^{-\Phi/kT}$	$j \propto T^2 \cdot e^{-(\Phi-\Delta\Phi)/kT}$ mit $\Delta\Phi = \sqrt{\frac{e^3 E}{4\pi\epsilon_0}}$	Fowler-Nordheim-Equ. $j \propto \frac{E^2}{\Phi} \cdot e^{-\Phi^{3/2}/E}$
Axial gun brightness [A/cm ² /sr]	$\beta = 10^5$	$\beta = 10^8$	$\beta = 10^9$
Energy spread [eV]	1	0,5	0,2
Size of the electron source	50 μm (crossover, reell)	20 nm (virtual source)	2,5 nm (virtual source)
E-Field [V/cm]	$\approx 10^4$	$\approx 10^6$	$\approx 10^7$
Vacuum requirement	low $p \sim 10^{-5}$ mbar	high $p \sim 10^{-8}$ mbar	high $p \sim 10^{-10}$ mbar

j: Current density
 T: Temperature
 Φ : Work function
 k: Boltzmann constant
 E: electrical field strength at the tip
 r: Radius of the tip

Axial gun brightness β : Current ΔI per area ΔA of the emitter surface and solid angle. β is constant along the optical axis.

$$\beta = \frac{\Delta I}{\Delta A \cdot \Delta \Omega} = \frac{j}{\pi \alpha^2} = \text{const}$$

ΔI Electron current
 ΔA surface element of the emitter
 $\Delta \Omega$ solid angle, $\Delta \Omega = \pi \alpha^2$
 α beam aperture, the semi-apex angle of the incident electron cone

1.2 Electron Lenses

The lens system of the SEM consists of one or two magnetic condenser lenses and one magnetic objective lens. It causes a multi-stage demagnification/reduction of the crossover onto the sample surface (reduction of about 10,000 means: 10 μm crossover - > 1 nm electron probe).

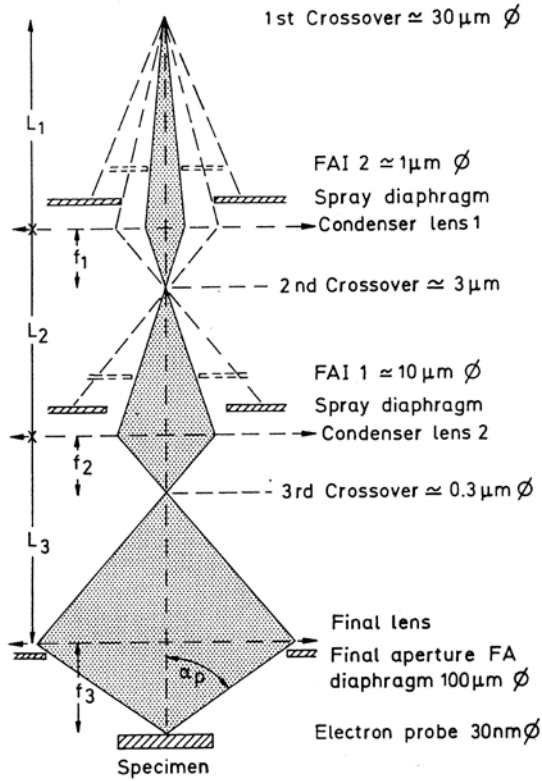


Figure 5: Schematic ray in the electron-optical column of a SEM.

(Abb. 5, 6: © L.Reimer: Scanning Electron Microscopy, 2nd ed. 1998, Fig. 2.13, 2.6, mit freundlicher Genehmigung von Springer Science and Business Media)

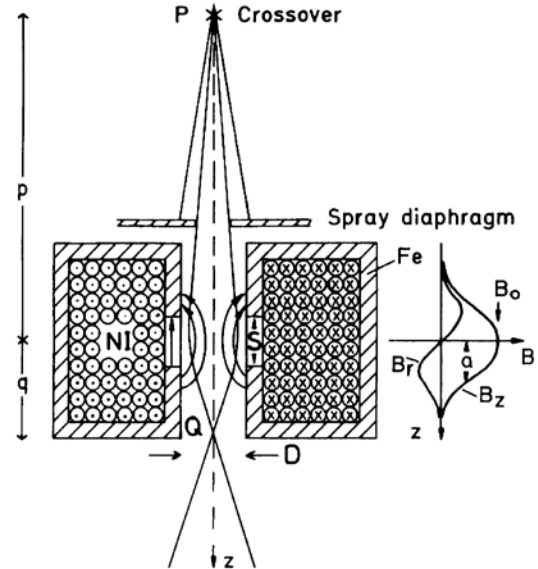


Figure 6: Typical electro-magnetic lens used in SEM.

Lens Aberrations / Resolution:

In principle, the resolution of the SEM is limited by the size of the primary electrons (PE) probe d_p on the sample surface. The size of the PE probe is determined by the size of the ideal reduced image of the crossover d_0 and the fault discs of the various lens aberrations.

The reduced image of the crossover d_0 can be calculated as a function of the gun brightness β , the probe current I_p and the probe aperture α :

$$\beta = \frac{\Delta I}{\Delta A \cdot \Delta \Omega} = \frac{I_p}{\frac{\pi}{4} d_0^2 \cdot \pi \alpha^2} \Rightarrow d_0 = \frac{2}{\pi} \sqrt{\frac{I_p}{\beta}} \frac{1}{\alpha} = C_0 \frac{1}{\alpha}$$

In order to achieve a sufficient signal-to-noise ratio, a probe current I_p of at least 10^{-12} - 10^{-11} A is necessary.

Spherical Aberration: $d_s = \frac{1}{2} C_s \alpha^3$ C_s spherical aberration coefficient
 $C_s \approx 10\text{-}20\text{ mm}$ in SEM ($C_s \approx 1\text{-}2\text{ mm}$ in TEM)

Diffraction Error: $d_B = 0.6 \frac{\lambda}{\alpha}$ $\lambda = h/mv$ DeBroglie-wavelength of electrons

Calculation of $\lambda = h/mv$:

For the kinetic energy E of an electron accelerated by a voltage U it is:

$$E = eU = (m - m_0)c^2 = mc^2 - E_0 \rightarrow m = m_0(1 + E/E_0)$$

where $E_0 = m_0 c^2 = 511\text{ keV}$.

m is the relativistic mass, m_0 is the rest mass of the electrons.

$$m = \frac{m_0}{\sqrt{1 - v^2/c^2}} \Rightarrow v = c \sqrt{1 - \frac{1}{(1 + E/E_0)^2}} \Rightarrow mv = \sqrt{2m_0 E(1 + E/2E_0)}$$

$$\Rightarrow \lambda = \frac{h}{mv} = \frac{hc}{\sqrt{2EE_0 + E^2}} \quad \text{Example:}$$

E	1 eV	10 eV	1 keV	10 keV	100 keV
λ	1,2 nm	388 pm	39 pm	12 pm	4 pm

Chromatic Aberration: $d_C = C_C \frac{\Delta E}{E} \alpha$ C_C chromatic aberration coefficient
 E energy of the PE
 ΔE energy spread of the electron gun

Axial Astigmatism: $d_A = \Delta f_A \alpha$ (can be compensated with a stigmator, e.g. quadrupole field)
 Δf_A difference in focal length

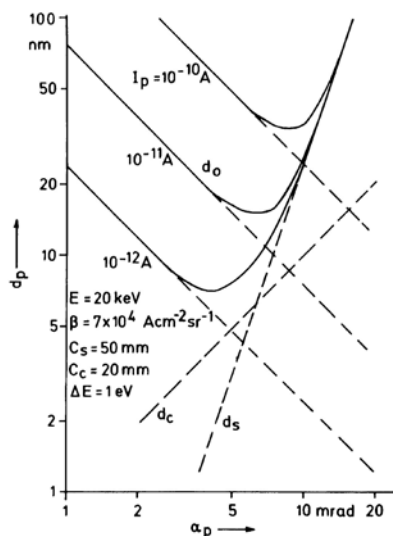
The resulting probe diameter d_p can be estimated by quadratic superposition of the error components:

$$d_p = \sqrt{d_o^2 + d_s^2 + d_B^2 + d_C^2} = d_p(\alpha)$$

In a SEM with a thermal cathode, C_0 is much larger than the DeBroglie wavelength λ , so that the diffraction error can be ignored. For electron energies in the range of $E = 10\text{-}20\text{ keV}$, the chromatic aberration is also small compared to d_0 and d_s due to the term $\Delta E/E$.

