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**Improved understanding of scattering
processes and their meaning for
quantitative image interpretation in
advanced scanning electron microscopy**

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Abstract

Environmental scanning electron microscopy (ESEM) enables the opportunity to investigate uncoated insulators, organic, biological or even wet samples in their original state. The presence of the imaging gas inside the specimen chamber is responsible for the secondary electron detection caused by gas amplification and the generated positive gas ions suppress charging artefacts. The drawback of this technique is the scattering of primary beam electrons inside the gaseous environment of the specimen chamber, which degrades the signal to noise ratio and complicates X-ray spectrometry.

The probability that a scattering event between an electron and a gas molecule occurs is described by the total scattering cross-section. In literature there is no relevant information about this cross-section for energy ranges used in electron microscopy. Therefore, a method is presented to determine the total scattering cross-section experimentally. Results for nitrogen, argon, water vapour and ambient air are presented for electron energies between 5 and 30 keV. To compare the results with theoretical calculations several Matlab programs were developed which draw suitable conclusions for optimising the experiment. The electron beam transfer through the gaseous environment is one of the most important factors which influences the performance of an ESEM. In this thesis, two different methods are presented to measure this primary beam scattering above the pole piece quantitatively. These methods allow to compare and to evaluate different microscope designs and their effect on the primary beam transfer through the imaging gas.

New technologies in high vacuum scanning electron microscopy enable the investigation of samples at very low electron energies (< 1 keV). The interaction volume and the signal diffusion is strongly decreased which improves the lateral resolution.

The interpretation of low voltage electron micrographs is complicated by new image formations and insufficiently known detection principles. In this thesis, model-like samples are investigated using low voltage backscattered electron images. Non-linear material contrasts of carbon/gold, aluminium/gold, silver/gold and iron/gold layer systems are shown and the contrast progressions compared with Monte Carlo simulations. Different elastic scattering theories are compared and their effect on backscatter coefficients is discussed. The experimentally determined cross-over energies are in good comparison with the Monte Carlo simulations using the interpolated Mott cross-sections, which indicates that the established basic model is correct and that the contrast progressions in low voltage electron backscatter micrographs can be determined with high accuracy.

In low voltage electron microscopy hydrocarbon contamination can complicate or even prevent the image acquisition. In order to understand this effect quantitatively, atomic force microscopy was used to measure the layer thickness. A decontamination device was constructed to decrease the layer thickness. An oxygen jet is directed towards the electron probe inside the specimen chamber, which leads to ozone generation. It can be shown that the influence of the hydrocarbon contamination on low voltage images is strongly decreased, but it can not be completely prevented.

Kurzfassung

Das Abbildungsgas in einem Environmental Scanning Electron Microscope (ESEM) ermöglicht die Untersuchung von elektrisch nichtleitenden, biologischen, organischen oder sogar feuchten Proben ohne zusätzlichen präparativen Aufwand. Der Nachteil dieser Methode ist die Streuung der Primärelektronen im Abbildungsgas. Dies verursacht ein verschlechtertes Signal-Rausch-Verhältnis und erschwert die Interpretation von Röntgenspektren.

Die Wahrscheinlichkeit, dass ein Streuprozess stattfindet, wird durch den totalen Streuquerschnitt beschrieben. Für ESEM relevante Abbildungsgase und Energiebereiche sind in der Fachliteratur nur unzureichende Ergebnisse verfügbar. Deshalb wird in dieser Arbeit eine Methode präsentiert, mit der dieser physikalische Parameter experimentell bestimmt werden kann. Um die Ergebnisse mit theoretischen Werten zu vergleichen wurden einige Matlab Programme realisiert. Dies ermöglicht die Verbesserung der experimentellen Methode und Rückschlüsse auf die Gültigkeit von Vereinfachungen und Annahmen in der Theorie können getroffen werden. In dieser Arbeit werden die Ergebnisse für Stickstoff, Argon, Wasserdampf und Umgebungsluft in einem Energiebereich von 5 bis 30 keV präsentiert. Die Leistungsfähigkeit eines ESEM wird maßgeblich durch den Transfer des Elektronenstrahls durch das Abbildungsgas beeinflusst. Es werden zwei Methoden präsentiert, die es ermöglichen den Elektronenstrahltransfer quantitativ zu bestimmen. Diese Methoden erlauben es zukünftig das Design von ESEMs zu optimieren.

Seit einigen Jahren können in der Hochvakuum-Elektronenmikroskopie Untersuchungen bei sehr niedrigen Beschleunigungsspannungen ($< 1\text{keV}$) durchgeführt werden. Das Interaktionsvolumen ist deutlich verringert, was zu einer Verbesserung der Oberflächensensitivität und der lateralen Auflösung führt.

Die Interpretation von Niederenergie-Abbildungen wird allerdings durch die Beobachtung neuer Kontrasterscheinungen und unzureichender Kenntnisse über die speziellen Detektoreigenschaften verkompliziert. Deshalb wurden in dieser Arbeit modellähnliche Proben mittels Niederenergie-Rückstreuелеktronen-Abbildungen untersucht. In den Materialsystemen Kohlenstoff/Gold, Aluminium/Gold, Silber/Gold und Eisen/Gold Systemen konnte eine Inversion im Bildkontrast nachgewiesen werden. Der Kontrastverlauf der Niederenergie-Rückstreuелеktronen-Abbildungen ist in guter Übereinstimmung mit Monte Carlo Simulationen, die mit Hilfe von interpolierten Mott-Streuquerschnitten berechnet wurden.

Insbesondere bei Niederenergie-Untersuchungen kann in der Rasterelektronenmikroskopie die Kontamination mit Kohlenwasserstoffen die Bildgebung entscheidend verschlechtern oder sogar verhindern. Um die Kontamination quantitativ zu bestimmen wurden Rasterkraftmikroskopische Messungen durchgeführt und diese mit Monte Carlo Simulationen verglichen. Eine am Institut entwickelte Dekontaminationseinrichtung wurde im Zusammenhang mit Niederenergie-Untersuchungen getestet. Ein Sauerstoffgas-Strahl wird in der Probenkammer auf die zu untersuchende Stelle gerichtet und damit Ozon erzeugt. Es konnte gezeigt werden, dass es zu einer starken Verringerung der Kontamination kommt.

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1 Motivation

In environmental scanning electron microscopy (ESEM) the presence of the imaging gas influences the image quality and the contrast mechanisms in a fundamental way. Especially the primary electron beam scattering in the imaging gas inside the specimen chamber degrades the signal to noise ratio and complicates X-ray spectrometry in the ESEM.

The progress in environmental scanning electron microscopy is complicated by a lack of information concerning the physical parameters which describe the probability that a scattering event takes place. As a result systematic errors in X-ray correction procedures occur and sophisticated research is absolutely necessary to calculate this effect in a correct way.

One of the most important factors which influence the signal to noise ratio, hence the performance of an ESEM, is the beam transfer through the gaseous environment. It is well known that commercial instruments are still far away from fundamental physical limits. Unacceptable noise in high pressure images is a result of inadequate beam transfer and the tremendous advantage of field emission guns is wasted. A lack of understanding of the gas flow inside the specimen chamber and the electron column hinders the ultimate enhancements of modern microscopes. Consequently, fundamental research is relevant for microscope manufacturers as well as for every microscope user.

In electron microscopy a quantitative understanding of contrast mechanism and image formation is of central importance.

New technologies in modern electron microscopes improve the lateral resolution in secondary and backscatter images, the signal to noise ratio and the surface sensitivity. The key technology for this progress is to decrease the electron energy which also means to reduce the signal diffusion. This trend to low voltage scanning electron microscopy poses a challenge for microscopists because contradicting literature studies concerning the contrast mechanisms and the image formation are available. Intuitive image interpretation is impeded, because well known and longstanding scattering theories are losing their validity at very low electron energies. Fundamental research on simplified samples is necessary for an improved understanding of electron solid interactions at low electron energies and comparison between experimental results and simulations are absolutely necessary for a well-founded image interpretation.

The new sophisticated developments in secondary and backscatter electron detection systems are insufficiently studied which additionally complicates image interpretations. Detailed research of these specialised detection systems and their influence on image formation is indispensable for every practitioner.

2 Fundamentals of Scanning Electron Microscopy

In a scanning electron microscope (SEM) a focused electron beam with a well defined size and energy scans line by line over the sample. The primary beam electrons interact with the specimen and the thereby produced particles are detected and give detailed information about the topography, morphology, chemistry and crystallography of the specimen. For this investigations an electron gun, an electron optical system to demagnify and focus the electron probe and several detectors are essential.

To avoid interactions of high energetic electrons with gas atoms or molecules by elastic and inelastic scattering and to prevent flashovers in areas with high electric field strengths a vacuum system is needed. A complete schematic setup of a scanning electron microscope is given in figure 2.1.

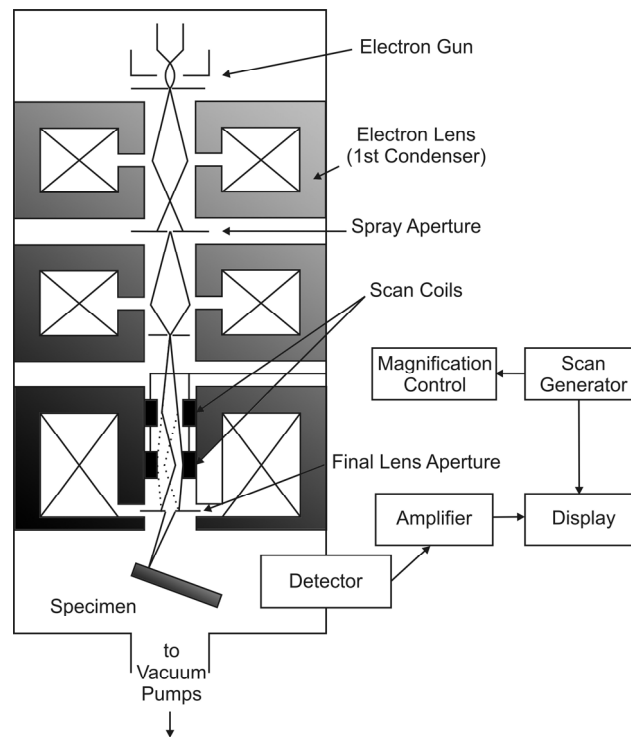


Figure 2.1 Schematic setup of a SEM (Goldstein et al., 2003)

2.1 Electron Guns

In a SEM a thermionic, a Schottky or a field emission cathode emits electrons which are accelerated towards the anode to energies between 0.1 and 30 keV. The main quality criterions of electrons guns are the brightness, defined as current density per unit solid angle of the source, the energy spread of the emitted electrons and the stability of the electron beam current.

2.1.1 Thermionic Electron Guns

By thermionic excitation, the electrons from the Fermi level of the cathode material can overcome the work function. In the strong electric field between the thermionic cathode and the anode the electrons are accelerated and applied to the electron optical system.

The Richardson law describes the emission current density J_C of a thermionic electron gun (formula 2.1):

$$J_c = A_c \cdot T_c^2 \cdot \exp\left(\frac{-\Phi_w}{k_B \cdot T_C}\right) \quad 2.1$$

where T_C is the emission temperature, A_C and Φ_w the material constant and the work function of the cathode and k_B the Boltzmann constant.

For practical reasons the cathodes are made of tungsten or lanthanum hexaboride (LaB_6) and heated between 2500 and 3000 K for tungsten and between 1400 and 2000 K for LaB_6 (Reimer, 1998). Because of the lower work function of LaB_6 ($\Phi_w(\text{LaB}_6) = 2.7 \text{ eV}$ in comparison with $\Phi_w(\text{tungsten}) = 4.5 \text{ eV}$) the emission current density of a LaB_6 cathode is of the order of $20\text{-}50 \text{ A cm}^{-2}$ in comparison to 3 A cm^{-2} for a tungsten cathode.

A crucial factor is that the hair pin design of a tungsten thermionic cathode is directly heated in comparison with an indirectly heated tungsten tip. LaB_6 polished tips are heated indirectly by squeezing them between carbon electrodes. Because of the dependency of the emission on the crystal orientation of the LaB_6 cathode the crystals are (100) oriented for a 10 times higher emission.

Tungsten hair pin tips unfortunately evaporate during operation which limits the lifetime to 40-200 hours. To prevent oxidation of tungsten tips a gun vacuum of $1\text{-}5 \cdot 10^{-3} \text{ Pa}$ is needed while for the protection of a LaB_6 tip a vacuum better than $10 \cdot 10^{-4} \text{ Pa}$ extends the lifetime to a few thousands of hours.

A negatively biased Wehnelt cup between the cathode and the anode forms the first cross-over of the electron beam (figure 2.2).

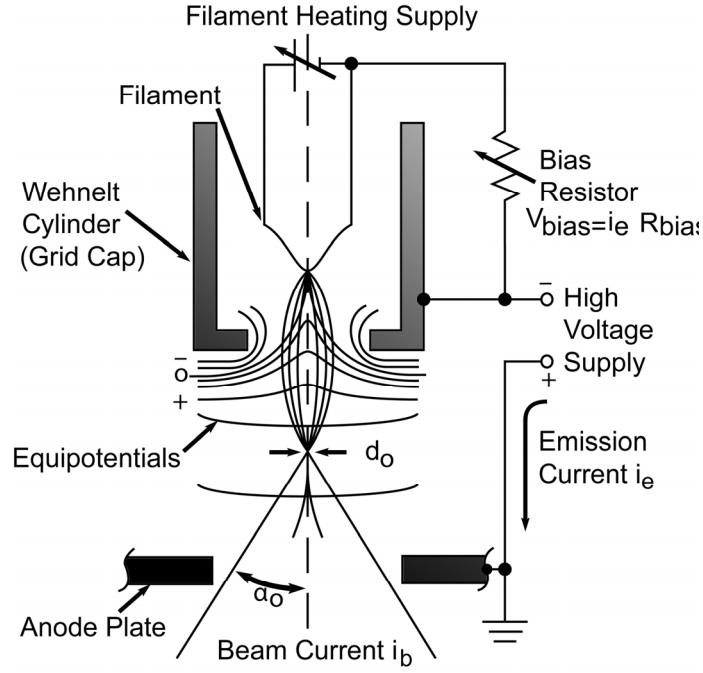


Figure 2.2 Scheme of a thermionic gun with Wehnelt cup (Goldstein et al., 2003)

The normal component of the initial exit momentum and the Boersch effect are responsible for the energy spread of thermionic electron guns ($\Delta E = 1\text{-}3\text{ eV}$ for tungsten cathodes, $\Delta E = 0.5\text{-}2\text{ eV}$ for LaB_6 cathodes).

The brightness β which is the most important quality criterion for an electron gun is (formula 2.2):

$$\beta = \frac{I}{\Delta S \cdot \Delta \Omega} = \frac{j}{\pi \cdot \alpha^2} \quad 2.2$$

where I is the beam current, ΔS is the cross-sectional area of the beam, $\Delta \Omega = \pi \cdot \alpha^2$ is the solid angle, α the beam aperture and $j = I/\Delta S$ the current density.

By using the Richardson law for the current density and a mean value of $\langle \alpha^2 \rangle$ after acceleration to an energy E , the maximum brightness is given by formula 2.3:

$$\beta_{\max} = \frac{J_C}{\pi} \cdot \left[1 + \frac{E}{k_B \cdot T_C} \cdot \left(1 + \frac{E}{2 \cdot E_0} \right) \right] \cong \frac{J_C \cdot E}{\pi \cdot k_B \cdot T_C} \quad 2.3$$

For thermionic electron guns typical brightness values at 25 keV are $0.3\text{-}2 \cdot 10^4\text{ A cm}^{-2}\text{ sr}^{-1}$ (tungsten tip at 2700 K) and a ten times higher value for a LaB_6 tip.

The linear decrease of the brightness with decreasing electron energy avoids the use of thermionic electron guns in low voltage scanning electron microscopy, in comparison to a Schottky or field emission gun.

2.1.2 Schottky Emission Guns

By using a tip diameter of $r \leq 1 \mu\text{m}$ the electric field strength at the cathode is at the order of 10^6 V/cm which decreases the work function.

The effective working function Φ_{eff} is (formula 2.4):

$$\Phi_{\text{eff}} = \Phi_W - \Delta\Phi_W = \Phi_W - e \sqrt{\frac{e \cdot |\vec{E}_{\text{efs}}|}{4 \cdot \pi \cdot e_0}} \quad 2.4$$

where e is the elementary charge, e_0 is the electric constant and the vector $\vec{E}_{\text{efs}}$ is the electric field strength.

The field strength is not strong enough to enable wave mechanical tunnelling, but it assists the thermionic emission. The electric field strength decreases the work function of the zirconium dioxide (ZrO_2) coating of the wolfram (100) tip from 4.5 eV to 2.7 eV. Typical values for a Schottky emission gun are: tip radius $r \sim 0.1$ to $1 \mu\text{m}$; energy spread $\Delta E \sim 0.5 \text{ eV}$, emission current density $j_c \sim 500 \text{ A cm}^{-2}$, gun brightness $\beta \sim 10^8 \text{ A cm}^{-2} \text{ sr}$ and a virtual source diameter of about 15 nm.

2.1.3 Field Emission Guns

In field emission guns (FEG) the electrons can penetrate the potential barrier by wave mechanical tunnelling. Electric field strengths higher than 10^7 V/cm and tungsten tip diameters smaller than 200 nm lead to this quantum mechanical effect by decreasing the work function. The potential barriers for the three different types of electron emission sources are shown in figure 2.3.

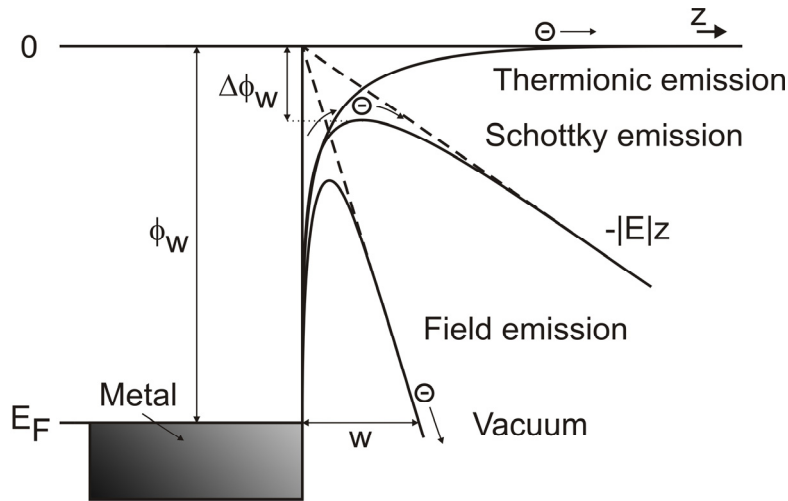


Figure 2.3 Potential barrier at the metal to vacuum boundary and decrease of potential energy with increasing external field for thermionic, Schottky and field emission (Reimer, 1998)

There are two types of FEGs: cold field emission works at room temperature and heated FEGs at 1000-1500 K. These systems need two anodes. One regulates the emission current by the applied field strength and the second one accelerates the electrons to their final kinetic energy.

The benefits of these electron guns are the outstanding brightness ($\beta = 10^8$ - 10^9 A cm⁻² sr at 20 keV), the small energy spread ($\Delta E = 0.2$ - 0.3 eV for the cold FEG and $\Delta E = 0.3$ - 0.5 eV for the heated FEG)) and the small virtual source size (3-5nm). Therefore, only one demagnifying lens is needed to produce a spot size with less than 1 nm diameter.

The drawback of this design is an inevitable pressure less than 10^{-6} Pa to protect the source from flashovers caused by the strong electric field.

2.2 Electron Optics

The electron optical system demagnifies the smallest beam cross-section at the gun, the cross-over, by a two or three stage lens system to an electron probe of 1-10 nm diameter. Of vital importance is not only the quality of the electron optics but also the diameter of the cross-over. By using a field emission gun or Schottky emitter with a very small virtual source size (10-100 nm) it is much easier to obtain a small probe diameter in comparison with a thermionic emission gun with a cross-over of about 10-50 μ m.

2.2.1 Magnetic Lenses

In an axial magnetic field with rotational symmetry the electron travels along a screw trajectory due to the Lorentz force (formula 2.5 and figure 2.4)

The Lorentz force $\mathbf{F}$ is given as:

$$\vec{F} = -e \cdot (\vec{E}_{efs} + \vec{v} \times \vec{B}) \quad 2.5$$

where $\mathbf{v}$ is the electron velocity and $\mathbf{B}$ is the magnetic field strength.

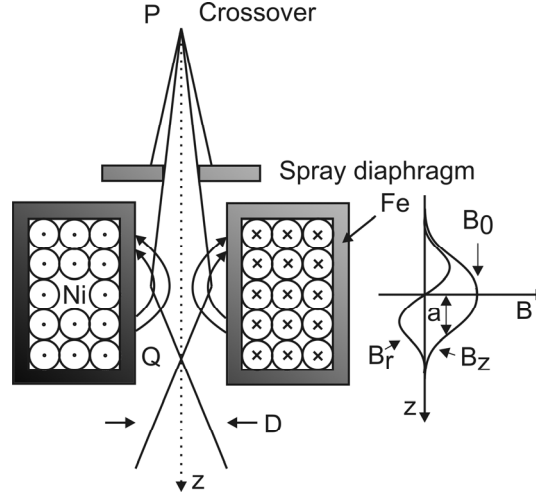


Figure 2.4 Schematic cross-section of a symmetric magnetic lens (Reimer, 1998)

The focal length f is defined by formula 2.6:

$$\frac{1}{f} = \frac{e}{8 \cdot m_0 \cdot U} \cdot \int_{-\infty}^{+\infty} B_z^2 dz \quad 2.6$$

where m_0 is the electron rest mass, U is the acceleration voltage, B_z the magnetic field component in z direction and $k_{strength}$ is a dimensionless parameter (formula 2.7), which is a measure for the strength of the magnetic lens. By using this parameter and the half width of the symmetric bell shaped magnetic field $2 \cdot a$, the focal length is given by formula 2.8:

$$k_{strength}^2 = \frac{e \cdot B_0^2 \cdot a^2}{8 \cdot m_0 \cdot U} \quad 2.7$$

$$f = \frac{2 \cdot a}{\pi \cdot k^2} \quad \text{for } k \ll 1 \quad 2.8$$

where B_0 is the maximum of the symmetric bell shaped magnetic field.

The reason for the screw trajectories is the Lorentz force which is perpendicular to the magnetic field lines. Therefore, the electron trajectories coiled round the optical axis results in

a rotation of the image depending on the focus of the lens, an artefact which is compensated in modern SEMs.

The final probe forming lens or objective lens has a longer focal distance for longer working distances which are necessary for most of the secondary and backscatter detectors, energy and wavelength X-ray spectrometry and electron backscatter diffraction.

The polepiece contains the scan coils, which are responsible for the scanning pattern of the electron beam on the specimen. The schema of the electron ray traces through a column is shown in figure 2.5.

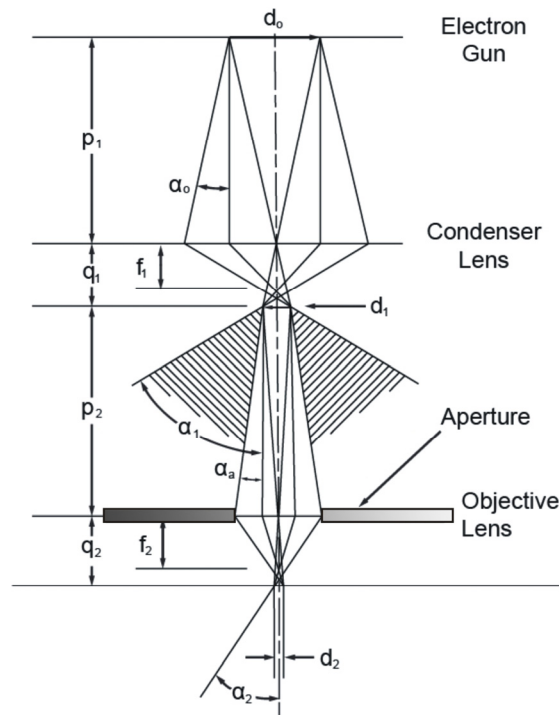


Figure 2.5 Electron ray traces through a schematic SEM column (Goldstein et al., 2003)

2.2.2 Special Lens Systems

Special designed microscope lens systems decrease aberrations and simultaneously increase detector efficiencies.

2.2.2.1 In-lens Operation

By minimising the working distance, which is the distance between polepiece and focus plane, the spherical and the chromatic aberration strongly decreases.

The chromatic aberration coefficient increases by decreasing energies and therefore this advantage is used in low voltage electron microscopy (Reimer, 1993; Reimer, 1998).

Because of the reduction in aberrations, smaller electron probe sizes are available which improves the lateral resolution. There are two different types of design, the complete in-lens design of the specimen and a semi in-lens design. The main disadvantage of the in-lens design is the restricted specimen size, which is necessary to place the specimen at the end of polepiece in the holder. By using working distances between one or two millimetres, which is realised in semi in-lens designs, a tradeoff between advantages and disadvantages can be achieved.

Another difference to a conventional SEM column is the detection of secondary and backscatter electrons which is discussed in chapter 5.

2.2.2.2 Magnetic Electrostatic Compound Objective Lens

The electrostatic lens consists of two parts. The inner part is at acceleration potential, the outer part at landing potential of the electrons. At the whole electron column the electron beam is at higher potential and in the gap between them the electrons are decelerated to the final energy. Therefore, magnetic and electrostatic stray fields can be shielded and the chromatic aberration is decreased. This makes the system very powerful for low voltage scanning electron microscopy (Tsunai et al., 1986; Frosien et al., 1989; Zach and Rose, 1988) (see figure 2.6). In comparison to conventional SEMs a different secondary and backscatter electron detection system is available which is discussed in chapter 5.

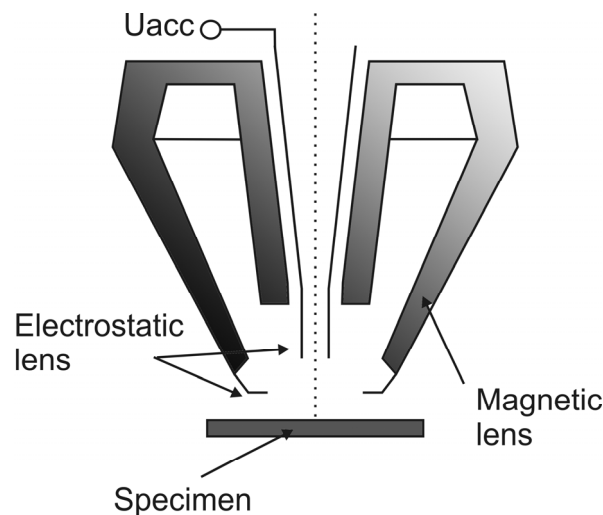


Figure 2.6 Schematic view of a magnetic electrostatic compound objective lens

2.3 Lens Aberrations

Several lens aberrations avoid perfect focusing of electrons in a magnetic lens system. Parallel electrons are not focused at the same focal length which leads to a plane of least confusion

instead of a focal point. The four main responsible aberrations are schematically shown in figure 2.7.

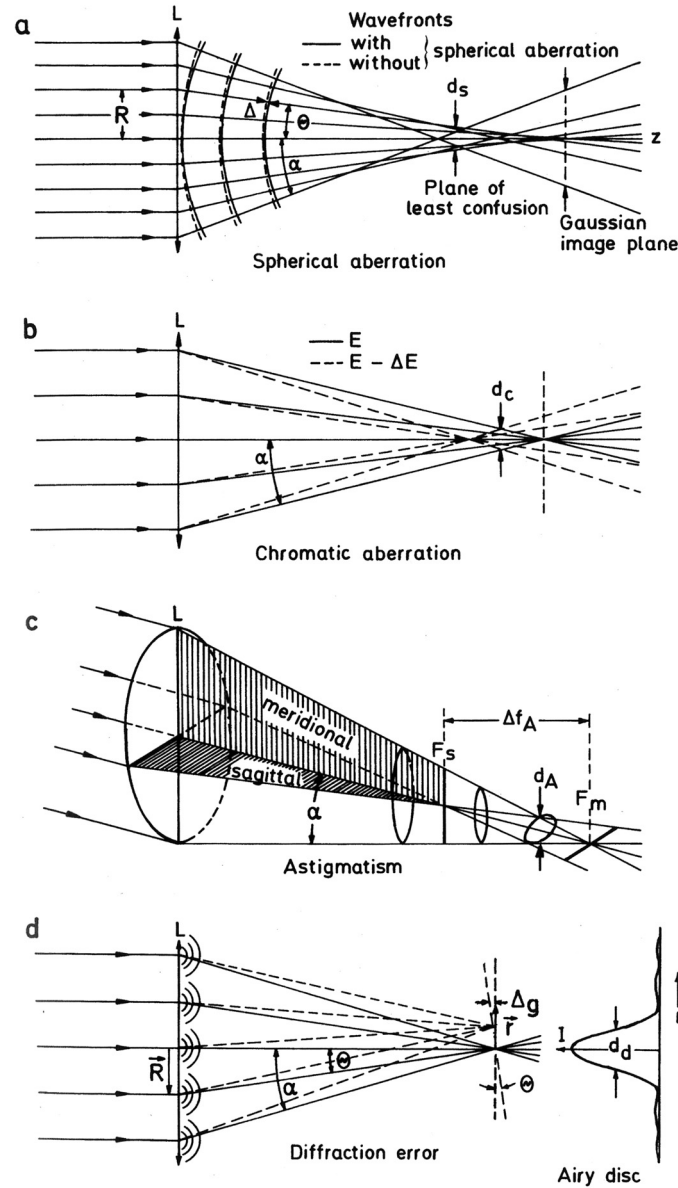


Figure 2.7 Illustration of the spherical and chromatic aberration and of the axial astigmatism and the diffraction error (Reimer, 1998)

2.3.1 Spherical Aberration

Parallel rays have different focus length depending on their distance to the optical axis. Electrons with a bigger distance from the optical axis are focused closer to the lens. By using the aperture limiting semi angle α_L and the spherical aberration coefficient C_s the diameter of the plane of least confusion d_s can be described by formula 2.9.

$$d_s = 0.5 \cdot C_s \cdot \alpha_L^3 \quad 2.9$$

For weak lenses used in microscopes with large working distances, the spherical aberration coefficient is about 20 to 100 mm. By using a microscope with an in-lens position of the specimen, this coefficient can be reduced to a few millimetres.

2.3.2 Chromatic Aberration

The focusing qualities of a magnetic lens depend on the energy spread of the electron beam. This spread is responsible for focusing in a disc of least confusion. Electrons of higher energy have a longer focal length than electrons with a smaller energy. For $k_{\text{strength}}^2 \ll 1$ the chromatic aberration coefficient C_C has the order of the focal length and the diameter in the plane of least confusion d_C caused by the energy spread ΔE is (formula 2.10):

$$d_C = C_C \cdot \frac{\Delta E}{E} \cdot \alpha_L \quad 2.10$$

2.3.3 Axial Astigmatism

Asymmetries in the focusing field are responsible for the axial astigmatism. Electrons in planes sagittal and meridional have different focal lengths. The sources of these asymmetries are magnetic inhomogeneities of the polepiece and charging effects. This astigmatism can be corrected by a cylindrical lens near the polepiece gap, which is controlled by the operator of the microscope.

The diameter of the disc of least confusion caused by axial astigmatism d_A is given by formula 2.11:

$$d_A = \Delta f_A \cdot \alpha_L \quad 2.11$$

where Δf_A is the difference between sagittal and meridional focus lengths.

2.3.4 Diffraction Error

The previously discussed aberrations increase with higher α_L and therefore a diaphragm in the objective lens lower the aperture limiting semi angle and minimises these effects. However, as a result of the diaphragm the electron beam with electron wavelength λ forms a Fraunhofer diffraction pattern at the focal plane. The plane of least confusion caused by the diffraction error d_D is described by formula 2.12.

$$d_D = \frac{0.6 \cdot \lambda}{\alpha_L} \quad 2.12$$

2.4 Vacuum System

In a conventional SEM a good vacuum is needed. By using a high resolution SEM with a FEG or Schottky emitter, turbo molecular, ion getter and rotary pumps are indispensable to produce the required vacuum. The strong electric field caused by the electron gun is capable to ionise any residual gas very easily which leads to flashovers and eventually to the destruction of the emitter.

This regulation also applies to the specimen chamber where the commonly used secondary electron detector, the Everhart Thornley detector (ETD), needs high vacuum because of its high voltage (~ 12 kV). However, a good vacuum is not only necessarily needed for the safety of the microscope but also to avoid elastic and inelastic collisions of the primary beam electrons with gas atoms or molecules. The main important physical parameter in this context is the mean free path Λ of the electrons in the gas which is described by the particle density n and the total scattering cross-section σ_T (formula 2.13).

$$\Lambda = \frac{1}{n \cdot \sigma_T} \quad 2.13$$

3 Environmental Scanning Electron Microscopy

Microscopists have always desired to investigate samples in the natural state. In contrast to SEM, environmental scanning electron microscopy (ESEM) enables the opportunity to investigate electrically insulating, vacuum intolerant and vacuum unfriendly samples.

3.1 Electrically Insulating Samples

In conventional SEM there are three possibilities to investigate insulating samples (Reimer, 1998).

The first possibility is by evaporating or sputtering the sample with a conductive layer, but this additional layer above the sample reduces the material contrast from the sample itself. A convolution of the signal from the sample and from the conductive layer is the result. The secondary electron yield from Au/Pd sputtered samples increase, nevertheless at the same time the contrasts in backscatter images degrade. A Monte Carlo simulation performed with the program Casino v2.42 (Drouin et al., 2007) demonstrates that a sputtered Au/Pd (80/20) layer on a polymer is almost completely responsible for the electron backscattering at 5 keV electron energy (see figure 3.1).



Figure 3.1 Depth distribution of backscattered electrons
(20 nm AuPd on polymer; 5 keV (left))

Trajectories of primary and backscattered electrons (red)
(20 nm AuPd on polymer; 5 keV (right))

Another disadvantage is that elements from the sputtered layer can complicate X-ray spectrometry. A possibility to overcome this problem is by evaporating a carbon layer, but then the secondary electron yield is decreased by simulations decrease in material contrast.

The second possibility to investigate insulating samples is using low voltage electron microscopy. The total electron yield σ (backscatter coefficient η plus secondary electron yield δ) increases with decreasing energy. At the critical energies E_1 and E_2 the total electron yield σ becomes 1 and no charging occurs because the amount of primary beam electrons hitting the specimen is equal with the amount of backscattered and secondary electrons leaving the sample. If $\sigma < 1$ the specimen charges negatively otherwise positively (figure 3.2). The critical energy E_1 is lower than 50–200 eV in comparison with E_2 (polymer: 1–2 keV) (Joy and Joy, 1999). In comparison to E_1 , image acquisition at energies around E_2 is strongly simplified. The primary beam electrons are accelerated or decelerated depending on the negative or positive charging of the sample. Therefore, the resulting landing energy of the primary beam electrons comes closer to the critical energy E_2 .

$$\sigma(\text{at } E_1 \text{ and } E_2) = \eta + \delta = 1$$

3.1

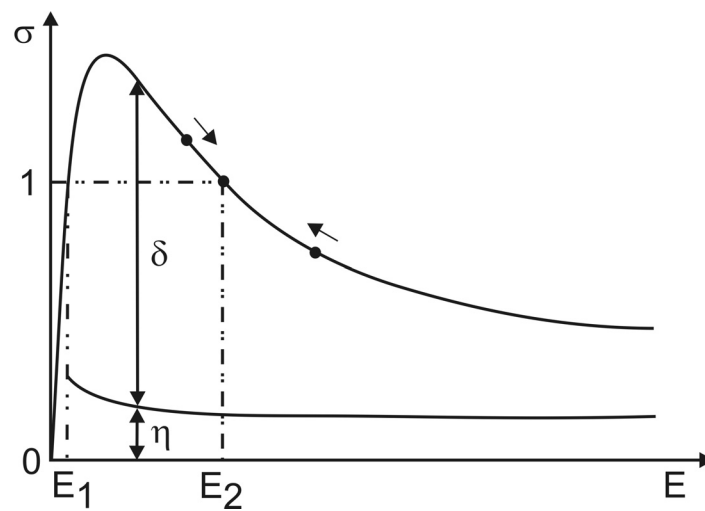


Figure 3.2 Total electron yield as a function of electron energy (Reimer, 1998)

In practice this method makes a lot of problems because the total electron yield is depending on the specimen tilt and on the compound. Therefore, insulating rough samples or samples with different chemical compositions are difficult to investigate by using this method.

The third method to investigate electrically insulating samples is by using ESEM.

3.2 Fundamentals of Environmental Scanning Electron Microscopy

A common problem in conventional SEM is the outgassing and drying out of samples. The vacuum environment leads to vaporisation of water and wet or biological samples can not be investigated. That means biological samples change their structure inside the vacuum of a

SEM. Drying, freeze drying and critical point drying are only a few possibilities to overcome these problems in a conventional SEM, but it is always a manipulation of the sample.

By cooling the sample the partial pressure of the surrounding gas decreases, but even then wet samples can not be investigated in a conventional SEM. Even at 0 degree Celsius the chamber pressure must be higher than 600 Pa to prevent vaporisation of wet samples (see figure 3.3).