In Figure 7, the probe diameter in dependence of the aperture is shown. The smallest probe diameter is achieved for an optimum aperture α_{opt} , which can be calculated from $\partial d_p / \partial \alpha = 0$. The result is:

$$\alpha_{opt} = (4/3)^{1/8} (C_0 / C_s)^{1/4} \approx 5\text{-}10\text{ mrad} \quad \text{and} \quad d_{p,min} = (4/3)^{3/8} (C_0^3 C_s)^{1/4} \approx 5\text{-}10\text{ nm}.$$



Depth of Focus T

The depth of focus is:

$$T = \delta / \tan \alpha.$$

The small aperture in the SEM consequently leads to a large depth of focus.

δ denotes the minimal size of object details still resolvable on the monitor.

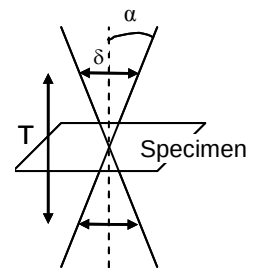


Figure 7: Probe diameter d_p as a function of aperture α for different probe currents I_p .

(© L.Reimer: Scanning Electron Microscopy, 2nd ed. 1998, Fig. 2.14, mit freundlicher Genehmigung von Springer Science and Business Media)

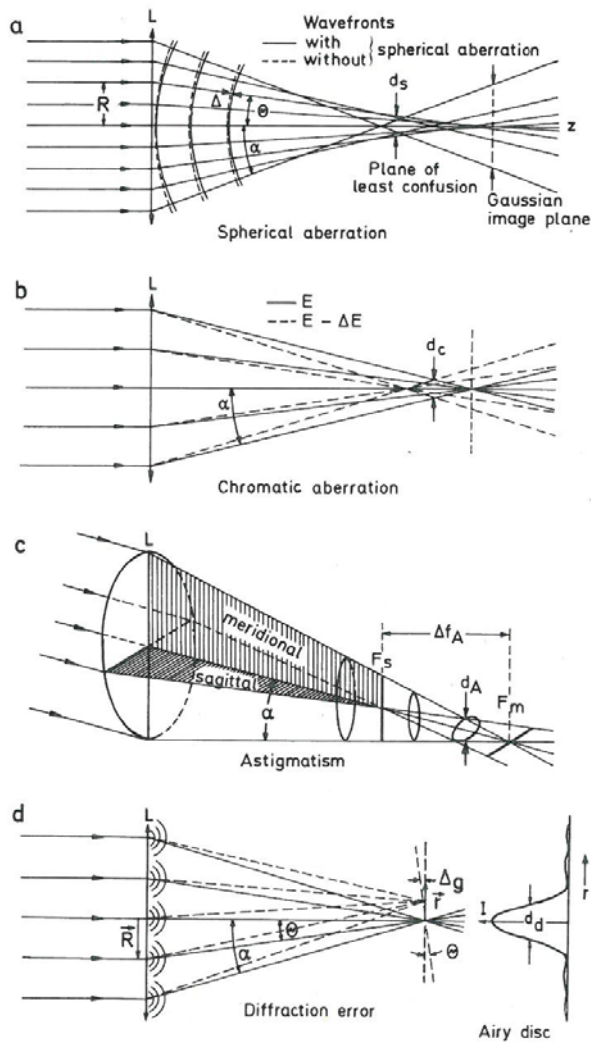


Figure 8: The most important lens aberrations in SEM

1.3 Deflection Elements

a.) Scanning Unit

The scanning unit deflects the finely focused PE-beam row-by-row over the sample area to be examined. For this purpose, a double deflection system is used (in x- and y-direction), in order to ensure that the PE-beam during scanning always runs through the center of the aperture (s. Fig. 9).

The interaction products (see below) caused by the PE in the scanned area point-by-point are detected and brought to the screen display. The magnification M of this scanning process is given by:

$$M = \frac{\text{Width of the monitor display}}{\text{Width of the scanned sample area}}$$

b.) Stigmator

Small asymmetries of the pole pieces or deviations from the ideal roundness of the apertures (caused by charges for example) can lead to an astigmatic image of the PE-beam probe on the sample. This astigmatism can be compensated by the electrostatic quadrupole field of a so-called stigmator (see figure on the right).

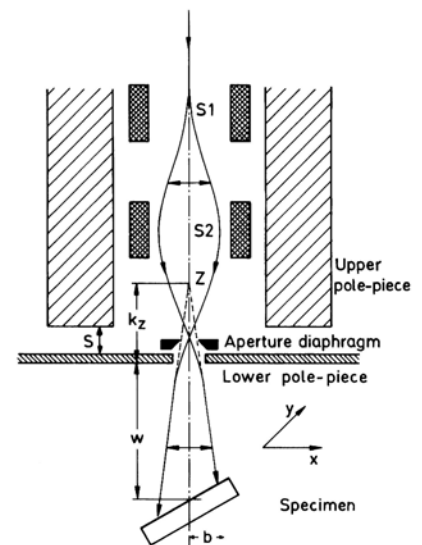
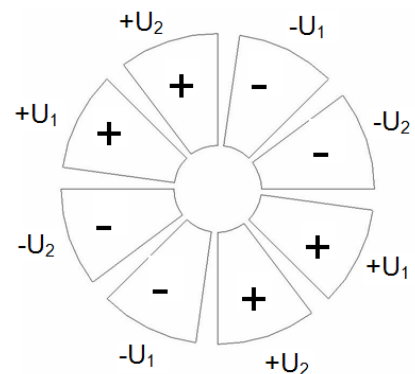


Figure 9: Objective lens with scanning unit



1.4 Detector

For detection of the interaction products caused by the PE such as secondary electrons (SE), backscattered electrons (BSE), X-rays (see below), different detectors are used:

Everhart-Thornley-Detektor (is used in the practical):

The Everhart-Thornley detector is a very low-noise high-bandwidth detector system for the detection of secondary and backscattered electrons (SE, BSE).

It consists of a combination of a scintillator and a photomultiplier. In the scintillator (plastic scintillator), the electrons generate photons due cathodoluminescence (10-15 photons per 10keV-electron, most of the electron energy is converted into heat). The head of the scintillator is surrounded by a collector with a grid whose potential can be varied from -300 V to +300 V. When the grid is at a positive potential, the SE are attracted and collected. When a negative potential (<-50 V) is applied at the collector grid no SE can overcome the grid, only the more energetic BSE can then reach the scintillator.

The surface of the scintillator is vaporized with a 50-100 nm thick aluminum layer and a potential of +10 kV is applied. The SE, which have passed the collector grid, therefore are accelerated to the scintillator, penetrate the metal layer and produce about 3000 electron-hole pairs, of which approximately 1-3% recombine at luminescent centers to photons.