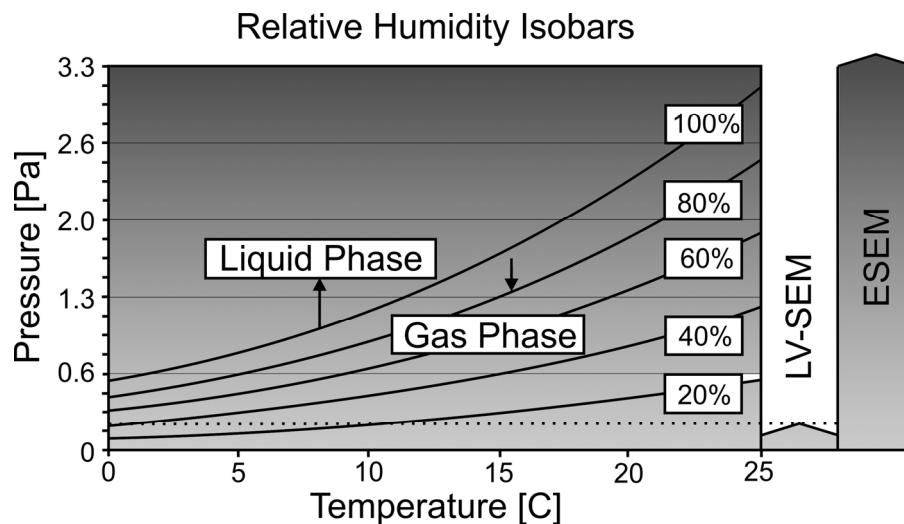


Figure 3.3 Relative humidity isobars for water (FEI, 1996)

Consequently from the beginning of electron microscopy there was a demand to investigate insulating samples or biological materials without outgassing at its natural state and ESEM technology allows us to overcome these problems.

The main challenge is the high vacuum environment in the specimen chamber and in the gun column. Because of the strong electric field nearby the electron gun (up to 10^7 V/m for a FEG), high vacuum or ultra high vacuum is necessary to avoid flashovers. Also the scattering of the primary beam electrons due to the presence of gas must be considered.

In addition to an essentially redesigned vacuum system, a complete new secondary electron detection system is necessary to resist the low vacuum environment in the specimen chamber. In the late 70's and 80's some innovations like separating the vacuum environment inside the gun chamber from the specimen chamber and the gaseous amplification detection system paved the triumphal way for the environmental scanning electron microscope (Kersker, 2001).

3.2.1 Vacuum System of an ESEM

To separate the high vacuum environment in the gun chamber from the low vacuum environment in the specimen chamber two pressure limiting apertures (PLA) are used. The position of these apertures at the end of the polepiece guarantees the pressure gradient between the different regions. The diameter of the PLAs must be large enough to allow the

electron beam to pass and small enough to decrease the gas flow from the specimen chamber upwards the electron column.

However, the apertures are not enough to stabilise the pressure regions. Between the apertures a part of the up streaming gas is pumped by an additional vacuum system.

To maintain a stable pressure during permanently pumping the microscope chamber, a gas source with an inlet in the specimen chamber is needed. Therefore, the resulting pressure in the specimen chamber depends on the gas flow into and out of the ESEM. The inflow from a selected source through an automatic metering valve is controlled by the operator and as a result the pressure in the specimen chamber keeps at a desired equilibrium.

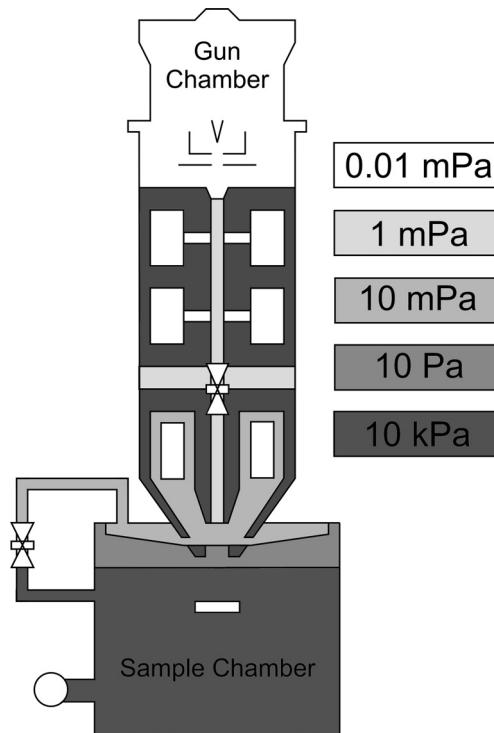


Figure 3.4 Pressure gradient in an ESEM (FEI, 1996)

With this system the electron gun is simultaneously under ultra high vacuum conditions ($<10^{-6}$ Pa for a FEG) during low vacuum conditions inside the specimen chamber (figure 3.4).

Even though the PLAs are located at the end of the electron column the primary beam electrons undergo elastic and inelastic scattering in the gaseous environment inside the specimen chamber which changes their trajectories. This so called skirt effect degrades the signal to noise ratio by generating gaseous secondary electrons as well as secondary electrons from regions far away from the focused probe. Therefore, the primary beam loses exponentially electrons to a broadly dispersed skirt along the way inside the gas (Danilatos, 1990).

The signal loss is described in formula 3.2:

$$I = I_0 \cdot e^{-\sigma_T \cdot n \cdot BGPL} = I_0 \cdot e^{-m} \quad 3.2$$

where I is the remaining unscattered primary beam, I_0 the primary beam current (scattered and unscattered), BGPL is the beam gas path length and m the average number of interactions per electron with the imaging gas atoms or molecules.

By increasing the pressure the signal to noise ratio degrades, which can prevent image acquisition at high pressure regions. This average number of collisions is very important for environmental scanning electron microscopy. As a reference point this value should be less than 3. Above this value the total scattering regime starts and image acquisition is not possible any more (Stokes, 2009).

The beam gas path length is described by the distance an electron travels inside the gaseous environment. The transition from high vacuum in the electron column to the specimen chamber is not a sharp one and therefore the BGPL is not equal to the distance between the PLA and the specimen. It depends on the type of gas and the pressure difference between the column and the chamber. In figure 3.5 a simulation of this transition region can be seen. The pressure starts to decrease at a diameter D below PLA1 and decreases up to a distance h above this PLA. From that point the pressure is particular uniform again (Danilatos, 1994).

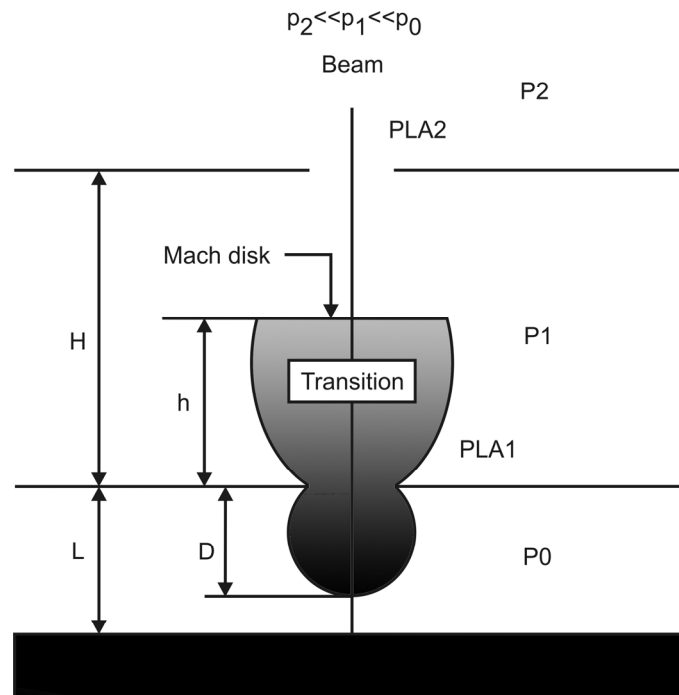


Figure 3.5 Gas density zones between specimen and pressure limiting aperture (Danilatos, 1994)

3.2.2 ESEM Secondary Electron Detectors

A strong electric field inside a conventional SEM is produced by the secondary electron detector, which is normally the Everhart Thornley detector. Therefore, a totally new physical principle for the detection of secondary electrons (SEs) was needed to work under low vacuum conditions.

The simplest construction of an ESEM secondary electron detector is a positively biased electrode. The emitted secondary electrons are attracted by the electric field of the detector and accelerate towards the electrode. On their way through the gas they undergo collision ionisations which lead to a multiplication of the charge carriers. This physical effect amplifies the secondary electron signal which is collected by the detector (figure 3.6).

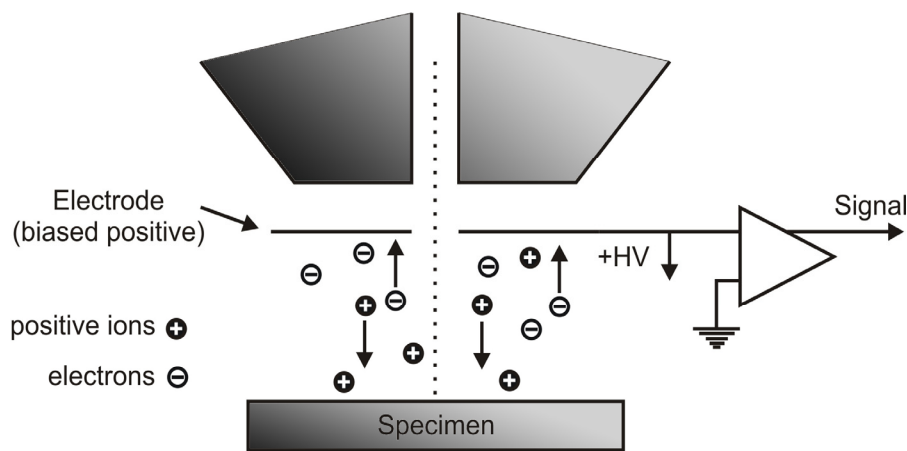


Figure 3.6 Secondary electron detection in an ESEM (Goldstein et al., 2003)

Essential for the amplification effect is the imaging gas type, the electric field geometry and strength, the gas pressure and the distance between detector and focused point. The distance between detector and specimen and therefore the amplification can be changed by varying the working distance. The bias of the detector can be controlled by the user interface as well as the chamber pressure and the type of imaging gas. For an uniform electric field the amplification is described by formula 3.3 (Thiel, 2003):

$$I_G(SE) = \delta \cdot I_0 \cdot e^{\alpha_{Townsend} \cdot ds} \quad 3.3$$

where $I_G(SE)$ is the ion current attributed to secondary electron amplification, $\alpha_{Townsend}$ is Townsend's first ionisation coefficient and ds the detector specimen gap.

The relatively small bias (a few hundred volts) of the detector normally avoids flashovers and secondary electron detection under low vacuum conditions is possible. However, not only the SEs lead to gas ionisation. The collisions of the primary beam electrons and the backscattered electrons (BSE) with the gas atoms or molecules decrease the signal to noise ratio (Stokes, 2009) (formula 3.4). These signals are additionally amplified in the specimen chamber:

$$I_{amp} = I_G(PE) + I_G(BSE) + I_G(SE)$$

3.4

where I_{amp} is the amplified electron current and $I_G(PE, BSE, SE)$ the amount attributed to primary beam electrons, backscattered electrons and secondary electrons.

Meredith et. al. published 1996 the contribution of this signal for a carbon specimen under water vapour conditions with 2 mm electrode specimen gap, a bias of +375 V and an electron energy of 15 keV.

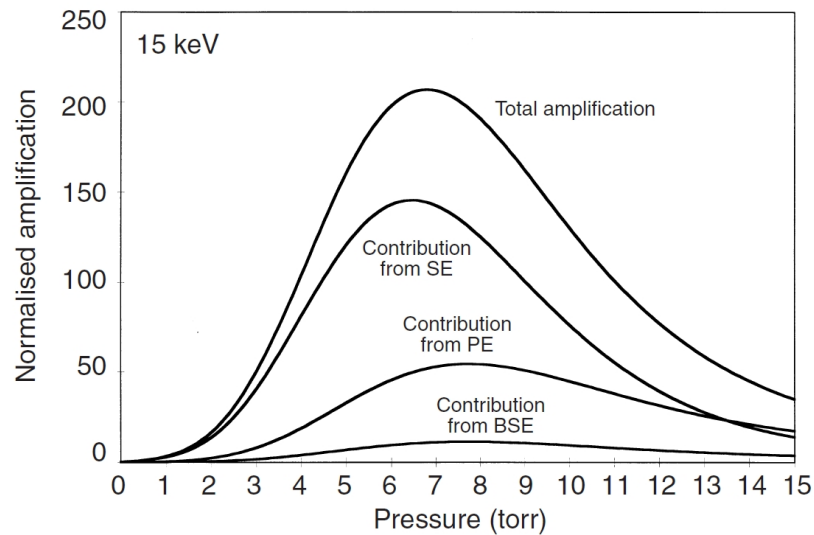


Figure 3.7 Contributions to the total amplification (Meredith 1996)

At the ESEM Quanta 600 two different types of gaseous secondary electron detectors are available, the large field detector (LFD) and the gaseous secondary electron detector (GSED) (figure 3.8).

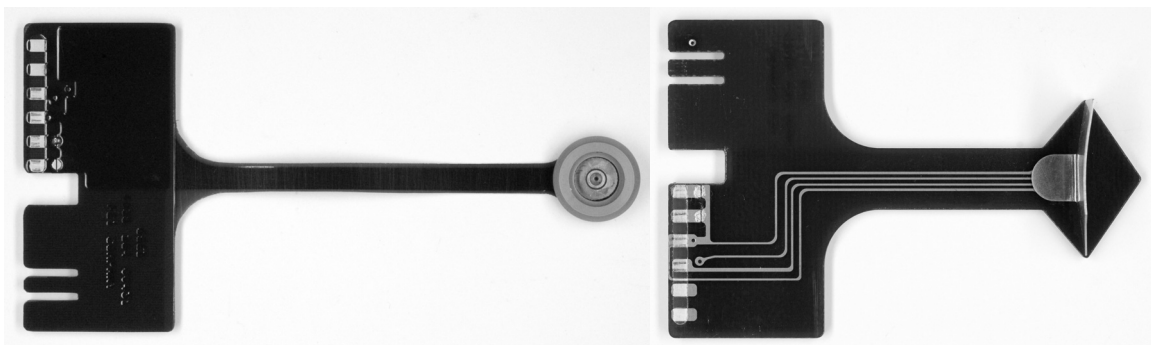


Figure 3.8 Gaseous secondary electron detector (GSED) (left),
large field detector (LFD) (right)

The LFD is a dedicated low vacuum detector, which means that the upper pressure limit is about 200 Pa. There is no second PLA which minimises the gas flow upwards the electron column. The benefit of this detector is the larger field of view in comparison with the GSED and the possibility to collect secondary electrons and backscatter electrons at the same time.

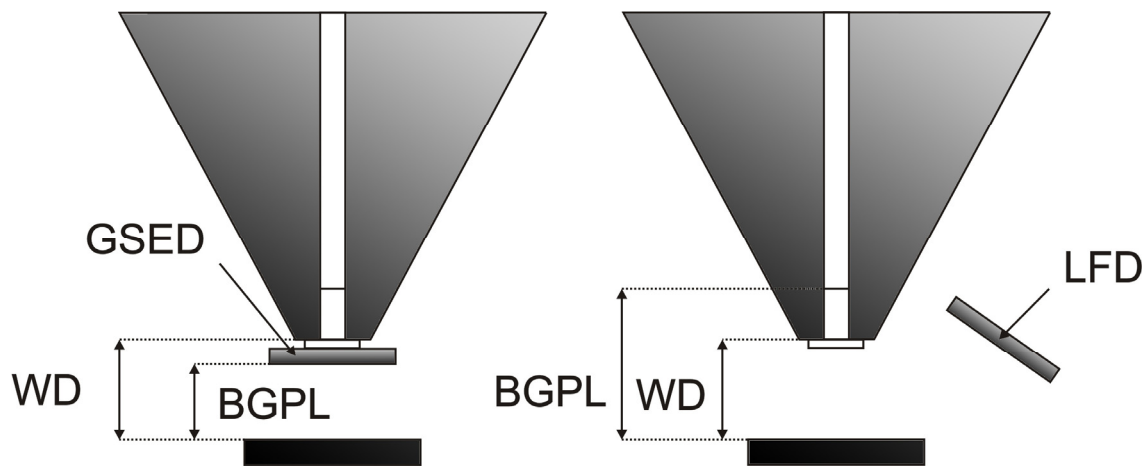


Figure 3.9 Position of the GSED (left) and the LFD (right)

By using the GSED, no backscatter detector is available because of the position of the detector at the end of the polepiece (figure 3.9). The benefit of this detector is the possibility to increase the chamber pressure. The second PLA is located inside the detector, which enables full ESEM pressure (maximum pressure around 3 kPa) inside the specimen chamber. With this detector even wet samples can be investigated.

The GSED has a suppressor electrode to prevent the detection of SE3 electrons (figure 3.10). Caused by the position of the detector at the end of the polepiece, the amount of the detected backscatter electrons is higher in comparison to the LFD.

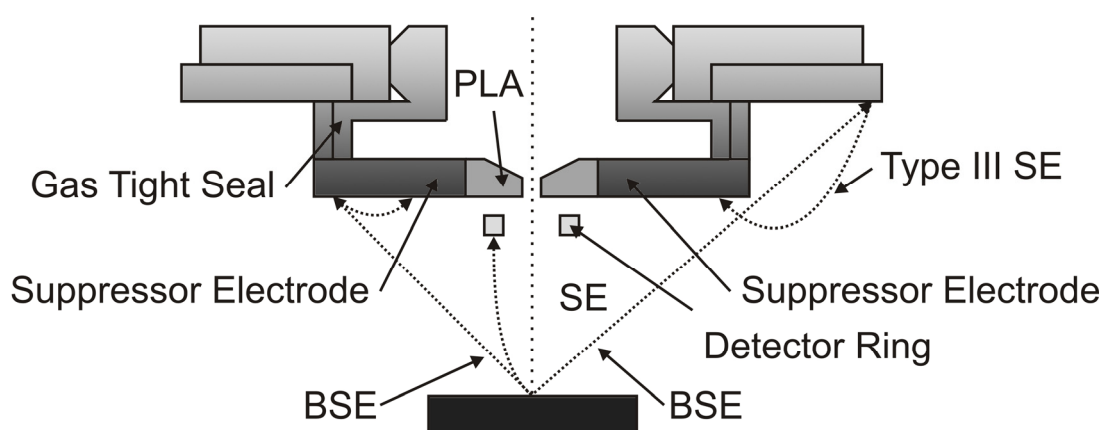


Figure 3.10 Schematic drawing of the GSED detector (FEI, 1996)

To enhance the signal to noise ratio a decrease of the primary beam scattering is necessary. However, by decreasing the working distance the gap between the specimen and the detector is reduced which influences the secondary electron amplification.

Therefore, a new detection device was designed, which is available in the latest generation of some high end microscopes. The Helix detector from FEI combines the two necessary parameters for an ESEM detector (Knowles et al., 2005).

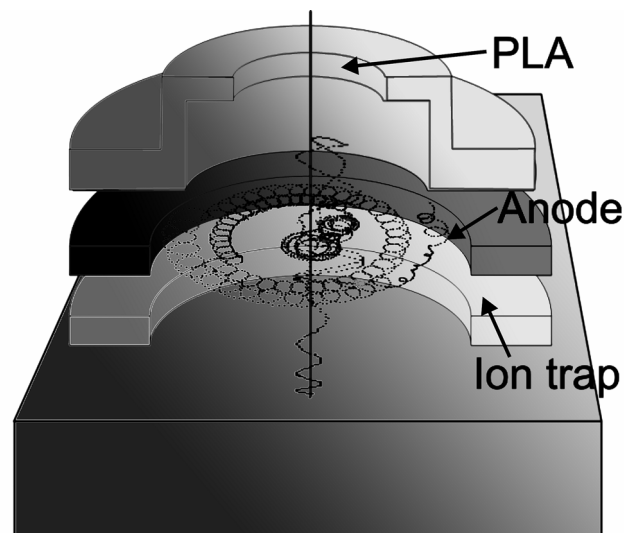


Figure 3.11 Schematic drawing of the Helix detector (Knowles et al., 2005)

By using a well thought out electromagnetic field between the specimen and the polepiece it is possible to achieve a very long amplification distance at very short working distances. The path of one secondary electron from the specimen to the anode is shown in figure 3.11.

The electrons travel about three orders of magnitude faster than the heavier ions, which become noticeable in ion current measurements (Morgan and Phillips, 2001) and in secondary electron images acquired at different scan speeds (Toth and Phillips, 2000). Reason therefore is the build up of a space charge between the specimen and the detector, which influences the electric amplification field.

Besides the space charge, secondary electron ion recombination at the sample surface influences and complicates the image interpretation.

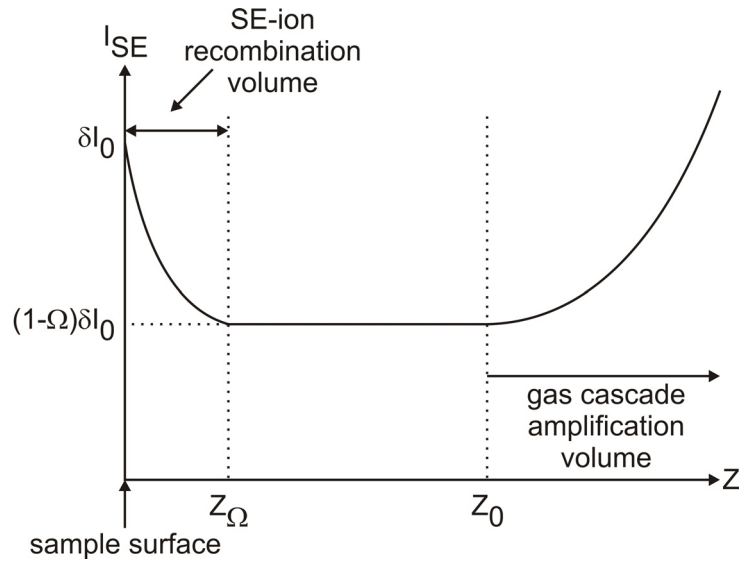


Figure 3.12 SE signal as a function of distance from the surface (Thiel and Toth, 2005)

The probability that SE-ion recombination takes place is depending on the mean recombination probability Ω . By accelerating the secondary electrons this probability decreases and therefore recombination takes place only in the vicinity of the surface. After further acceleration the electrons have enough kinetic energy to ionise the imaging gas and the amplification process starts (figure 3.12). This whole process is time depending again and changes in contrasts can be observed (Toth et al., 2002).

The positive ions are not only complicating contrast mechanism, they can be the origin of new ones. Griffin et. al. (2000) presented the so called charge contrast. Thereby the growth defect zones of gibbsite can be visualised depending on scan speed and chamber pressure.

All these positive and negative effects are a result of the big amount of gas ions. Therefore, investigations with devices which decrease the ion current were realised (Thiel, 2003). The ion gap included in the Helix detector can be biased negatively to attract the imaging gas ions (see figure 3.11). This bias can be controlled by the user interface and the microscopist him/herself chooses the amount of ions hitting the specimen.

3.2.3 Charge Suppression

By investigating insulating materials, charging artefacts play an important role in scanning electron microscopy. The charging of the sample is a result of the bombardment with charge carriers. Under normal acceleration voltage conditions an insulating specimen charges negatively because more primary electrons are captured inside the sample than secondary and backscatter electrons could leave.

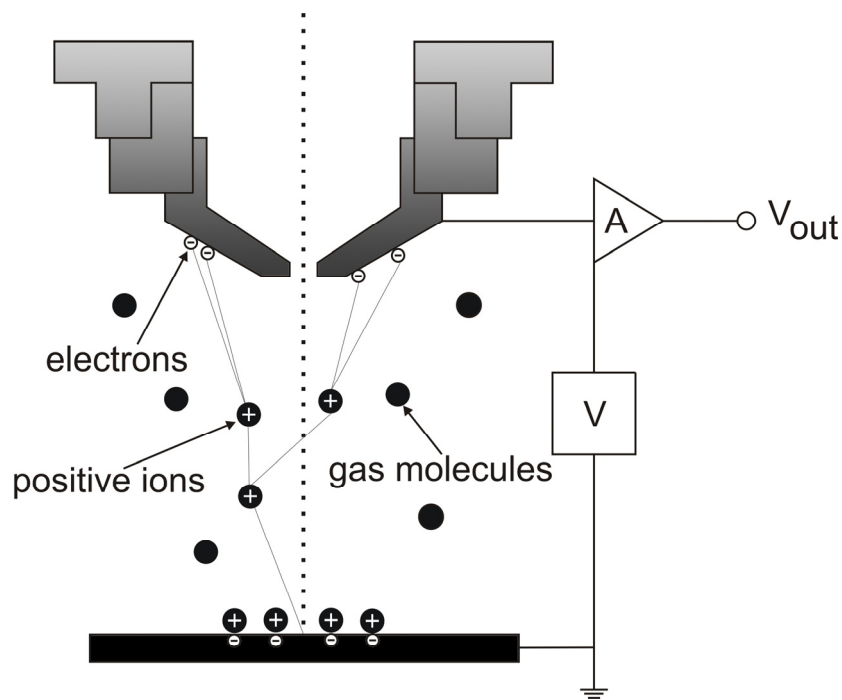


Figure 3.13 Charge suppression inside an ESEM

An outstanding benefit of the ESEM is the charge suppression (figure 3.13). As a by-product of the secondary electron gas amplification, positive gas ions are generated. These ions are deflected by the electric field of the secondary electron detector and attracted by the negatively charged sample. On the surface they recombine and suppress charging of the insulating sample.

3.2.4 X-ray spectrometry in Gaseous Environment

The scattering of the primary beam electrons complicates X-ray spectrometry in a fundamental way. It is known that primary beam electrons hit the specimen not only in the focused probe point and therefore characteristic X-rays which originate also from the surrounding but not interesting area are detected (figure 3.14).

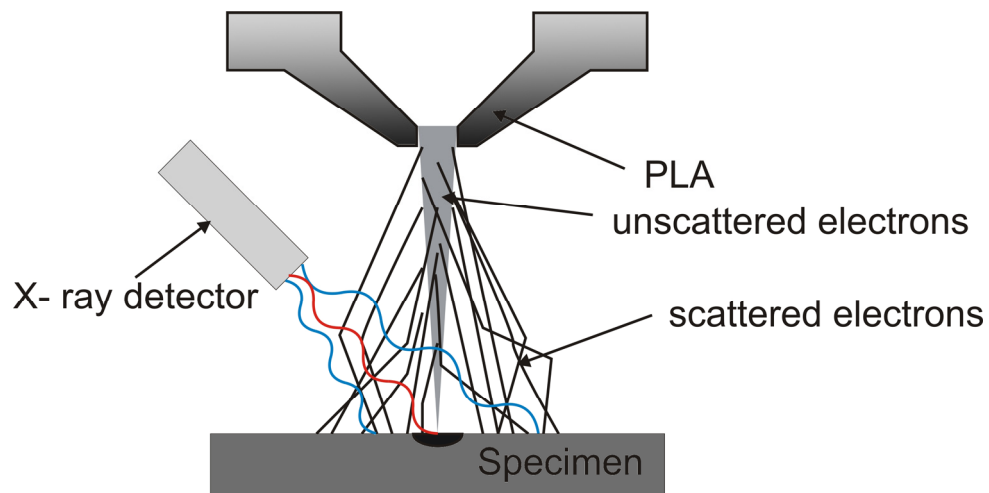


Figure 3.14 X-ray spectrometry in an ESEM

Therefore, detailed knowledge of the beam profile is necessary to correct the influence of the scattered electrons on the spectrums. In order to demonstrate this effect a sample was investigated under low vacuum and high vacuum conditions (see figure 3.15). The beam was focused on a segregation area in steel and the spectra were compared. The iron signal in figure 3.16 is strongly reduced in comparison with the high vacuum spectrum. Therefore, additional correction procedures are necessary for a correct quantification of such spectra.

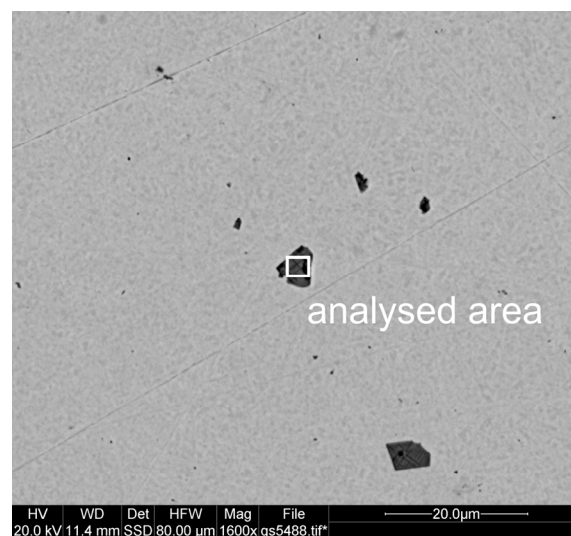


Figure 3.15 BSE image of the analysed area (segregation in steel sample)

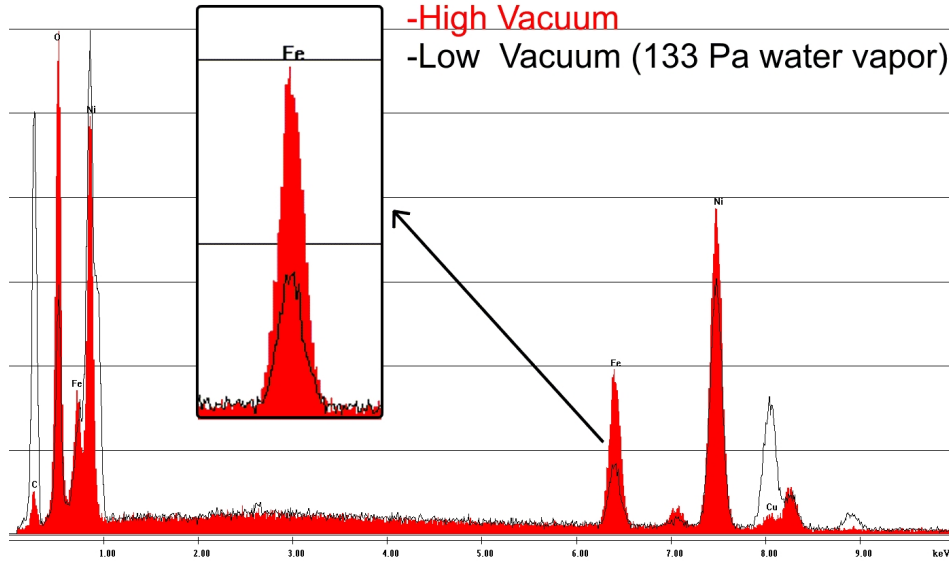


Figure 3.16 Comparison of EDX spectra (low vacuum and high vacuum)

There are two basic different correction procedures, the beam stop procedure and the pressure variation procedure. The beam stop procedure is not very practical because a micromanipulator needle of a well known element is needed, and therefore this method is not presented in this thesis (Mansfield, 2000). The pressure variation method needs no additional equipment and is therefore much more practically orientated (Doehen, 1997; Gauvin, 1999). By using formula 3.2 and f_p as the fraction of the beam which is not scattered, I_p as the intensity contributed to the unscattered beam and I_M as the intensity contributed to the scattered beam, the pressure variation method can be described in formula 3.5, 3.6 and 3.7.

$$I = I_0 \cdot e^{-m} = I_0 \cdot f_p \quad 3.5$$

The measured intensity under specific pressure conditions is given by:

$$I = f_p \cdot I_p + (1 - f_p) \cdot I_M \quad 3.6$$

rewriting gives:

$$I = (I_p - I_M) \cdot f_p + I_M \quad 3.7$$

The intensity is a linear function of the fraction of unscattered electrons. Knowledge of the fraction of unscattered electrons and the intensities of at least two different pressure values are necessary for the correction procedure.

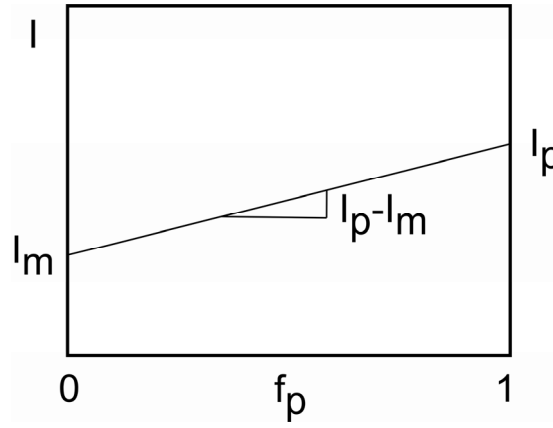


Figure 3.17 X-ray intensity as a function of the fraction of unscattered electrons f_p
(Gauvin, 1999)

First of all the intensities must be measured for two different chamber pressure conditions (I_1 and I_2). By calculating the fraction of unscattered electrons for both pressures (f_{p1} and f_{p2}), the linear dependency can be used to calculate the slope of this graph (see figure 3.17). With this slope the intensity I_p can be calculated at $f_p=1$ (without scattering).

$$I_p = \frac{I_1 \cdot (1 - f_{p2}) - I_2 \cdot (1 - f_{p1})}{f_{p1} - f_{p2}} \quad 3.8$$

The main problem with this correction procedure is the correct calculation of the fraction of unscattered electrons. Knowledge of the total scattering cross-section of the imaging gas (see chapter 6 and chapter 7) and of the beam gas path length (see chapter 8) are necessary to calculate this value in a correct way.

4 Elastic and Inelastic Electron Scattering in Solids

The electron specimen interactions can be divided in elastic and inelastic interactions. Inelastic interactions between an electron and an atom lead to an energy transfer from the electron to the atomic jellium. A deflection of the electron trajectories and secondary particles like secondary electrons, auger electrons, characteristic X-rays caused by inner shell ionisations and continuous Bremsstrahlung are some of the main results of such interactions. By elastic scattering the energy of the electrons will be conserved but the trajectories will be changed which strongly influences the backscattering of electrons.

Both scattering effects influence the trajectories of an accelerated electron inside a solid. As the electrons follow a zig-zag path through the specimen, a part of the electrons leave the specimen as backscattered electrons another part decelerates by inelastic collisions and rest inside the solid (figure 4.1).

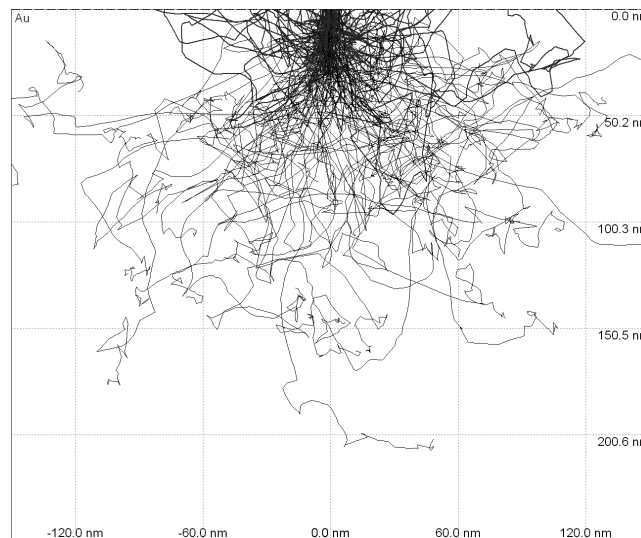


Figure 4.1 The zig-zag trajectories of 10 keV electrons in a gold sample caused by elastic and inelastic scattering (calculated with Casino 2.42)

4.1 Elastic Scattering

Backscattering and diffusion of electrons in solids are strongly influenced by elastic scattering, because the elastic scattering is mainly responsible for large angle scattering. In this context the concept of the differential cross-section is of main importance. This differential cross-section is proportional to the probability that an interaction between an electron and an atom results in a scattering event into an infinitesimal solid angle.

4.1.1 Rutherford Scattering Theory at an Unscreened Nucleus

The simplest way to describe the elastic scattering in a solid was presented by Rutherford (1911) who explained the scattering of alpha particles in a thin metal foil. Based on this classical mechanics theory the scattering of electrons at atoms can be described as follows. The nucleus is assumed to stay at rest because of the enormous mass in comparison with the mass of the electrons and the screening of the Coulomb field of the nucleus by the shell electrons is neglected (figure 4.2). These assumptions simplify the problem dramatically and the change in electron trajectory can be described in a comparable way as the deflection of an asteroid passing the gravity field of a planet.