The photons are lead due to total internal reflection along the light guide (plexiglass, quartz glass) to the photocathode of the photomultiplier, where they generate photoelectrons with a yield of 5-20% (Note: Each SE, striking the scintillator, leads to only about 1-10 photoelectrons. However, this relatively small conversion rate shows an exceptionally low noise). The photoelectrons are then accelerated to the first dynode (approximately +100 V) where they generate secondary electrons, which are avalanche amplified by the following dynodes. By changing the voltage of the photomultiplier, the gain can be varied over several orders of magnitude.

At the output resistance R (~1 MΩ), the electron pulse generates a voltage pulse, which can be further amplified electronically and is used for image control.

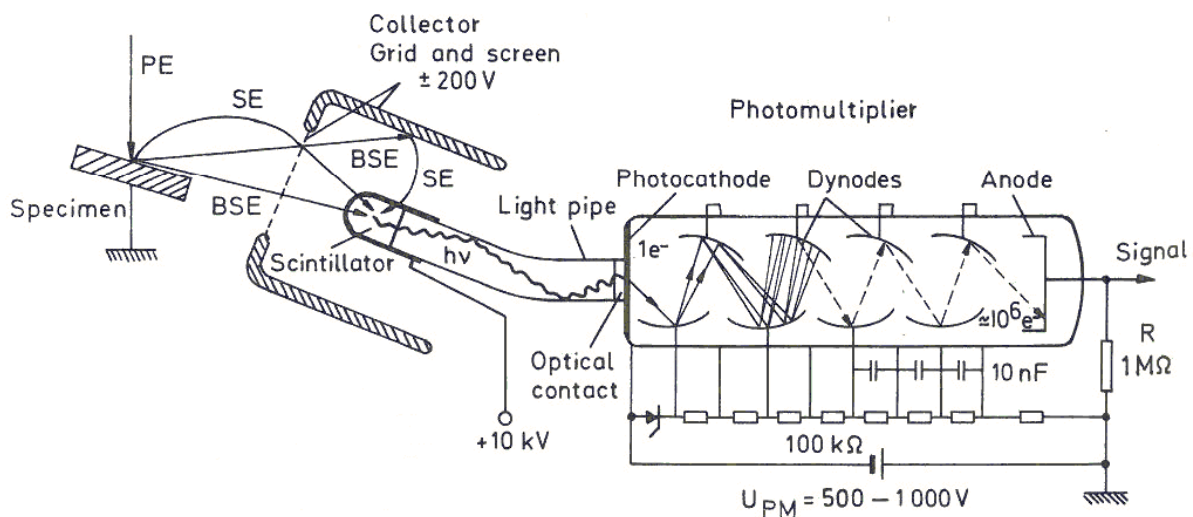


Figure 10: Schematic of the Everhart-Thornley-Detector.

(© L.Reimer, G. Pfefferkorn: Rasterelektronenmikroskopie, 2.Aufl. 1977, Fig. 5.1, mit freundlicher Genehmigung)

Semiconductor Detector:

This type of detector is suitable for the detection of backscattered electrons (BSE).

The detected BSE generate electron-hole pairs in a pn-junction. The charge carriers are separated in the depletion zone, which leads to a current signal.

Semiconductor detectors are characterized by a very compact design. With a semiconductor detector arranged above the sample as a ring, BSE can be detected in a wide angular range.

1.5 Imaging System

The SEM image is displayed on a monitor, with the image display being synchronized with the scanning of the PE-beam over the sample. The photomultiplier output signal controls the image brightness, that can be adjusted by varying the voltage of the photomultiplier dynodes. The contrast can be increased by subtracting a DC voltage (underground) from the video signal, in order to make small intensity differences visible.

2 Electron – Sample Interaction

The PE, incident on the sample, interact in elastic and inelastic way with the sample material. The sample area in which the interactions are taking place is referred to as electron diffusion cloud (which often has the form of a pear).

The range R of the PE in the sample is dependent on the PE-energy and the sample material.

R is approximately $R \approx E_{PE}^n$ mit $1.3 \leq n \leq 1.7$. $\rightarrow R \approx 0.1-10 \mu\text{m}$.

The maximum depth of information T is about half of the PE range $T \approx \frac{1}{2}R$.

In Figure 11 the electron diffusion cloud with the most important interaction processes and their information areas (a) as well as the dependence of the shape and size of the scattering area on the atomic number Z and the PE energy E_{PE} (b) is shown schematically.

Due to the interaction of PE with the sample, a spectrum of electrons with different energies is emitted (see Fig. 12).

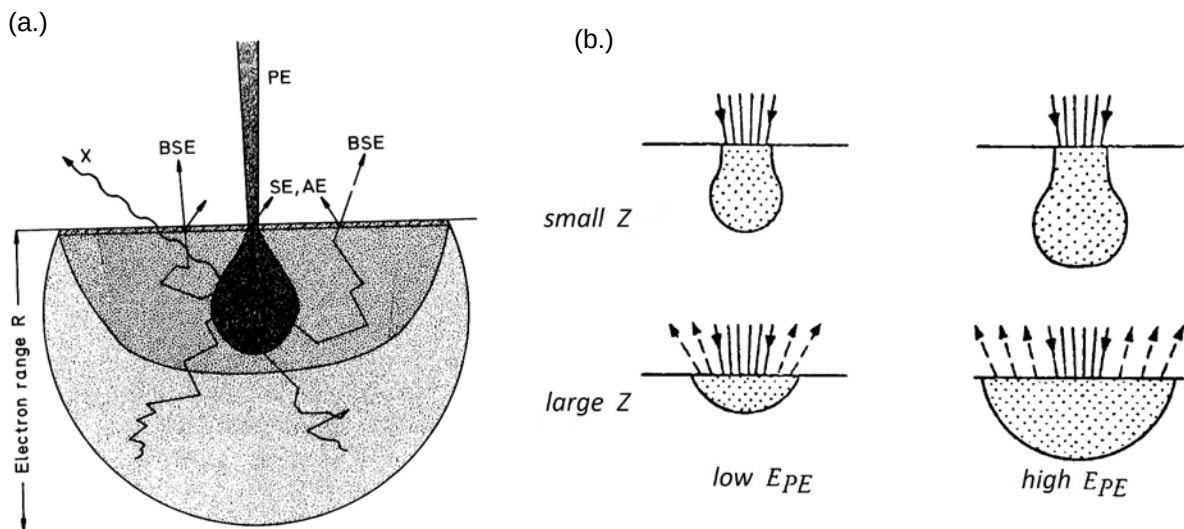


Figure 11: Electron diffusion cloud

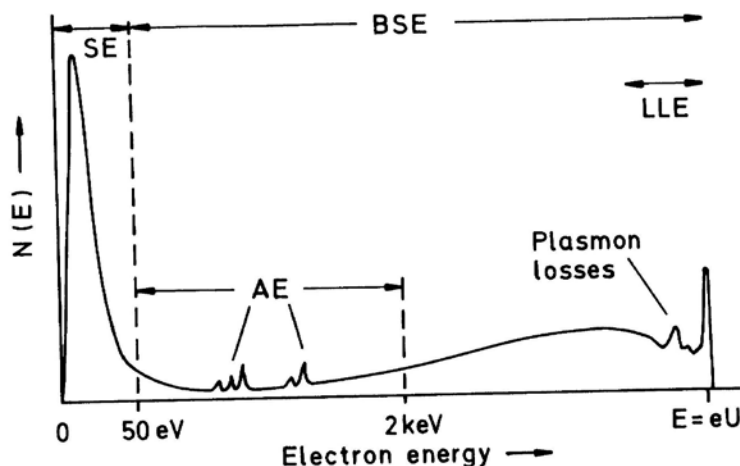


Figure 12: Energy spectrum of the emitted electrons

In the following the most important interaction products are briefly explained:

Secondary Electrons (SE)

SE are all electrons, whose energy is below 50 eV. Their most probable energy is 2-5 eV. Due to their low energy and therefore due to their low range, the SE come from a thin surface layer of thickness of 0.5 to 2 nm. The SE are caused by inelastic interaction of PE with the atomic shell of the sample material (ionization).