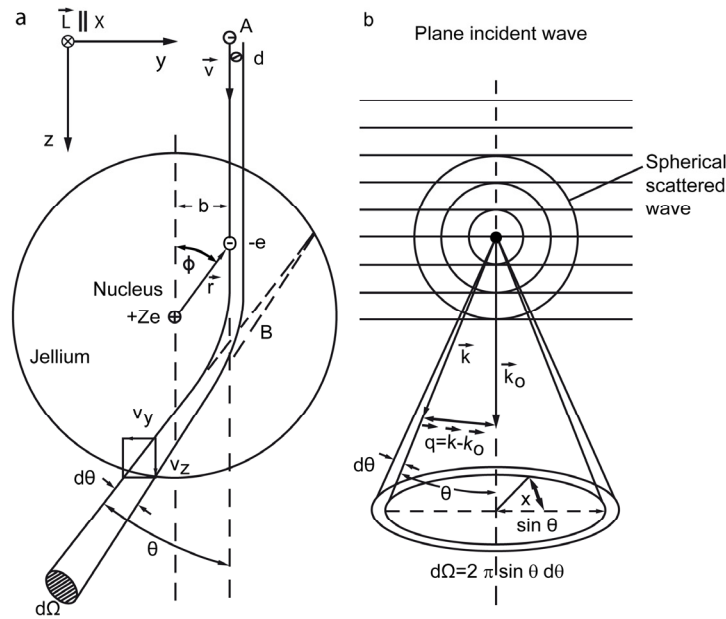


Figure 4.2 Description of the presented problem (Reimer, 1998)

Using Newton's equation and the attractive Coulomb force of the nucleus on the electron gives formula 4.1:

$$\frac{d\vec{p}}{dt} = m_e \cdot \ddot{\vec{r}} = -\frac{e^2 \cdot Z \cdot \vec{r}}{4 \cdot \pi \cdot \epsilon_0 \cdot r^3} \quad 4.1$$

where $\mathbf{p}$ is the impulse vector of the electron, m_e is the electron mass, Z is the atomic number, $\mathbf{r}$ is the vector electron nucleus and r is the distance electron nucleus.

Under consideration of the conservation of energy the differential cross-section can be calculated (formula 4.2):

$$\frac{d\sigma_{Ru}}{d\Omega} = \frac{e^4 \cdot Z^2}{4 \cdot (4 \cdot \pi \cdot e_0)^2 \cdot m^2 \cdot v^4} \cdot \frac{1}{\sin^4\left(\frac{\theta}{2}\right)} \quad 4.2$$

where $d\sigma_{Ru}/d\Omega$ is the differential cross-section at an unscreened nucleus and θ the scattering angle.

4.1.2 Rutherford Scattering Theory at a Screened Nucleus

The singularity at $\theta \rightarrow 0$ is a result of the inconsideration of the screening of the Coulomb field (see figure 4.3). The long range Coulomb force is not screened and therefore a scattering process which results into a deflection of 0 degrees is not described. Only when considering a screening of the Coulomb field by the shell electrons this singularity disappears.

However, the screening of the nucleus is described correctly by means of quantum mechanics. Because of the elastic scattering no energy transfer into higher atomic states occurs and the screening potential can be approximated with the Wentzel model with one exponential term (formula 4.3 and 4.4):

$$V(r) = -\frac{e^2 \cdot Z}{4 \cdot \pi \cdot e_0 \cdot r} \cdot e^{-\frac{r}{R}} \quad 4.3$$

with

$$R = a_H \cdot Z^{-\frac{1}{3}} \quad 4.4$$

where $V(r)$ is the Wentzel potential, R the screening radius and a_H the Bohr radius.

Especially the correct choice of the screening radius is very important. This value describes the effective distance over which the Coulomb force of the nucleus is active under consideration of the screening of the orbiting electrons.

With these assumptions the screening radius can be described by the screening angle θ_0 and the differential cross-section of a screened nucleus $d\sigma_{Rs}/d\Omega$ is given by formula 4.5:

$$\text{for } \sin\left(\frac{\theta}{2}\right) \cong \left(\frac{\theta}{2}\right)$$

$$\frac{d\sigma_{Rs}}{d\Omega} = \frac{e^4 \cdot Z^2}{4 \cdot (4 \cdot \pi \cdot \varepsilon_0)^2 \cdot m^2 \cdot v^4} \cdot \frac{1}{\left[\sin^2\left(\frac{\theta}{2}\right) + \sin^2\left(\frac{\theta_0}{2}\right)\right]^2} \cong \frac{e^4 \cdot Z^2}{4 \cdot \pi^2 \cdot \varepsilon_0^2 \cdot m^2 \cdot v^4} \cdot \frac{1}{(\theta^2 + \theta_0^2)^2} \quad 4.5$$

Considering only small angle scattering ($\theta \ll \theta_0$) and setting the scattering angle to zero (unscreened nucleus),

$$\sin^2\left(\frac{\theta}{2}\right) \cong \sin^4\left(\frac{\theta}{4}\right)$$

the screened differential cross-section simplifies to the unscreened formula 4.2.

The presented results are considering a free neutral atom, which is rarely found in reality. Because of the dense packing in solids the potentials overlap, which influences scattering processes.

The muffin-tin model, the WKB (Wentzel, Kramer, and Brillouin) or the partial wave method can be used to describe this overlapping in an accurate way. Formula 4.6 describes the screening potential in the muffin-tin model:

$$\begin{aligned} V_{eff} &= V(r) + V(2 \cdot b - r) - 2 \cdot V(b) & \text{for } r \leq b \\ V_{eff} &= 0 & \text{for } r > b \end{aligned} \quad 4.6$$

where V_{eff} is the effective screening potential and $2 \cdot b$ is the distance between neighbouring atoms.

4.1.3 Mott Scattering Theory

In quantum mechanics a scattering of a plane wave field can be described by a superposition of a plane wave field and a spherical wave. Therefore, the atom causes a phase shift of the incident plane after the scattering process and the differential cross-section can be calculated by determining the scattering amplitude $f(\theta)$ of the spherical wave (Mott and Massey, 1965) (formula 4.7).

$$\frac{d\sigma}{d\Omega} = |f(\theta)|^2 \quad 4.7$$

The only way to describe the elastic scattering of electrons in solids in a correct way is by using the Schrödinger or Pauli-Dirac equations with a screened Coulomb potential. An additional model must be used to consider the dense packing of the atoms, hence the overlapping of the potentials.

In contrast to the Rutherford scattering model no analytical expression for the differential cross-section can be formed, numerical methods are necessary to calculate the phase shift η_l and η_{l-1} of the scattered spherical wave. This is most likely the reason for the common use of

the Rutherford model in scanning electron microscopy, although this model has several weaknesses and simplifications which do not represent reality.

By using the Pauli-Dirac equation the two different spin directions must be solved separately and in comparison with the Rutherford model a second amplitude, the spin flip amplitude $g(\theta, \chi)$ occurs. This amplitude considers that the second spin direction may also be present after the interaction.

For the +z spin direction infinite series of Legendre polynomials (formula 4.8) describe the two amplitudes (Czyzewski et al., 1990):

$$\begin{aligned} f_1(\theta) &= \frac{1}{2 \cdot i \cdot k} \sum_{l=0}^{\infty} \{ (l+1) \cdot [\exp(2 \cdot i \cdot \eta_l) - 1] + l \cdot [\exp(2 \cdot i \cdot \eta_{-l-1}) - 1] \} \cdot P_l(\cos(\theta)) \\ g_1(\theta, \chi) &= \frac{1}{2 \cdot i \cdot k} \sum_{l=0}^{\infty} -\exp(2 \cdot i \cdot \eta_l) + \exp(2 \cdot i \cdot \eta_{-l-1}) \cdot P_l^1(\cos(\theta)) \cdot e^{i \cdot \chi} \end{aligned} \quad 4.8$$

and for the -z spin direction:

$$\begin{aligned} f_2 &= f_1 = f(\theta) \\ g_2 &= -g_1 \cdot e^{-2 \cdot i \cdot \chi} = -g(\theta) \cdot e^{-i \cdot \chi} \end{aligned} \quad 4.9$$

where η is the phase shift, P_l the ordinary Legendre polynomial, P_l^1 the associated Legendre polynomial and χ the azimuth angle.

In a scanning electron microscope the electron beam is unpolarised and the dependency on the azimuth angle χ disappears. The Mott differential cross-section $d\sigma_M/d\Omega$ of an unpolarised electron beam with superposition of both spin directions is given by formula 4.10:

$$\frac{d\sigma_M}{d\Omega} = |f|^2 + |g|^2 \quad 4.10$$

In the logarithmic polar plot (see figure 4.3) the main differences in results of Rutherford and Mott scattering theory can clearly be seen. No singularity at $\theta \rightarrow 0$ occurs using the Mott scattering theory because the screening effects are considered. There are differences in high angle scattering, especially at high atomic numbers. The Rutherford scattering theory is based on classical mechanics and therefore the spin orbit coupling of electrons can not be considered, an oversimplification which results in strong deviations especially at high atomic numbers and low electron energies.

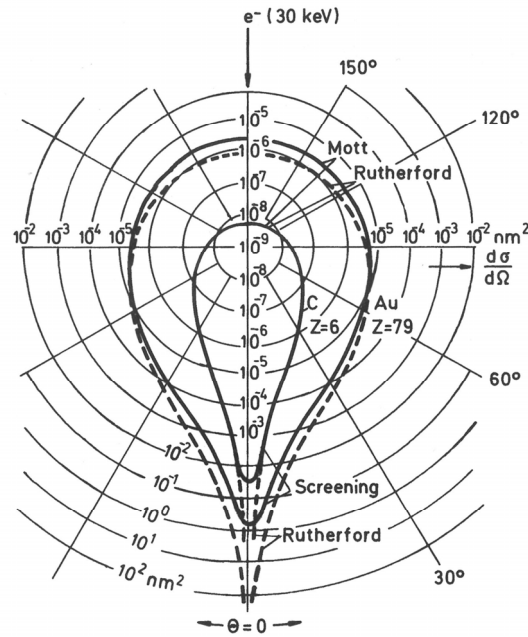


Figure 4.3 Logarithmic polar plot of the differential cross-section of single carbon and gold atoms (Rutherford: ---; Mott: -) (Reimer, 1998)

The complex curve progression of the differential cross-section based on Mott theory, especially at high scattering angles and low electron energies are shown in figure 4.4.

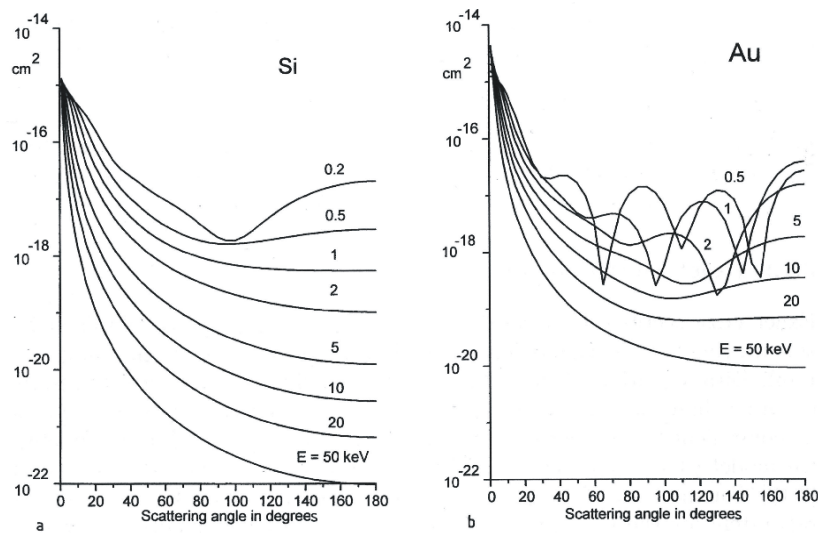


Figure 4.4 Differential cross-section of silicon and gold for different electron energies (Reimer, 1998)

Because of the complex mathematical calculations and the necessity of numerical methods to calculate Mott differential cross-sections, databases for different electron energies and different atomic numbers are available (Czyzewski et al. 1990; Joy et al., 2001). In order to

visualise the content a Matlab program was developed (the code can be seen in chapter 11.5.1). The results for silicon and lead can be seen in figures 3.5 to 3.8. Lead has a more complex orbital structure which becomes noticeable in the differential cross-section. The angular dependency strongly increases with decreasing electron energy and minima and maxima appear.

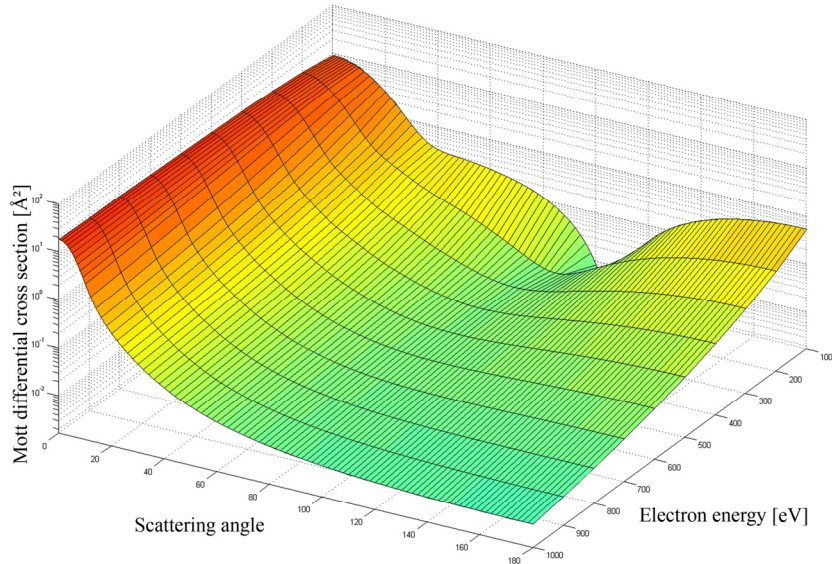


Figure 4.5 Mott differential cross-section of silicon (100 eV-1 keV)
(calculated with the program MOTT (see chapter 11.5.1))

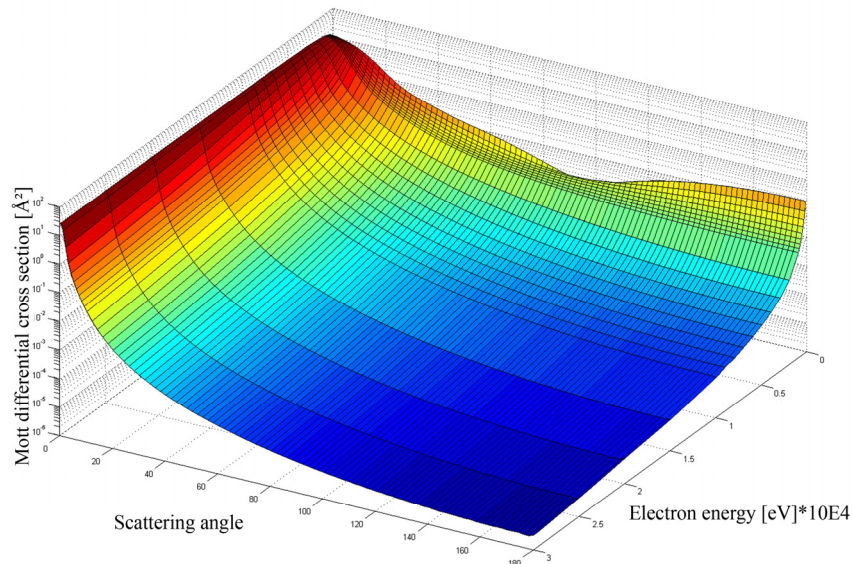


Figure 4.6 Mott differential cross-section of silicon (100 eV-30 keV)
(calculated with the program MOTT (see chapter 11.5.1))

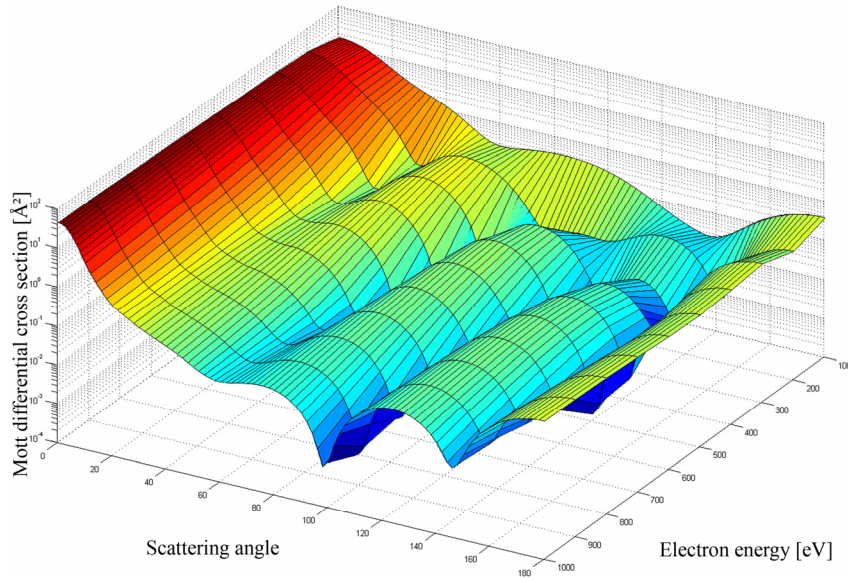


Figure 4.7 Mott differential cross-section of lead (100 eV-1 keV)
(calculated with the program MOTT (see chapter 11.5.1))

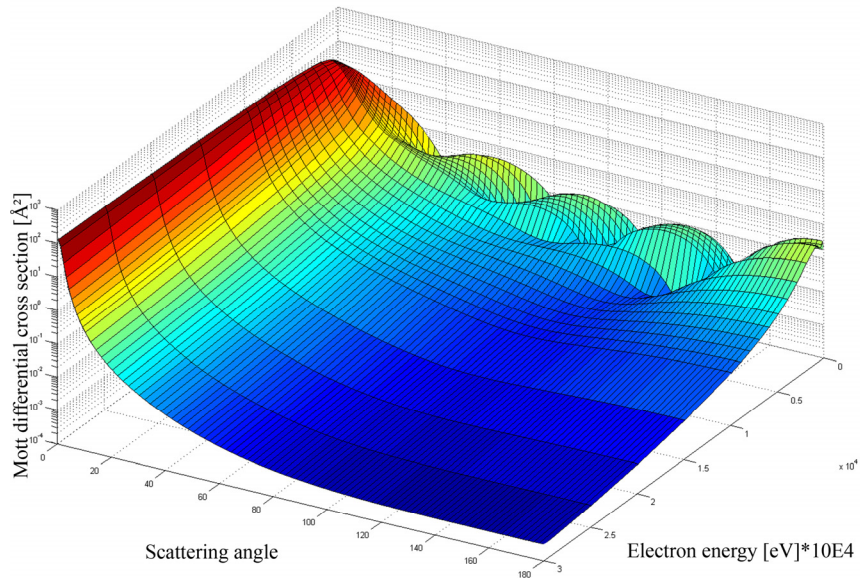


Figure 4.8 Mott differential cross-section of lead (100 eV-30 keV)
(calculated with the program MOTT (see chapter 11.5.1))

The ratio between Mott and Rutherford differential cross-section $r(\theta)$ is used to clarify the differences in results (formula 4.11). It can be seen, that the two models do not vary for low atomic numbers and low scattering angles, but there are significant differences for high atomic numbers, complex orbital structures and high angle scattering (see figure 4.9 and figure 4.10)

$$r(\theta) = \frac{d\sigma_M}{d\Omega} \bigg/ \frac{d\sigma_R}{d\Omega}$$

4.11

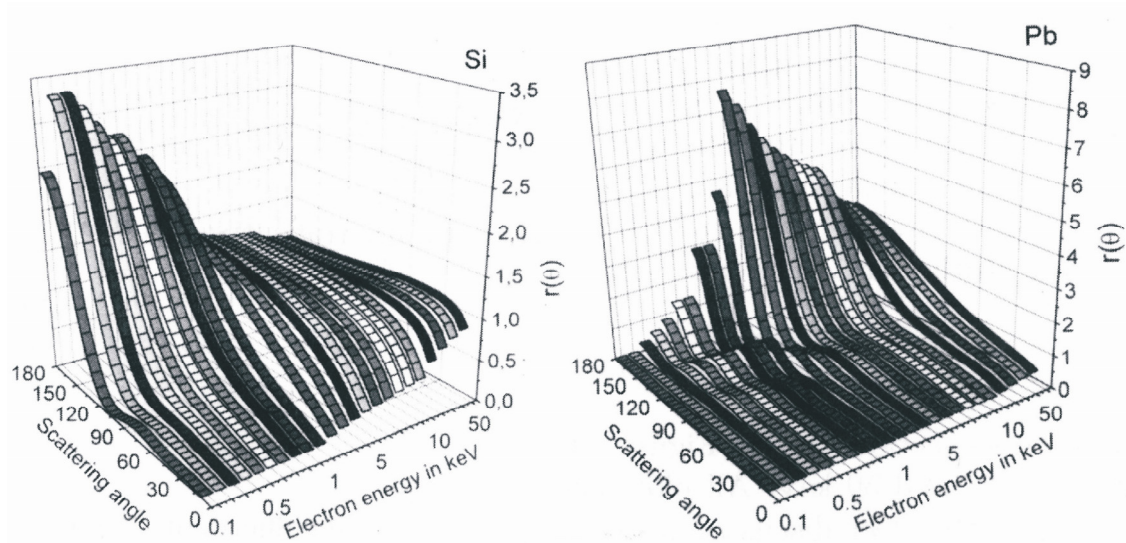


Figure 4.9 Ratio Mott to Rutherford differential cross-section for different electron energies (Reimer, 1998)

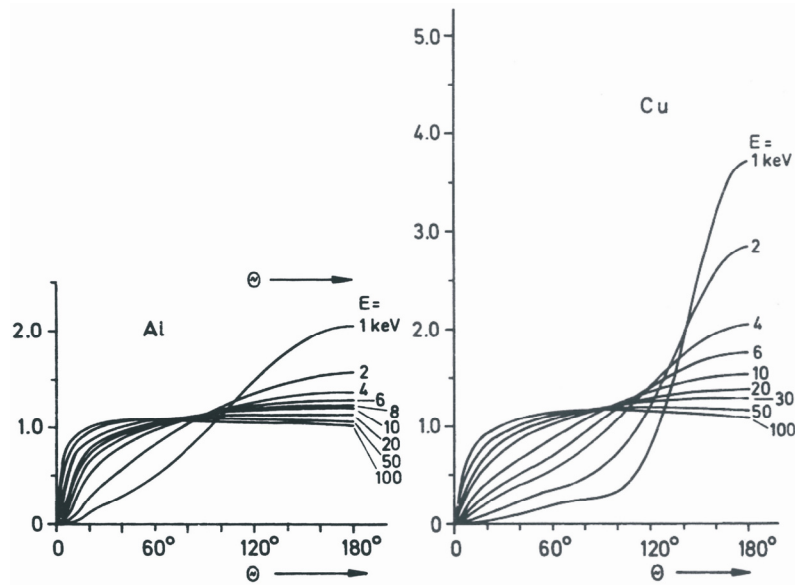


Figure 4.10 Ratio of Mott to Rutherford (unscreened) cross-section for aluminium and copper (Reimer, 1993)

Good approaches in addition to databases are empirical forms for the total elastic scattering cross-sections (Browning et al., 1991; Browning et al., 1994; Browning et al., 1995) (formula 4.12). This empirical formula is not valid as stand-alone cross-section; it should be used

together with Monte Carlo simulations. “The two reasons for this restriction are, first, the final fitting process was dependent on both parts of the cross-section, and, second, the Monte Carlo calculations are simulating a multiple scattering phenomenon which smoothes out quantum effects.” (Browning et al., 1994)

$$\sigma_T = \frac{3 \cdot 10^{-18} \cdot Z^{1.7}}{E + 0.005 \cdot Z^{1.7} \cdot E^{0.5} + \frac{0.0007 \cdot Z^2}{E^{0.5}}} \quad 4.12$$

4.2 Inelastic Scattering

The inelastic interactions between accelerated electrons and solids lead to an energy transfer and a deflection of the electron trajectories. The energy transfer takes place in one or several of the following interaction processes:

- excitation of a shell electron to an excited state in the same band (intraband transition) or to another band (interband transition)
- formation of electron hole pairs in semiconductors
- excitation of plasmons, which are collective longitudinal charge density waves of the valence or conductive band electrons
- excitation of vibrations in a molecule or phonones in a solid
- ionisation of inner shells with and excitation of the electron to a state above the Fermi level
- deceleration of electrons by the Coulomb potential exciting continuous Bremsstrahlung

For the calculation of the inelastic differential cross-section from the ground state to an excited state m , a quantum mechanically approach is necessary. The electron impact leads to an excitation and a change into a higher state ($0 \rightarrow m$).

By summing up over all possible excitation states m the inelastic differential cross-section $d\sigma_{inel}/d\Omega$ is given by formula 4.13:

$$\frac{d\sigma_{inel}}{d\Omega} = \frac{4 \cdot e^4 \cdot Z}{(4 \cdot \pi \cdot e_0)^2 \cdot m_0^2 \cdot v^4} \cdot \frac{1 - \left(\frac{1}{1 + (\theta^2 + \theta_E^2)} \right)^2}{(\theta^2 + \theta_E^2)^2} \quad 4.13$$

with

$$\theta_E = \frac{J_{mean}}{4 \cdot E}$$

where J_{mean} is the mean ionisation potential.

A comparison between the elastic differential cross-section and the inelastic cross-section clearly shows that inelastic scattering concentrates within much smaller angles and therefore inelastic scattering events do not strongly influence the backscattering of electrons. For very large scattering the ratio between inelastic and elastic scattering cross-section is given by formula 4.14:

$$\frac{\frac{d\sigma_{inel}}{d\Omega}}{\frac{d\sigma_{el}}{d\Omega}} = \frac{1}{Z} \quad \text{for } \theta \gg \theta_0 \gg \theta_E \quad 4.14$$

5 Signal Formation in the SEM

Elastic and inelastic scattering of accelerated electrons in solids lead to zig-zag trajectories of the electrons. The elastic scattering is mainly responsible for large angle scattering and hence for electron backscattering. Inelastic scattering leads to a small deflection of the electron path and to an energy transfer to the solid. The detection of the results of these interactions contains a lot of information about the sample like topography, elemental composition, crystallography and electrical potential (figure 5.1). Caused by the sample penetration the focused electron probe diffuses, which decreases the lateral resolution. This penetration depth is depending on the electron energy, the average atomic number of the investigated material and on the crystalline structure.

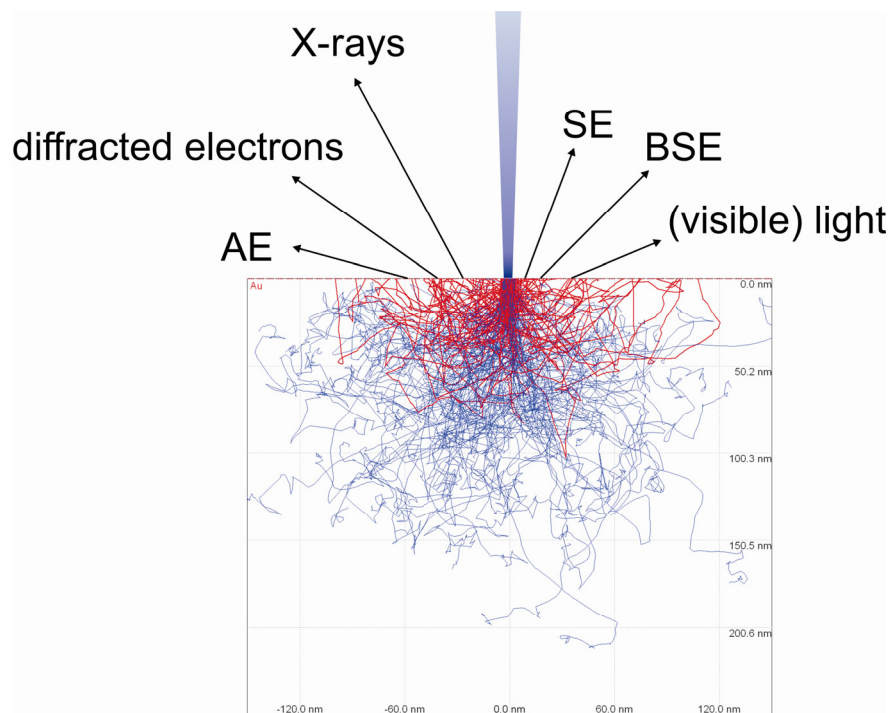


Figure 5.1 The interaction of beam electrons and sample (calculated with Casino 2.42)

Detecting characteristic X-ray using energy dispersive X-ray spectrometer (EDXS) and/or wave length dispersive X-ray spectrometer (WDXS) gives information about the chemical composition of the specimen. The Auger electrons can be detected by an Auger electron spectrometer (AES) which gives very surface sensitive compositional information of the specimen. The emission of visible light can be used to characterise the sample by cathodoluminescence spectrometry (CL). Detecting the diffracted electrons gives information about the crystalline structure of the specimen which is used in low electron energy diffraction (LEED) and reflection high energy electron diffraction (RHEED).

The secondary electron yield is mainly depending on the surface tilt and therefore the specimen topography can be seen in SE images. In scanning electron microscopy the dependency of the elastic scattering on the average atomic number is responsible for the material contrast in backscatter electron images.

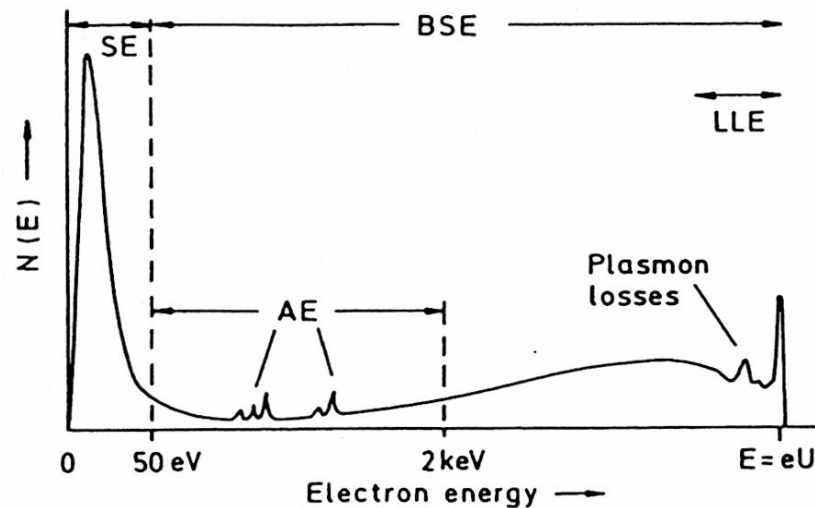


Figure 5.2 Energy distribution of electrons emitted from a target (LLE-Low Loss Electrons; AE-Auger Electrons) (Reimer, 1998)

In scanning electron microscopy the most commonly used signals are the characteristic X-rays and the secondary and backscattered electrons. The classification of the different emitted electrons is shown in figure 5.2. The secondary electrons have per definition an energy between 0 and 50 eV, the backscattered electrons an energy >50 eV. According to their different energies, they have completely different escape depths (figure 5.3).

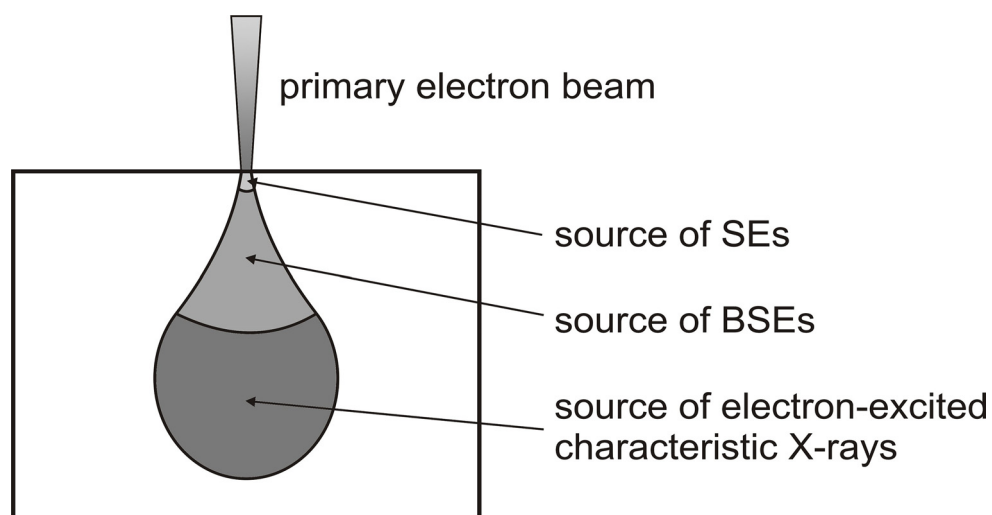


Figure 5.3 Schematic drawing of the different volumes of interaction

5.1 Secondary Electrons

The secondary electron yield δ is defined as number of secondary electron per incident beam electron (formula 5.1):

$$\delta = \frac{n_{SE}}{n_B} = \frac{i_{SE}}{I} \quad 5.1$$

where n_{SE} is the number of secondary electrons, n_B the number of primary beam electrons and i_{SE} is the secondary electron current.

The secondary electrons are defined purely on the basis of their kinetic energy (0 – 50 eV) and every electron with energy more than 50 eV is defined as backscattered electron. They are produced along the beam trajectories, but the low energy of secondary electrons is responsible for the small exit depth. For metals the depth is of the order of 0.5 – 1.5 nm, for carbon about 10 nm and about 10–20 nm for insulators (Reimer, 1998).

Therefore, imaging with SE is extremely surface sensitive which is the origin of the good lateral resolution. The SE yield at normal incident $\delta(0,Z)$ strongly increases with surface tilt, which is responsible for topographic contrast. The secondary electron dependency on the surface tilt angle ϕ and the emission angle ζ relative to the surface normal is given by formula 5.2.

$$\frac{d\delta}{d\Omega} = \frac{\delta(0,Z)}{\pi} \cdot \sec(\phi) \cdot \cos(\zeta) \quad 5.2$$

By integrating over $d\Omega = 2 \cdot \pi \cdot \sin(\zeta) \cdot d\zeta$, the total secondary electron yield can be calculated. The Bethe stopping power expression can be used to calculate the secondary electron yield (formula 5.3):

$$\delta = \frac{0.5}{\varepsilon} \cdot \left| \frac{dE_m}{ds} \right| \cdot \sec(\phi) \cdot \int_0^\infty \exp\left(\frac{-z}{t_{SE}}\right) dz \propto E^{-0.8} \cdot \sec(\phi) \cdot t_{SE} \quad 5.3$$

where t_{SE} is the escape depth, dE_m is the mean energy loss and ε is the mean energy loss to produce one secondary electron.

5.1.1 Detection of Secondary Electrons

The commonly used secondary electron detector is the Everhard Thornley detector. The positively biased grid collects the low energy secondary electrons in front of a scintillator. A photomultiplier amplifies the signal (see figure 5.4).

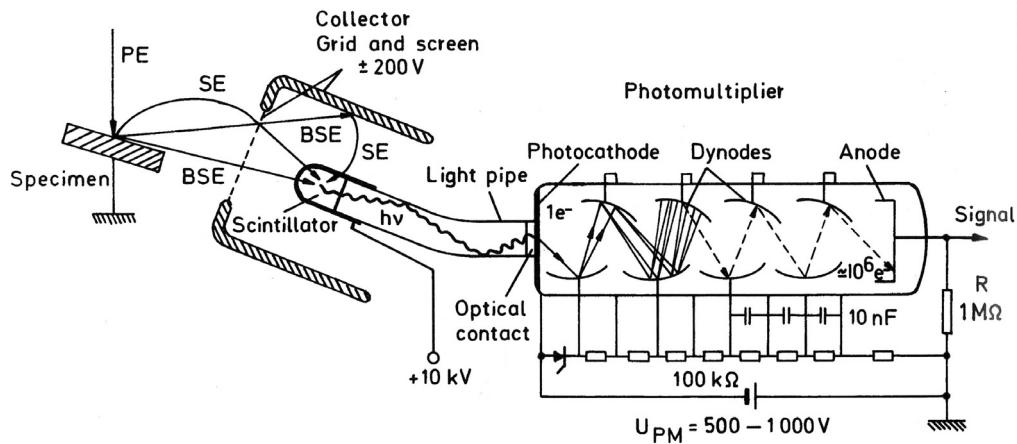


Figure 5.4 Everhard Thornley secondary electron detector (Reimer, 1998)

Secondary electrons in a SEM can be divided, according to their origins (figure 5.5). SEs produced by the primary electron beam in the vicinity of the focused electron probe are called SE1s. By leaving the specimen, backscatter electrons cross the surface of the specimen and therefore they are generating secondary electrons within the escape depth of SEs, these are the SE2s. Secondary electrons which are generated by backscattered electrons hitting the polepiece or the specimen chamber wall are called SE3s. Primary beam electrons which hit an aperture in the electron column generate SE4s. Therefore, detecting SE1 leads to the best lateral resolution and SE3 and SE4 decrease the signal to noise ratio. The Everhard Thornley detector, which is the standard detector in conventional SEMs, has the following distribution using a gold specimen: SE1 9 %, SE2 28 %, SE3 61 % and SE4 2 % (Goldstein et al., 2003).

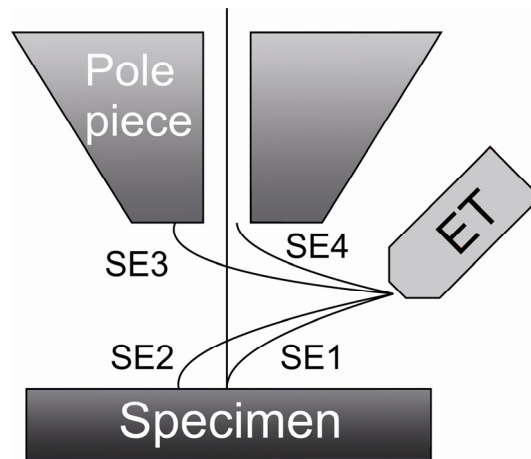


Figure 5.5 Schema of different secondary electron types

For low voltage SEM, special detector designs are necessary to guarantee a good signal to noise ratio and a good discrimination between secondary and backscatter electrons.