Backscattered Electrons (BSE)

All electrons with energy greater than 50 eV are referred to as BSE.

The broad spectrum of BSE (50 eV up to the PE-energy $E_{PE} = eU$) is caused by PE, who have lost different amounts of energy due to multiple scattering within the electron diffusion cloud.

The RE emerge from the cut of the sample surface with the electron diffusion cloud. The diameter of the emitting surface, as well as the escape depth T , is of the order of microns (μm).

Auger-Electrons (AE)

AE arise due to ionization and excitation of an inner atomic shell E_1 . During the transition of an electron from a higher energy shell E_2 to a lower ionized energy level, the energy $\Delta E = E_2 - E_1$ is emitted in the form of an X-ray quantum $h\nu = \Delta E$, or the energy can be transferred to another electron in the atomic shell that leaves the sample as an Auger electron with an energy characteristic for the sample material. As well as the SE, the AE originate from a small sample depth of a few nanometers due to their low energy.

Plasmon-Loss-Electrons (PL)

Electrons that excite density oscillations in the electron plasma suffer a characteristic energy loss.

Elastically Scattered Electrons (ESE)

In order to achieve high resolution also with BSE, those BSE have to be used that are almost elastically scattered (so-called low-loss electrons). Like the SE and AE, the ESE come from a sample layer very close to the surface.

Characteristic X-Rays

In consequence of the excitation and ionization of an inner atomic shell and subsequent transition to the ground state, emission of characteristic X-rays occurs (as a competing process to the production of Auger electrons). It is element-specific and can be used for analysis of the sample material (like the Auger electrons).

With atomic nucleus with a low atomic number ($Z < 20$), mainly emission of Auger electrons occurs.

With higher atomic numbers, the emission of characteristic X-rays dominate.

Cathodoluminescence

In addition to X-ray quanta, photons in the visible range can be emitted due to the excitation and relaxation of sample atoms.

Phonons / Specimen Heating

The largest portion of PE-energy is dissipated due to inelastic interaction of the PE in the form of phonon excitation or as heat. In general, the thermal conductivity of the sample is large enough so that thermal heating of the sample is not a problem.

In principle, all interaction products shown above can be used for image display. However for topographic imaging of samples, we restrict ourselves mostly to the detection of secondary and backscattered electrons. Their properties will be discussed in more detail below.

Definitions:

Sekundary Electron Yield δ :

$$\delta = \frac{\text{number of emitted secondary electrons}}{\text{number of incident primary electrons}}$$

Backscattering Coefficient η :

$$\eta = \frac{\text{number of emitted backscattered electrons}}{\text{number of incident primary electrons}}$$

3.1 Secondary Electrons (SE)

Energy Spectrum of SE:

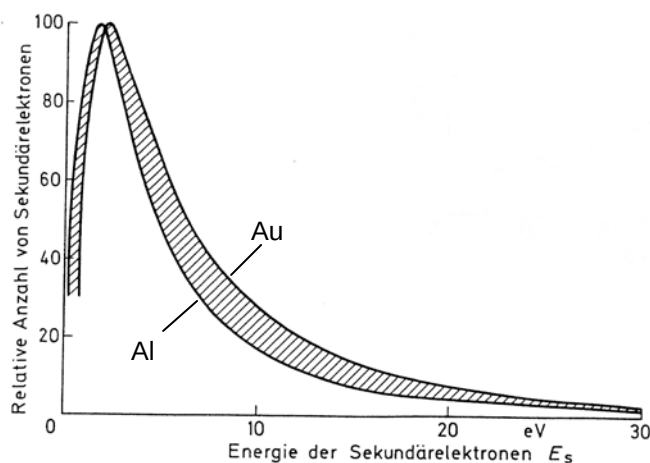


Abbildung 13: Energy spectra of secondary electrons from different metals normalized to unity.

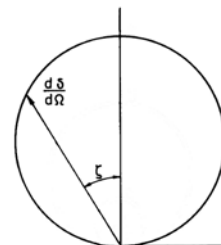
(© L.Reimer, G. Pfefferkorn: Rasterelektronenmikroskopie, 2.Aufl. 1977, Fig. 2.21, mit freundlicher Genehmigung von Springer Science and Business Media)

Angular distribution:

The directional distribution of the SE follows Lambert's law:

$$\frac{d\delta}{d\Omega} = \frac{\delta}{\pi} \cos \xi$$

(ξ is the angle of emission with respect to the surface normal)



Dependence on PE-Energy E_{PE} :

δ increases with increasing PE-energy up to a maximum at an PE-energy of several 100 eV and then decreases again (see Fig. 14).

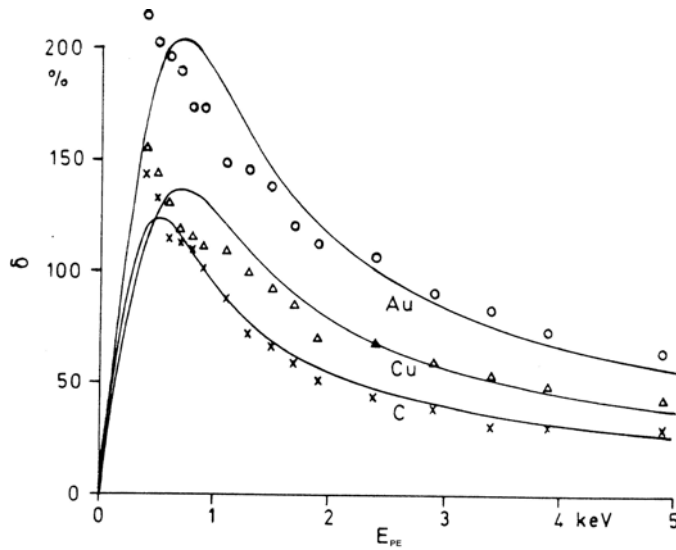


Figure 14: δ as a function of PE-energy.

Dependence on the sample material:

On average, δ increases slightly with increasing atomic number Z of the sample material (s. Fig. 17).

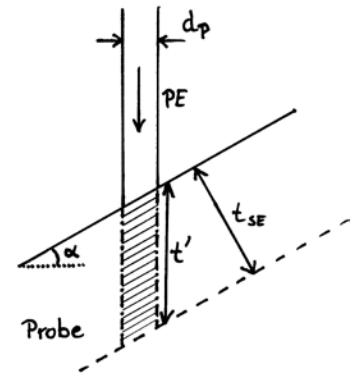
Dependence on the angle of incidence of the PE:

δ increases with increasing angle of incidence α of the PE to the surface normal.

With increasing angle α , the interaction path t' of the PE increases in the surface layer of thickness t_{SE} (escape depth of the SE).

Approximately, the following applies:

$$\delta \sim t' = t_{SE} / \cos \alpha \rightarrow \delta = \frac{\delta_0}{\cos \alpha}.$$



3.2 Backscattered Electrons (BSE)

Energy Distribution:

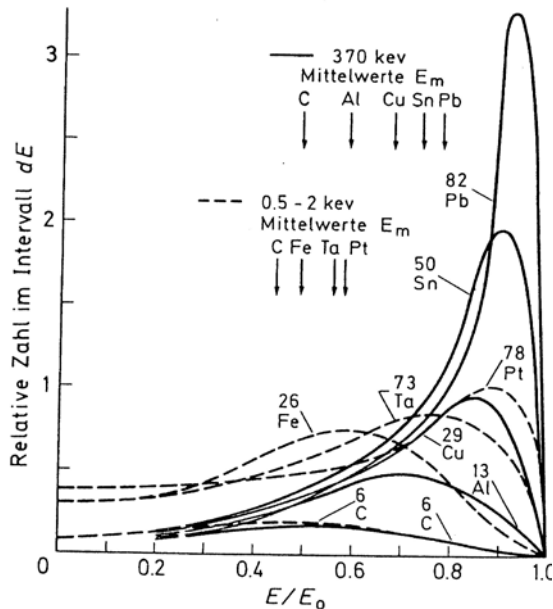


Figure 15: Energy spectra of backscattered electrons for different metals.

(© L.Reimer, G. Pfefferkorn: Rasterelektronenmikroskopie, 2.Aufl. 1977, Fig. 2.20, mit freundlicher Genehmigung von Springer Science and Business Media)

Angular distribution:

The directional distribution of the BSE follows Lambert's law for normal incidence of the PE:

$$\frac{d\eta}{d\Omega} = \frac{\eta}{\pi} \cos \xi \quad (\xi \text{ is the angle of emission with respect to the surface normal})$$

For non-normal incidence of the PE, the BSE-emission is more distinct in the opposite direction of the PE incidence direction (mirroring).

Dependence on PE-Energy E_{PE} :

η increases with increasing PE-energy, however, at $E_{PE} > 10$ keV remains approximately constant (see Fig. 16).

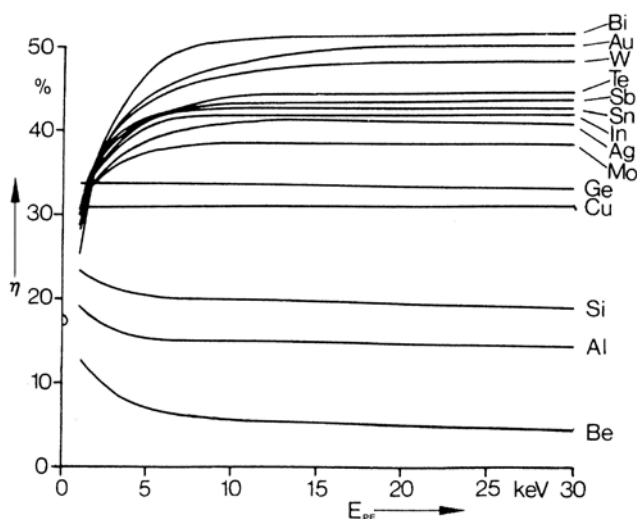


Figure 16: η as a function of PE-energy E_{PE}

(© L.Reimer: Scanning Electron Microscopy, 2nd ed. 1998, Fig. 4.9, mit freundlicher Genehmigung von Springer Science and Business Media)

Dependence on the sample material:

At PE-energies greater than ≈ 10 keV, η increases significantly with increasing atomic number Z of the sample material to (s. Fig. 17).

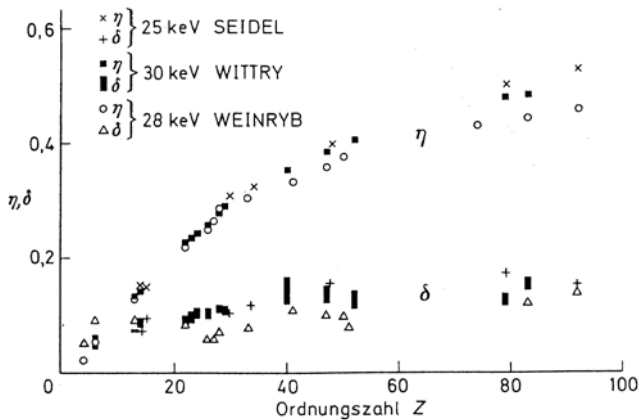


Figure 17: η as a function of atomic number Z

(© L.Reimer: Scanning Electron Microscopy, 2nd ed. 1998, Fig. 4.8, mit freundlicher Genehmigung von Springer Science and Business Media)

Dependence on the angle of incidence of the PE:

η increases with increasing angle of incidence of PE compared to the surface normal.

Contribution of backscattered electrons to the secondary electron yield:

Due to their low energy, the exit depth of the SE t_{SE} is only a few angstroms (e.g. t_{SE} (Si)=0.9 nm, t_{SE} (Au)=1.8 nm). The SE, which are triggered directly by PE (SE_1) come from the area of PE-probe and are responsible for the high resolution in SE mode.

However, BSE also excite secondary electrons (SE_2) on their path out through the surface layer of thickness t_{SE} .

The secondary electron yield is composed of the number of SE that are triggered per incident PE (δ_{PE}) and the number of SE that are triggered on average per BSE (δ_{RE}):

$\delta = \delta_{PE} + \eta \delta_{RE} = \delta_{PE} (1 + \eta \beta)$. The ratio $\beta = \frac{\delta_{RE}}{\delta_{PE}}$ indicates how much more SE are triggered per

BSE as per PE. In general, $\beta > 1$ (e.g. for Si at normal incidence of the PE with $E_{PE} = 4$ keV: $\beta \approx 5$, $\delta = 0.15$, $\delta_{PE} = 0.08$, $\eta = 0.19$, $\delta_{RE} = 0.37$).

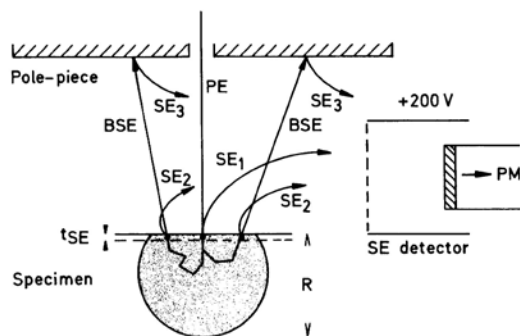


Figure 18: Contributions of SE_1 , SE_2 , SE_3 to the SE-signal.

(© L.Reimer: Scanning Electron Microscopy, 2nd ed. 1998, Fig. 4.27, mit freundlicher Genehmigung von Springer Science and Business Media)

In addition, SE which are triggered by BSE on the walls of the SEM chamber contribute to the secondary electron yield (SE_3).

At the resolution limit, the SE_1 contribute primarily to the image contrast and are responsible for the high resolution. The other SE-parts only contribute to the noise level because of their large exit area and therefore decrease the contrast at high resolution. The ratio of the SE_1 to the other SE-parts is more favorable, the smaller η is.

With increasing PE-energy, the SE_1 and SE_3 yield decreases. Since η increases or remains constant, the ratio of SE_1 / SE_2 becomes unfavorable.

Total Electron Yield σ :

The total electron yield σ is given by $\sigma = \delta + \eta$. Figure 19 shows the total electron yield as a function of PE-energy. At the energy values E_{c1} and E_{c2} it is $\sigma = 1$, so that the same amount of electrons leave the sample as much PE with energy E_{c1} or E_{c2} strike the sample. At these PE-energies, no electric charging of insulating samples is produced.

PE hitting on an insulating sample with an energy $E_{PE} < E_{c2}$, lead to $\sigma > 1$. More SE and RE leave the sample than PE arrive to the sample. The sample therefore charges positively, so that the PE reach the surface with more energy and σ decreases. The surface charges positively as long as $\sigma = 1$ is reached. However, the surface can only charge up to the positive collector potential of the Everhart-Thornley detector, otherwise the SE would again return to the surface.