By using an electrostatic objective lens system, an in-lens detection of secondary and backscatter electrons is possible (figure 5.6). The SEs are accelerated on axis by a positively biased electrode. Caused by the electric field, electrons with different energies follow different paths, which enable the discrimination between secondary and backscattered electrons. The detector efficiency increases with decreasing working distance, which is an advantage because of the decrease in spherical and chromatic aberration.

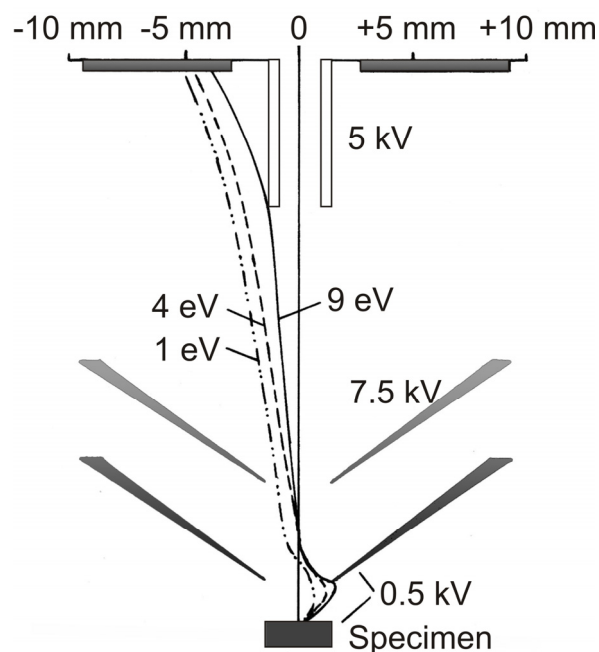


Figure 5.6 Electrostatic detector objective lens with in-lens SE detection (Reimer, 1993)

5.2 Backscattered Electrons

Elastic scattering of electrons in solids are mainly responsible for large angle scattering and therefore for electron backscattering. Because of the complexity of electron diffusion in solids there exists no exact theory of electron backscattering, a fact which clarifies the necessity of Monte Carlo simulations. The differences in backscatter coefficients (η_1 and η_2) depend on the specimen composition. This is responsible for the contrast C in backscatter images (formula 5.4).

$$C = \frac{\eta_1 - \eta_2}{\eta_1} \quad 5.4$$

The angular distribution of backscattered electrons $d\eta/d\Omega$ can be approximated with the Lambert's cosine law (formula 5.5 and figure 5.7):

$$\frac{d\eta}{d\Omega} = \frac{\eta}{\pi} \cdot \cos(\zeta) \quad 5.5$$

where ζ is the angle between surface normal and backscatter emission.

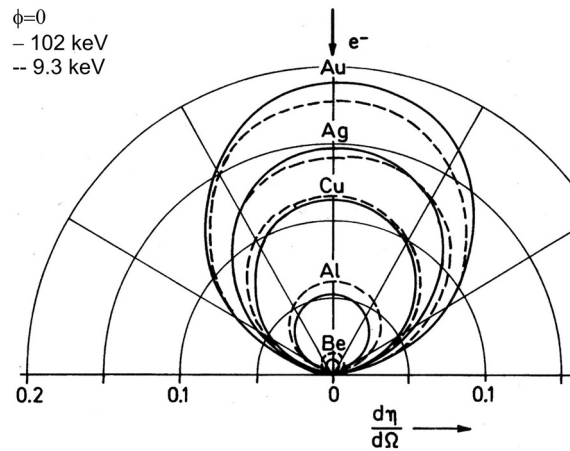


Figure 5.7 Angular dependencies of backscattered electrons (Reimer, 1998)

The spectrum of emitted electrons in figure 5.2 shows the wide range of electron energies, which are attributed to BSEs. The no-loss backscattered electrons have the same energy as the primary beam electrons and therefore their escape depth is extremely small. “The no-loss electrons will be scattered from layer of the order of the mean free path length for Plasmon losses on thickness, which increases as the electron energy” (Reimer, 1998).

The escape depth Δx of low loss backscattered electrons can be estimated with the Bethe stopping power:

$$\Delta x \propto \frac{A \cdot E \cdot dE_m}{Z} \quad 5.6$$

where A is the atomic weight.

5.2.1 Detection of Backscattered Electrons

The typical backscatter detector is a semiconductor detector, which is mounted below the polepiece (figure 5.8). A backscattered electron hitting the semiconductor detector produces a mean number of electron-hole pairs. By separating and collecting these charge carriers the backscatter signal can be converted into an external charge collection current. The mean number of electron hole pairs n_{mean} is direct proportional to the electron energy E and indirect proportional to the mean energy per excitation E_{i_mean} (for silicon 3.6 eV) (formula 5.7).

$$n_{\text{mean}} = \frac{E}{E_{i_ \text{mean}}} \quad 5.7$$

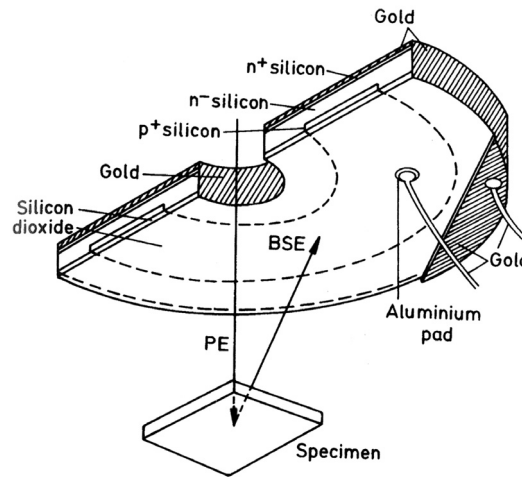


Figure 5.8 Typical semiconductor detector (below the polepiece) (Reimer, 1998)

Backscatter electrons also can be detected using the ET detector. The grid of the detector is negatively biased, which retards the SE and only BSE are detected. Nevertheless the solid angle of detection is relatively small (10^{-2} rad) which minimises the signal to noise ratio.

A scintillation detector can be used in a direct way without additional acceleration of the electrons. In conventional scanning electron microscopy the BSEs have enough energy to produce a large amount of light quanta which are again amplified in a photomultiplier. By using a magnetic-electrostatic compound objective lens system (figure 2.6 and figure 5.6) low voltage backscatter imaging is possible. This detection system is presented in more detail in chapter 2.2.2.2 and chapter 9.2.

5.3 Contrast in SEM Images

The contrast between two points is defined as:

$$C = \frac{S_{\max} - S_{\min}}{S_{\max}} \quad 5.8$$

where C is the Contrast (between 0 and 1), $S_{\max}$ and $S_{\min}$ are the maximum and minimum intensities.

The most useful contrasts in scanning electron microscopy are:

- Topographic contrast:
The secondary electron yield and the backscattered electron coefficient are depending on the surface tilt, which can be used for the visualisation of the surface topography.
- Material contrast:
Electron backscattering depends on the mean atomic number of the specimen, which is the reason for material contrast in BSE images. The material contrast in SE images is caused by the SE2 contribution and by differences in the work function, which influences the SE yield.
- Crystal orientation or channelling contrast:
Different crystal orientations in a polycrystalline specimen influence the electron backscattering which can be used for visualisation.
- Magnetic contrast:
External magnetic fields influence the secondary electron trajectories on their way to the detector (type 1 magnetic contrast) and internal magnetic fields influence the electron backscattering (type 2 magnetic contrast)
- Voltage or potential contrast:
The SE are repelled or attracted by negative or positive potential which can be seen in SE images

The most common contrasts in scanning electron microscopy are the topography contrast in SE images and the material contrast in BSE images.

5.3.1 Topographic Contrast in SE Images

The topographic SE contrast is influenced additionally to the surface tilt by the detection system and the primary electron energy.

A sideways to the polepiece located ET detector receives less signal from planes orientated away, which results in darker areas in the SE image than planes orientated towards the detector. With an in-lens detection system and hence a virtual illumination from the top no shadowing contrast appears.

The electron range and therefore the diffusion of the signal are depending on the primary electron energy. BSEs leaving the specimen at a distance to the electron probe generate SE2s which are responsible for the diffusion contrast. This contrast can be reduced by detection systems which can distinguish between SE1s and SE2s.

“Mass thickness contrast can be obtained, when the number of SE1 produced in a coating film is approximately proportional to the mass thickness the primary electrons have to penetrate” (Reimer, 1998). In real SE images these contrasts appear simultaneously and lead to a superposition of the detected signals (see figure 5.9).

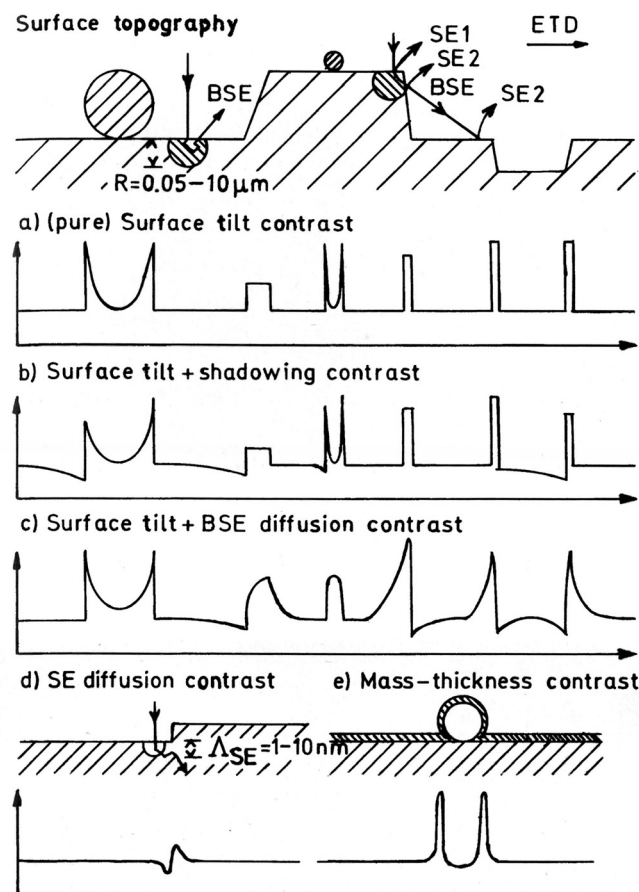


Figure 5.9 Contribution to the topographic contrast (Reimer, 1998)

5.3.2 Material Contrast in BSE Images

In conventional scanning electron microscopy the acceleration voltages are between 5 and 30 keV and the backscatter coefficient rises with increasing atomic number or weight fraction averaged Z for compound targets. This is the so called material or atomic number contrast. The backscatter coefficient depending on the atomic number can be calculated by using empirical formulae. For this energy range one of the most often used formulae is 5.9 (Reimer, 1998):

$$\eta = -0.0254 + 0.016 \cdot Z - 1.86 \cdot 10^{-4} \cdot Z^2 + 8.3 \cdot 10^{-7} \cdot Z^3 \quad 5.9$$

In figure 5.10 a typical example for linear material contrast in backscatter images can be seen. The backscatter coefficient of lead is much higher than of copper and therefore lead appears much brighter.

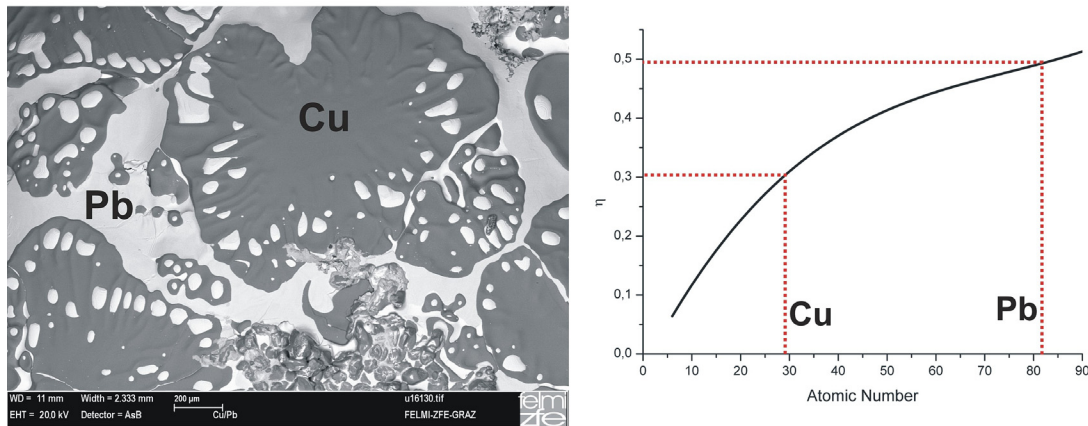


Figure 5.10 BSE image of a lead and copper sample (20 keV)(left); monotonically increasing material contrast (formula 4.9, 20 keV) (right)

The linear material contrast in conventional SEM can be described by the Rutherford scattering theory. The screening of the nucleus by the shell electrons and the spin orbit coupling of the electrons are not considered, which leads to wrong results especially at low electron energies and high scattering angles. Therefore, the Mott scattering theory must be used concerning low voltage electron microscopy. For this theory no analytic expression is known and the use of databases or Monte Carlo simulations are absolutely necessary (Drouin et al., 2007; Czyzewski et al., 1990; Joy et al., 2001).

6 Multiple Scattering of Electrons in Gaseous Environment

The trajectories of accelerated electrons in gaseous environment are strongly influenced by elastic and inelastic collisions. In an ESEM these interactions lead to a broadening of the focused electron probe which is known as skirt effect. Therefore, the focused electron probe loses exponentially electrons along the way through the gaseous environment. Especially, for X-ray spectrometry this new electron distribution is of main importance (see chapter 3.2.4). The main problem is described in figure 6.1. An electron passes the pressure limiting aperture and enters the gaseous environment. After travelling a distance z a scattering event takes place in δz . The electron is scattered into the infinitesimal angle $\delta\theta$ and hits the specimen at the annulus δr .

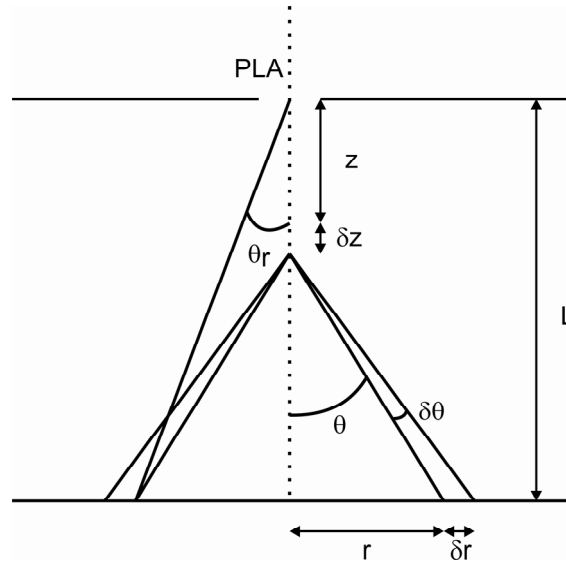


Figure 6.1 Parameters concerning the multiple scattering of electrons in a gaseous environment

To link the scattering probability with the gas pressure and the distance between specimen and PLA L , several assumptions are necessary (Danilatos, 1988) (formula 6.1). The average number of collisions m must be small as well as the average scattering angle and energy loss relative to the beam direction and beam energy. Therefore, the resulting average distance r is small in comparison with the covered distance and a sharp pressure transition between high vacuum and chamber pressure must be assumed exactly at the PLA.

$$m = \sigma_T \cdot n \cdot L \quad 6.1$$

The total scattering cross-section σ_T is the sum of all kinds of cross-sections σ_j . In most cases it is divided into elastic and inelastic scattering events (formula 6.2).

$$\sigma_T = \sum_j \sigma_j$$

6.2

Some of the inelastic first order (energy transfer from electron to atom) and second order (energy transfer from atom to electron) collisions can be seen in figure 6.2.

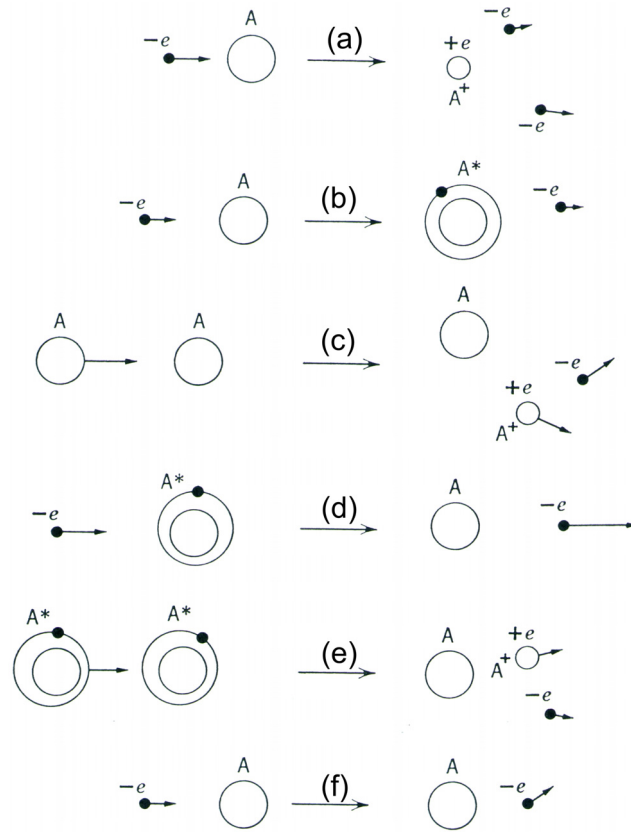


Figure 6.2 Some first order (a,b,c) , second order (d,e) inelastic collisions and elastic collision (f) (adapted from Nasser (1971))

Especially for multiple scattering problems knowledge of the mean free path (see formula 2.13) is of main importance. The mean free path is the distance an electron travels between two successful interactions.

The remaining unscattered density of the electron beam, which is a very important parameter for environmental scanning electron microscopy, can also be calculated by knowledge of the total scattering cross-section (see formula 3.2).

In summary the main important parameters, the mean free path, the remaining unscattered density and the average number of collisions can be calculated by knowledge of the total scattering cross-section.