For PE-energies $E_{PE} > E_{c2}$ (which is the case in most commercial SEMs), it is $\sigma < 1$, and the surface charges negatively. Therefore the incident PE are slowed down and reach the surface with lower energy, thereby increasing σ . An ideal insulator ($R \rightarrow \infty$) charges to a potential of $U - E_{c2}/e$, so that $\sigma = 1$ is reached (U is the acceleration voltage of the PE).

With an insulator having a finite electrical resistance, the energy eU_S of the PE in equilibrium can be constructed as the intersection point of the graph $\sigma(E_{PE})$ with a straight line through the point $(eU, 1)$ with gradient $1/(e I_p R)$ (see Fig.19).

At high PE-energies, considerable electric charging can be generated, that can almost reach the potential of the electron source. Sporadic surface discharges and breakthroughs through the insulator cause a temporally unstable charge distribution and thus an unstable image. Therefore, non-conductive objects have to be vapor-coated with an electrically conductive metal layer.

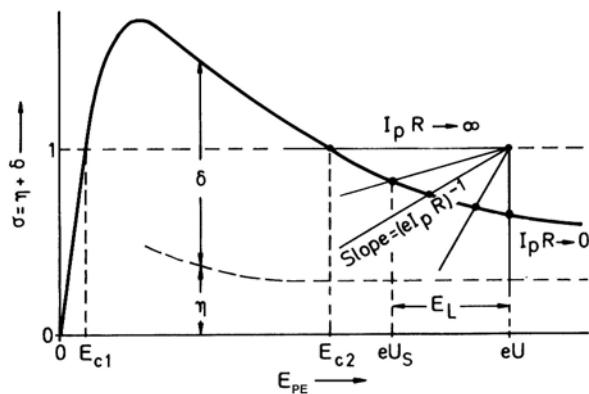


Figure 19: Total electron yield σ as a function of the PE-energy E_{PE}

(© L.Reimer: Scanning Electron Microscopy, 2nd ed. 1998, Fig. 3.31, mit freundlicher Genehmigung von Springer Science and Business Media)

3.3 Imaging of Surfaces – Contrast Modes

3.3.1 Topographic Contrast:

Imaging with the SEM provides a nearly spatial impression of the surface topography. For this purpose, different mechanisms are responsible:

- surface-tilt contrast
- shadow contrast
- increased electron yield at edges
- increased electron yield at rough surfaces
- high depth of focus

Surface-tilt contrast:

The smaller the angle between the sample surface and the PE-beam, the greater is the interaction path of the PE in the near-surface layer (from which SE_1 are triggered), and the greater is also the cut between the sample surface and the electron diffusion cloud (exit surface for BSE). Consequently, the more SE and BSE are emitted (i.e. the brighter surfaces appear), the flatter the PE beam incidents on the sample.

Shadow contrast:

With a detector that is positioned laterally at the side, shadows of objects can be observed. Object areas facing away from the detector appear darker than areas facing towards the detector. Electrons from object areas facing towards the detector are more easily detected by the collector, electrons from areas facing away from the detector, however, are partially reabsorbed (s. Fig. 20). The shadow effect is particularly evident in BSE-images (where only BSE contribute to the imaging signal due to a negative collector potential < -50 eV).

Increased electron yield at edges:

Outward edges between two inclined surfaces appear very bright (i.e. brighter than the adjacent sides), because on both sides near the edge SE and BSE emerge increasingly. Inward edges appear accordingly dark (see Fig. 21).

Increased electron yield at rough surfaces:

Due to the edge effect, a micro-rough surface appears brighter compared to a smooth surface of the same material.

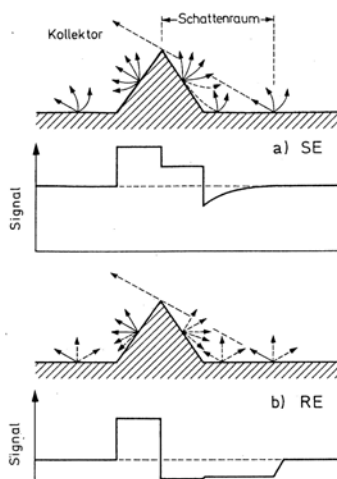


Figure 20: Shadow contrast

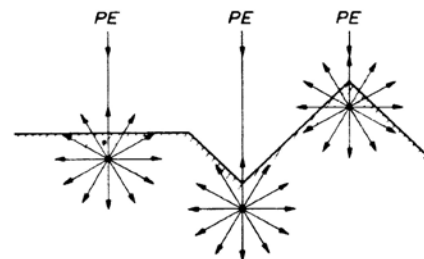


Abbildung 21: Increased/decreased contrast at edges

3.3.2 Material properties:

In addition to the topographic contrast types, contrasts that are based on material properties have to be considered:

- Material contrast
- Crystal orientation contrast
- Electrical potential contrast
- Magnetic field contrast

Contrasts due to different material properties are particularly well observed on topographically smooth surfaces, because there they are not mixed with topographic contrasts.

Material contrast:

Materials with different atomic numbers appear different bright in SEM, since the backscatter coefficient η depends on the atomic number Z of the specimen atoms (the dependence of the secondary electron yield δ on Z is less pronounced). Surface areas with larger Z appear brighter compared to areas with smaller Z .

Crystal orientation contrast:

The emission of BSE depends on the orientation of a lattice planes with respect to the incident PE-beam. Differently inclined crystallites on a smooth (polished) surface appear different bright.

(Electrical) Potential contrast (in SE-image):

Different electrical potentials on the sample surface cause different electric field distributions between the sample and the collector of the Everhart-Thornley detector.

SE emitted from negatively biased areas are more easily detected by the collector than electrons from positively biased areas. Consequently, negatively biased sample areas appear brighter than positively biased areas.

Magnetic field contrast:

External and internal magnetic fields affect the SE and BSE trajectories and thus their detection by the collector.

4. Elemental Analysis in SEM / EDX-Analysis

X-rays can be generated either by the deceleration of charged particles, e.g. the PE ($\rightarrow$ x-ray continuum or Bremsstrahlung), or by electron transition from a filled electron state into a vacancy in a lower atomic shell ($\rightarrow$ characteristic x-ray lines). The vacancy can be the result of ionization of sample atoms by the PE.

Characteristic X-rays are element specific and can therefore be used for the elemental analysis of a sample (Electron Probe Microanalysis). The energy of the emitted X-ray quanta corresponds to the difference between the energy levels involved in the emission, which is characteristic of atoms of an element.

The probability with which an ionized atom emits an X-ray is referred to as fluorescence yield ω_{ij} . This varies with the atomic number Z .

The X-ray bremsstrahlung forms a continuous background to the characteristic spectrum. The ratio of the characteristic X-ray radiation to bremsstrahlung decreases with increasing atomic number Z , so that the signal-to-background ratio, which determines the sensitivity of the analysis, is less favorable for heavier elements.

4.1. Detection

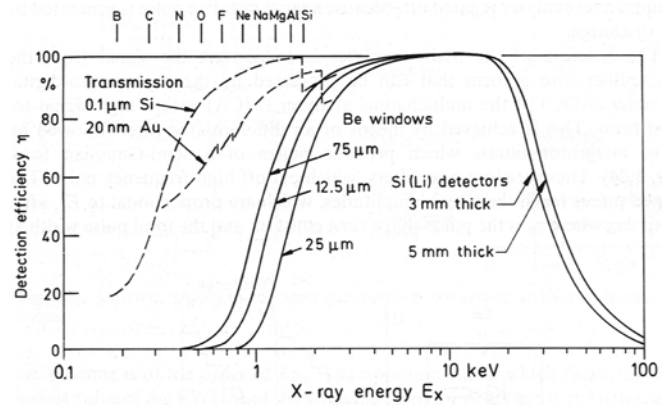
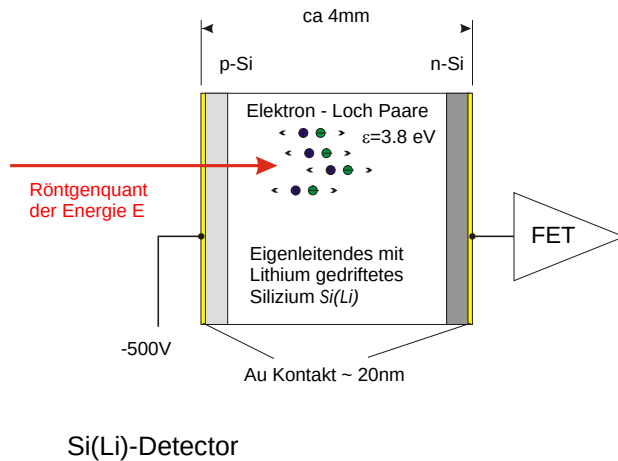
In the practical an energy-dispersive semiconductor detector (lithium-drifted silicon detector, abbreviated as "Si (Li)"), is used as an analytical extension to the SEM for the detection of X-ray radiation (energy-dispersive X-ray analysis, EDX). In principle, it is a Si-diode operated in the reverse direction, with a thin layer of p-type silicon, a thick middle layer of lithium compensated silicon and a layer of n-type silicon (constituting a p-i-n junction).

In order to achieve a possibly wide insulating and space-charge free depletion region for maximum absorption of X-ray quanta, lithium (Li) is diffused in the p-Si middle layer. Li acts as a donor and leads to a compensation of the present excess holes (acceptors). As a result, a p-i-n-diode (p-Si / insulator / n-Si) with a 3-5 mm wide intrinsic Si-zone is obtained.

X-rays are absorbed in the intrinsic zone and produce (inner) photoelectrons, which in turn diffuse through the crystal and release their energy to generate electron-hole pairs; the mean energy to produce an electron-hole pair in Si is 3.8 eV. In the applied electric field (biased in reverse direction), the charge carriers are separated and detected with a FET. Since the number of generated electron-hole pairs is proportional to the energy of the X-ray quantum, the X-ray energy can be determined from the amplitude of the current peaks.

In operation, Si (Li) detectors are cooled with liquid nitrogen, in order to prevent the diffusion of the highly mobile lithium ions, and to reduce the electronic noise of the detector. To protect the cooled detector from contaminations (freezing of water and freezable gases of the air) when venting the SEM sample chamber, a thin window is in front of the detector. Formerly, this was made of a $\sim 10 \mu\text{m}$ thin beryllium layer. Today ultrathin windows of polymer films are used for optimum transmission of the x-rays.

To be detected, the X-ray quantum must be absorbed within the detector crystal. At X-ray energies $>30 \text{ keV}$, the response, i.e. the percentage of detected X-ray quanta at a given energy, is limited by transmission through the crystal. At lower energies, the absorption of X-rays in the entrance window of the EDX-detector is the limiting factor.



Response of the Si(Li)-Detector

(© L.Reimer: Scanning Electron Microscopy, 2nd ed. 1998, Fig. 10.18, mit freundlicher Genehmigung von Springer Science and Business Media)

4.2. Quantitative Analysis

The aim of quantitative electron probe microanalysis is to determine the concentrations of all elements of an initially unknown sample from the spectral intensities of the X-ray emission lines.

In a first approximation, the intensity of an X-ray line of an element is proportional to its concentration c in the sample.

After that, for the determination of the concentration c_a of an element in a sample, the number n_a of γ -quanta of a characteristic line and the number n_s of γ -quanta of the same line of a pure element standard of known concentration c_s , measured under the same excitation conditions, would be sufficient:

$$k := n_a/n_s \approx c_a/c_s \rightarrow c_a \approx k \cdot c_s \quad (4.1)$$

However, in practice, with samples with a plurality of elements, the intensity of an X-ray emission line is generally not exactly proportional to the concentration of the corresponding element. The following effects are responsible for this lack of linearity:

- a.) Energy loss of PE along their path - depends on the specimen composition and influences the probability of ionization (no x-ray quanta can be excited once the electron energy has decreased below the ionization energy of the corresponding shell).
 - b.) Backscattering – depends on η and therefore on the specimen composition. The fraction of backscattered electrons get lost and therefore decrease the number of x-ray quanta generated by PE.
- These two effects are dependent on the atomic number Z and are considered in the so-called **→ Z-correction**.
- The x-rays can be absorbed inside the specimen, depending on the depth of x-ray generation and the mean mass attenuation coefficient of the sample.
→ (absorption) A-correction.
 - Characteristic x-ray quanta of an element 'A' of sufficient energy can ionize atoms of element 'B' by photoionization (fluorescence), resulting in an increase of x-ray quanta of element 'B' and a decrease of x-ray quanta of element 'A'. This is taken into account by the so-called **→ (fluorescence) F-correction.**

These three effects are considered in the quantitative analysis by correction factors k_Z , k_A , k_F (ZAF correction), i.e. Eq. 4.1 will be:

$$c_a = k \cdot c_s \cdot k_Z \cdot k_A \cdot k_F$$

To determine the correction factors, a set of intensities corresponding to the specimen composition is determined theoretically on the basis of a physical model. As the initially unknown concentrations of the elements in the sample enter in the calculation of the correction factors, the ZAF-correction is performed iteratively. It is the aim to find the set of concentrations, which explain the observed intensities of the characteristic X-ray lines of the elements involved at the very best. In the first step, estimates of the concentrations are assumed according to (4.1). The set of theoretical intensities calculated from these concentrations do not correspond to the observed intensities usually. Therefore,

a new set of concentrations is assumed, that is determined by comparison with the measured intensities according to

$$C_i^{(n+1)} = \frac{I_i^{\text{gemessen}}}{I_i^{\text{berechnet}}} \cdot C_i^{(n)} \quad (4.7)$$

This is repeated until all the theoretical and measured intensities match within a convergence criterion.

[*** The following can be skipped ***]

Ordnungszahlkorrektur (Z)

Die Berechnung der Anzahl primär in der Streubirne erzeugter Röntgenquanten ist Inhalt der sogenannten Ordnungszahl- (Z-) Korrektur. Sie berücksichtigt, dass in Proben unterschiedlicher Zusammensetzung im Mittel pro Elektron eine unterschiedliche Anzahl an Röntgenquanten einer Linie erzeugt wird. Die Anzahl an Ionisationsereignissen N_{ij} auf dem Weg eines Elektrons durch die Probe wird durch seinen Wechselwirkungsquerschnitt $\sigma_{ij}(E)$ entlang des Weges gegeben:

$$N_{ij} \propto \int_0^{s_{\max}} \sigma_{ij}(E) ds .$$

Der Ionisationsquerschnitt einer Schale ist im Verhältnis zum gesamten Wechselwirkungsquerschnitt für Elektronen vergleichsweise gering, so dass je nach Probenzusammensetzung statistisch gesehen pro Elektron nicht mehr als 10^{-5} - 10^{-4} Röntgenquanten freigesetzt werden. Leichte Elemente werden durch Elektronen wesentlich häufiger ionisiert, allerdings ist die Fluoreszenzausbeute hier recht gering.

Die Berechnung erfolgt für die Ionisation jedes Niveaus j (K-Schale, L-Schale etc.), das zu einer beobachteten Emissionslinie führt, eines jeden Elementes i der Probe. Der Weg eines Elektrons endet bei der Weglänge $s_{\max}$ entweder, wenn das Elektron seine kinetische Energie verloren hat oder als Rückstreuetelektron aus der Probe entweicht. Rückstreuetelektronen sind in der Regel noch energiereich genug, um Ionisationen in der Probe zu bewirken. Daher muß die Energie, die durch Rückstreuung zur Erzeugung von charakteristischer Röntgenstrahlung verlorengeht, durch einen Rückstreukorrekturfaktor R , in den der Rückstreckkoeffizient η der Probe eingeht, berücksichtigt werden.

Die Anzahl der aus den Ionisationsereignissen tatsächlich hervorgehenden, in der Probe primär durch den Elektronenstrahl erzeugten Röntgenquanten n_{ij} wird durch die Fluoreszenzausbeute ω_{ij} der jeweiligen Linie eines Elements beschrieben:

$$n_{ij} = \omega_{ij} N_{ij} \propto R \omega_{ij} \int_0^{s_{\max}} \sigma_{ij}(E) ds = R \omega_{ij} \int_0^{E_0} \frac{\sigma_{ij}(E)}{\left(\frac{dE}{ds}\right)} dE$$

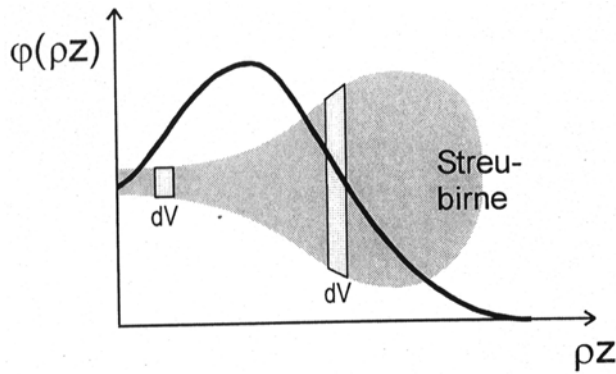
Da der Weg eines Elektrons und seine lafstreckenabhängige Energie keine zugänglichen Größen sind, wurde in der letzten Gleichung die Integration mit Hilfe des Energieverlustes pro Streckensegment dE/ds in eine Integration über die Energie übersetzt.

dE/ds und damit auch die Form der Streubirne werden wesentlich durch die mittlere Ordnungszahl der Probe bestimmt. Daher wird dieser Berechnungsschritt als Ordnungszahlkorrektur bezeichnet.

Eine alternative Form der Ordnungszahlkorrektur erhält man, wenn die Tiefenverteilung der Anzahl von Ionisationsereignissen zugrunde gelegt wird. Die Tiefenverteilungsfunktion wird üblicherweise als Funktion der Massendicke ρz der Probe angegeben und als $\Phi(\rho z)$ bezeichnet. Die Anzahl erzeugter Röntgenquanten ergibt sich in diesem Modell gemäß

$$n_{ij} = \omega_{ij} N_{ij} \propto \omega_{ij} \int_0^{\infty} \phi_{ij}(\rho z) d(\rho z) \quad (4.2)$$

Eine Rückstreukorrektur ist hier nicht notwendig, da die Tiefenverteilungsfunktion bereits ein Resultat der Wirkung aller Elektronen darstellt. Tiefenverteilungsfunktionen sind experimentell zugänglich oder können zuverlässig durch Monte-Carlo-Simulationen erzeugt werden. Verfahren, die auf Gl. (4.2) zurückgreifen, bezeichnet man als „ $\Phi(\rho z)$ -Verfahren“.



Tiefenverteilungsfunktion und Streubirne

Absorptionskorrektur (A)

Die Absorptionskorrektur berücksichtigt, dass die in der Probe erzeugte Strahlung zum Teil in der Probe wieder absorbiert wird und nicht vollständig zum Detektor gelangt. Die Schwächung von Röntgenstrahlung in Materie unterliegt dem LAMBERT-BEERSchen Gesetz und wird durch den Massenschwächungskoeffizienten μ beschrieben. Unter Verwendung der Tiefenverteilungsfunktion lässt sich die Anzahl nach aussen dringender Röntgenquanten berechnen:

$$n_{ij}^{\text{emittiert}} \propto \omega_{ij} \int_0^{\infty} \phi_{ij}(\rho z) e^{-\frac{\mu_{ij} \rho z}{\sin \alpha}} d(\rho z)$$

Hierin beschreibt α den Abnahmewinkel der Strahlung, d.h. den Winkel, den die Probenoberfläche mit der Verbindungsline zwischen dem Auftreffpunkt des Strahls auf der Probe und dem Mittelpunkt des Detektors einschließt. In diesem Bild ist die emittierte Intensität lediglich die LAPLACE-Transformation der Röntgentiefenverteilungsfunktion.

Die Röntgenabsorption der Probe für eine bestimmte Energie, z.B. bei der Energie E_{ij} der Linie j von Element i , setzt sich additiv aus den Einzelbeiträgen aller n Elemente der Probe zusammen :

$$\mu_{ij} = \sum_{k=1}^n c_k \mu_k(E_{ij})$$

Fluoreszenzkorrektur (F)

Die Intensitäten von Röntgenlinien können sich zusätzlich durch Fluoreszenzeffekte verschieben. Diese treten auf, wenn charakteristische Röntgenstrahlung auf ihrem Weg durch die Probe absorbiert wird und ihrerseits Emission charakteristischer Röntgenstrahlung geringerer Energie anregt. Dadurch wird die energiereichere Linie zusätzlich zur Absorption geschwächt, die energieärmere gewinnt hierdurch an Intensität. Das Ausmaß dieses Effektes hängt von der Probenzusammensetzung ab und kann Veränderungen um einige Prozent der Linienintensität bewirken. In die Berechnung des Fluoreszenzeffektes gehen hauptsächlich die Absorptionskoeffizienten μ der beteiligten Elemente bei den Energien der wechselwirkenden Linien sowie die Energien der Absorptionskanten ein.

Lab Work:

- Discussion/Repetition of the theoretical background.
- Introduction to the operation of the SEM.
- Investigation of some phenomena mentioned above using different samples.
- EDX-analysis: Qualitative and quantitative element analysis of a 1-Euro coin.

Protocol:

The following topics are expected in the protocol:

- Brief explanation of:
 - Principle structure and function of the SEM
 - Everhart-Thornley-detector, EDX (Si-Li)-detector
 - Interaction of PE with the specimen
 - Secondary electrons (SE-yield δ , dependencies)
 - Backscattered electrons (RE-coefficient η , dependencies)
 - Contrast modes (especially explanation of the surface-tilt contrast, potential contrast)
 - Resolution
 - Depth of focus (dependence on aperture. Why is the aperture in the SEM so small?)
 - EDX-analysis, ZAF-correction
- Detailed description and explanation of the observed effects with the investigated samples.
- Calculation of the x-ray energy of Cu-K α - and Cu-K β – peaks according to Moseley's law and comparison with the measured EDX-spectrum.
- Determination of the energy resolution of the EDX detector.

Literature:

1. L. Reimer *Scanning Electron Microscopy* (nat M 7501)
2. L. Reimer, G. Pfefferkorn *Rasterelektronenmikroskopie*
3. D. Cheshire, P. Goodhew *The Operation of Transmission and Scanning Electron Microscopy*
4. J.I. Goldstein, D.E. Newbury, P. Echlin, D.C. Joy, A.D. Romig, C.E. Lyman, C. Fiori, E. Lifshin, *Scanning Electron Microscopy and X-ray Microanalysis*, Plenum Press, **1992**. (nat M 7502)