6.1 Total Scattering Cross-Section

For the calculation of the total scattering cross-section knowledge of the elastic differential cross-section $d\sigma_e/d\Omega$ and inelastic differential cross-section $d\sigma_i/d\Omega$ is necessary (formula 6.3).

$$\sigma_T = \sigma_e + \sigma_i = 2 \cdot \pi \cdot \int_0^\pi \left(\frac{d\sigma_e}{d\Omega} + \frac{d\sigma_i}{d\Omega} \right) \cdot \sin(\theta) \cdot d\theta \quad 6.3$$

The mathematical expressions concerning the skirt effect were presented by Lenz (1954) and adapted by Jost and Kessler (1963) and Danilatos (1988). The elastic and inelastic scattering of electrons in gases must be separately discussed for monatomic gases and gas molecules because the binding effects of the atoms influence the scattering behaviour.

6.1.1 Monatomic Gases

The probability distribution of electron scattering in monatomic gases was presented by Jost and Kessler (1963). Based on this publication the elastic and inelastic differential cross-sections were adapted by Danilatos (1988) (formula 6.4 and 6.5):

$$\left(\frac{d\sigma_e}{d\Omega} \right)_A = \frac{A \cdot Z}{16 \cdot \left[\sin^2\left(\frac{\theta}{2}\right) + \sin^2\left(\frac{\theta_0}{2}\right) \right]^2} \quad 6.4$$

$$\left(\frac{d\sigma_i}{d\Omega} \right)_A = \frac{A \cdot (\theta^2 + \theta_E^2 + 2 \cdot \theta_0^2)}{(\theta^2 + \theta_E^2) \cdot (\theta^2 + \theta_E^2 + \theta_0^2)^2} \quad 6.5$$

where

$$\theta_0 = \frac{\lambda}{2 \cdot \pi \cdot R} \quad \theta_E = \frac{J}{4 \cdot E} \quad A = \frac{\lambda^4 \cdot Z \cdot \left(1 + \frac{E}{E_0}\right)^2}{4 \cdot \pi^4 \cdot a_H^2} \quad R = \left[f_e(0) \cdot \frac{a_H}{2 \cdot Z} \right]^{0.5} \quad 6.6$$

and where J is the first ionisation energy, R is the effective atomic radius, E_0 is the electron rest energy and $f_e(0)$ is the elastic scattering amplitude at 0 rad.

The expression for the elastic differential cross-section is valid for all angles in comparison with the inelastic expression, which is only valid for angles less than 0.17 rad (10°). Nevertheless the elastic scattering is mainly responsible for the scattering into higher angles and therefore this limitation does not influence the results when calculating the total scattering

cross-section. In figure 6.3 the strong decrease of the total differential cross-section with increasing scattering angle can be seen. For the calculation necessary parameters can be seen in table 1. Interesting in this context is that the probability of scattering into angles higher than $\pi/2$ is one thousand times smaller than the probability that a scattering event takes place (Danilatos, 1988). Therefore, backscattering from the imaging gas in an ESEM can be neglected.

Table 1 Values of atomic number Z , first ionisation potential J and scattering amplitude $f_e(0)$ (Danilatos, 1988, Linde et al., 1994)

	Z	$J[\text{eV}]$	$f_e(0)[\text{m}]$
H	1	13.60	0.529
N	7	14.53	2.200
O	8	13.62	2.010
Ne	10	21.56	1.660
Ar	18	15.76	4.710

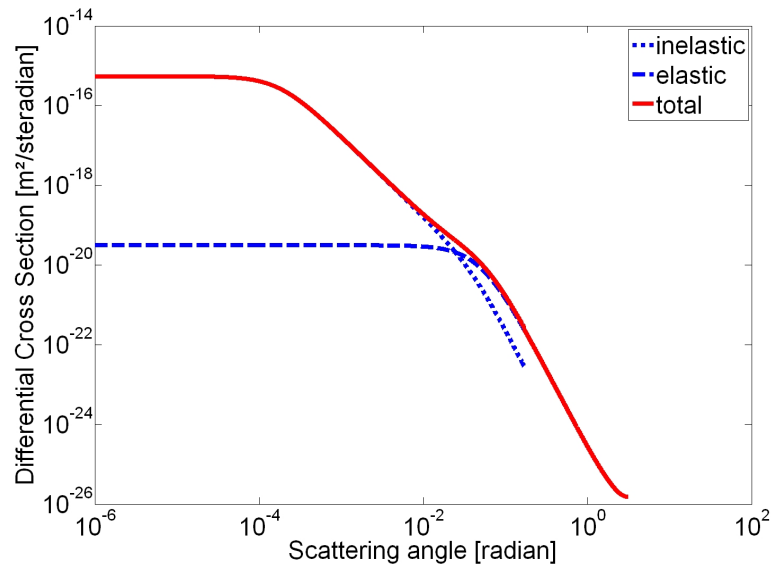


Figure 6.3 Elastic, inelastic and total differential cross-section of neon (calculated with the program TSCS of monatomic gases (see chapter 11.5.3))

Problematic parameters are the effective atomic radius, the distance over which the nucleus of the atom is active, and the ionisation potential. Danilatos (1988) calculated the elastic cross-sections by using the first ionisation potential but mentioned that the mean ionisation potential is maybe a better choice. Detailed discussions of these parameters are following in chapter 7.5.

The total scattering cross-section can be calculated by integration over the elastic and inelastic differential cross-section from zero to π . Unfortunately this integral can not be calculated analytically, numerical methods must be used.

For the numerical integration a lower integration limit must be defined. By comparison the numerical results with the analytical expression of the total scattering cross-section for monatomic gases presented by Jost and Kessler (1963) (formula 6.7), the lower integration limit was set to $1 \cdot 10^{-6}$ rad. Further lowering of the integration limit does not influence the results. The curve progressions between the analytical expression and the presented model coincide, which indicates that the established basic model is correct (see figure 6.4).

$$\sigma_T = \frac{\lambda^2 \cdot R^2 \cdot Z \cdot \left(1 + \frac{E}{E_o}\right)}{\pi \cdot a_H^2} \cdot (Z - 1 - 4 \cdot \ln(\beta)) \quad 6.7$$

with

$$\beta = \frac{\pi \cdot R \cdot J}{2 \cdot \lambda \cdot E}$$

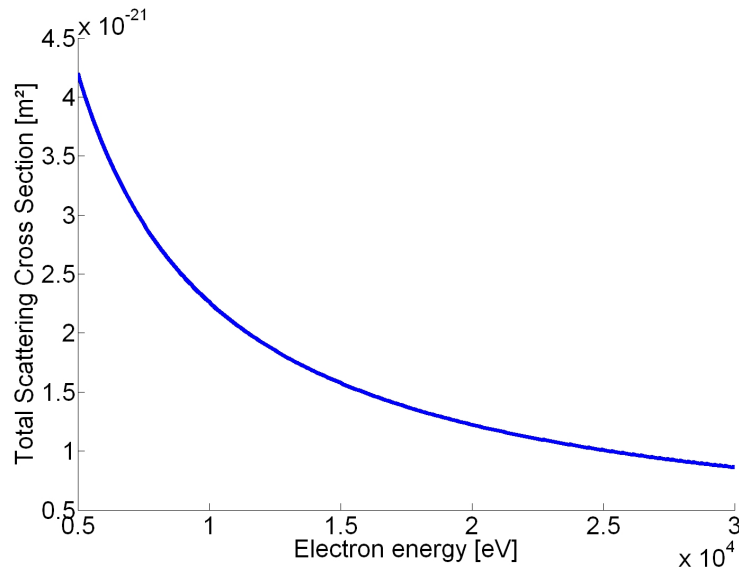


Figure 6.4 Total scattering cross-section of neon
(calculated with the program TSCS of monatomic gases (see chapter 11.5.3))

6.1.2 Molecular Gases

In first approximation Danilatos (1988) described the inelastic differential scattering cross-section for molecular gases as the sum of all individual cross-sections of the atoms forming the molecule (formula 6.8).

$$\left(\frac{d\sigma_i}{d\Omega}\right)_M = \sum_j \left(\frac{d\sigma_{ij}}{d\Omega}\right)_A \quad 6.8$$

Calculating the elastic scattering is more difficult, because the cross-section is not only the sum of all individual elastic differential cross-sections, binding effects between the atoms play an additional role.

The following condition (formula 6.9) must be fulfilled to apply the derivation for the elastic differential cross-section of molecules:

$$\frac{2 \cdot \pi \cdot d_0}{\lambda} \gg 1 \text{ and } d_0 \gg r_m \quad 6.9$$

where

$$r_m = \sqrt{\frac{\sigma_{eT}}{\pi}} \quad 6.10$$

and where d_0 is the minimum distance between scattering centres, σ_{eT} is the total elastic scattering cross-section and r_m is the maximum range of interaction, which is estimated with the radius of the total elastic scattering cross-section (formula 6.10).

Both conditions are adequately satisfied for the gas molecules H_2 , N_2 , O_2 and H_2O and electron energies between 5 keV and 25 keV (see table 2). It can clearly be seen that the conditions become more critical at low electron energies.

Table 2 Conditions for determining the total scattering cross-section of molecules
(Danilatos, 1988)

	r_{ij} [m]	r_m [m]		$\frac{2 \cdot \pi \cdot d_0}{\lambda}$	
		5	25	5	25
H ₂	$0.746 \cdot 10^{-10}$	$0.039 \cdot 10^{-10}$	$0.018 \cdot 10^{-10}$	27	61
N ₂	$1.098 \cdot 10^{-10}$	$0.211 \cdot 10^{-10}$	$0.098 \cdot 10^{-10}$	40	90
O ₂	$1.208 \cdot 10^{-10}$	$0.216 \cdot 10^{-10}$	$0.099 \cdot 10^{-10}$	44	99
H ₂ O	$0.958 \cdot 10^{-10}$			35	78
	$0.958 \cdot 10^{-10}$				
	$1.515 \cdot 10^{-10}$				

The scattering centres are named n and j and the distance between the centres is r_{nj} (see formula 6.11).

$$\left(\frac{d\sigma_e}{d\Omega} \right)_M = \sum_n \sum_j f_n(\theta) \cdot f_j(\theta) \cdot \frac{\sin(s \cdot r_{nj})}{s \cdot r_{nj}} \quad 6.11$$

where

$$s = \frac{4 \cdot \pi \cdot \sin\left(\frac{\theta}{2}\right)}{\lambda}$$

The scattering amplitudes $f(\theta)$ for formula 6.11 can be calculated by using formula 6.12.

$$\frac{d\sigma_e}{d\Omega} = |f_n(\theta)|^2 \quad 6.12$$

In the case of $i=j$ formula 6.11 simplifies to formula 6.13 by determining the limes of $r_{nj} \rightarrow 0$.

$$\lim_{r_{nj} \rightarrow 0} \left(f_n(\theta) f_j(\theta) \cdot \frac{\sin(s \cdot r_{nj})}{s \cdot r_{nj}} \right) = |f(\theta)|^2 \quad 6.13$$

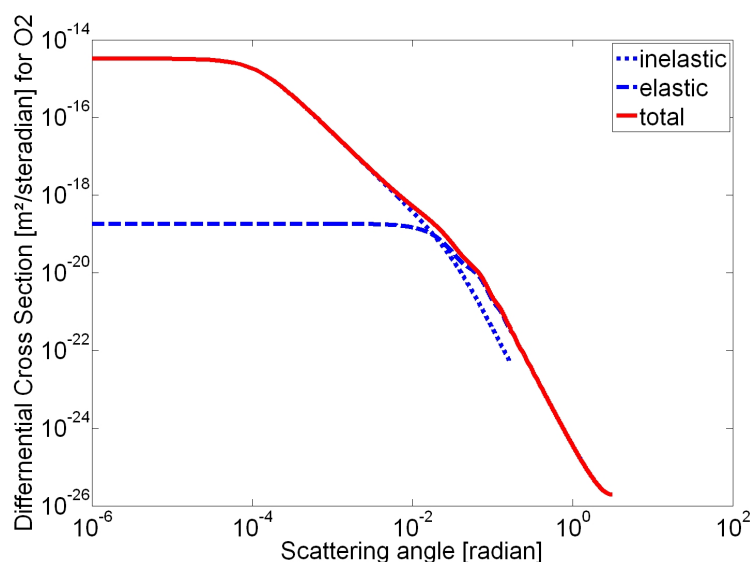


Figure 6.5 Elastic, inelastic and total differential cross-section of O₂
(calculated with the program TSCS of molecular gases (see chapter 11.5.4))

The differential cross-sections of O₂ can be seen in figure 6.5. Again the numerical integration over the elastic and inelastic differential cross-section from $1 \cdot 10^{-6}$ rad to π (formula 6.3) gives the total scattering cross-section (see figure 6.6).

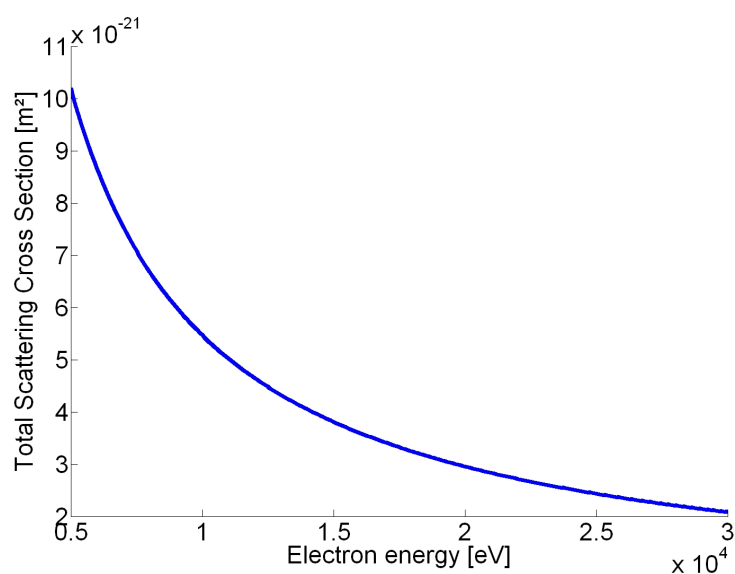


Figure 6.6 Total scattering cross-section of O₂
(calculated with the program TSCS of molecular gases (see chapter 11.5.4))

7 Determination of the Total Scattering Cross-Section

In environmental scanning electron microscopy the total scattering cross-section of the imaging gas is a very important physical parameter concerning the influence of the scattered primary beam electrons on X-ray spectrometry (Gauvin, 1999; Doehne, 1997) as well as for estimating the beam damage (Zankel et al., 2007). However, detailed knowledge of the total scattering cross-section is essential for further developments of the vacuum system and of the PLA design (Danilatos, 2009).

7.1 The Faraday Cup

In the following chapter a method is presented to determine the total scattering cross-section of imaging gases in a low vacuum scanning electron microscope (LVSEM) or environmental scanning electron microscope (ESEM). This physical parameter depends on the imaging gas type and on the electron energy.

Most of the scattering events results in very small scattering angles (Danilatos, 1988). Therefore, the most important factor of these measurements is the acceptance angle of the measuring device.

For these investigations a design was chosen to measure the unscattered fraction of the electron beam in a direct way. This is very difficult because the scattered electrons and the generated positive gas ions have to be shielded from the rest of the device. By using a normal Faraday cup the scattered primary beam electrons hit the cup at a certain distance away from the aperture and will be additionally measured. However, there is also a high amount of positive gas ions, because the secondary electron signal can be amplified several hundred times (Thiel, 2003) and a big amount of these ions recombine at the cup or the stage. Hence a positive specimen current can be measured caused by the high amount of positively charged gas particles.

A new Faraday cup which was developed inspired by the design of Flechter et. al. (1997), but in comparison to a 300 μm and a 30 μm aperture much smaller apertures were used (2 times 10 μm ; see figure 7.1).

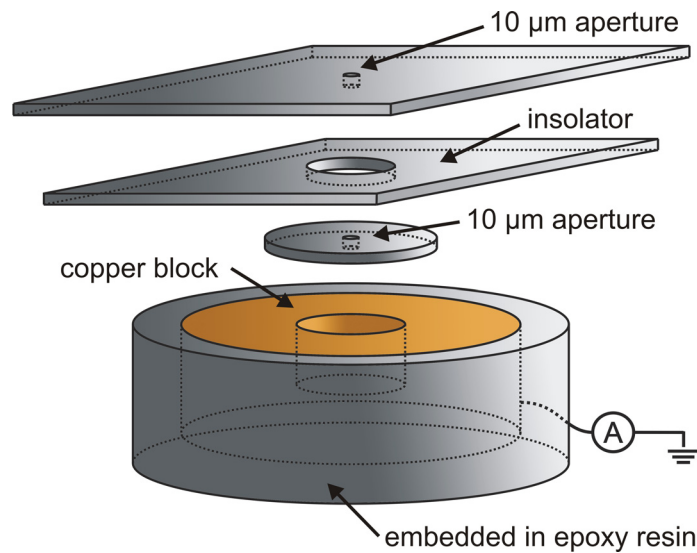


Figure 7.1 Schematic drawing of the Faraday cup (Rattenberger et al., 2008)

The main problem was the alignment of the two 10 μm apertures. As a first step, two 10 μm titanium apertures were used and aligned with the help of a transmission light microscope. However, the light diffracts at the upper aperture, which leads to a light transmission even in the case of misalignment.

Therefore, the cup was built in the focused ion beam microscope (FIB Nanolab Nova 200, FEI). A prefabricated titanium aperture was used in combination with an aluminium foil. The ion beam was focused through the aperture on the aluminium foil and the second aperture was milled by the ion beam. With this technique a perfect alignment of the two apertures can be achieved (figure 7.2 to figure 7.4).

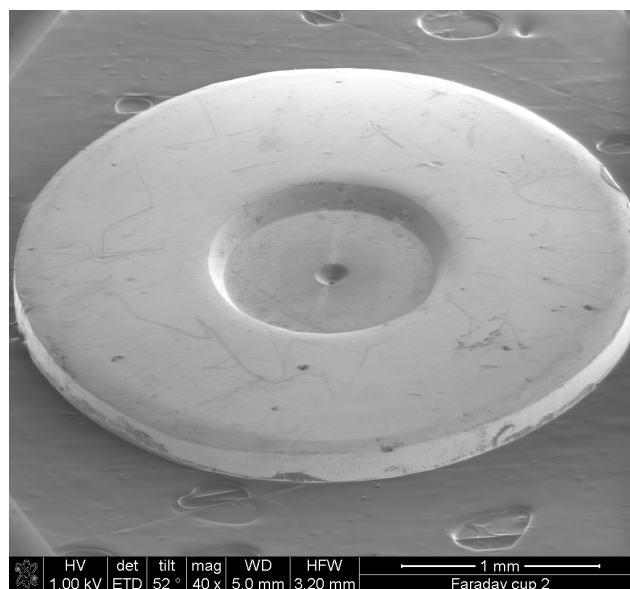


Figure 7.2 The titanium aperture on top of the insulating tape (SE image, tilt angle 52°, 1 kV, FIB Nanolab Nova 200, FEI)

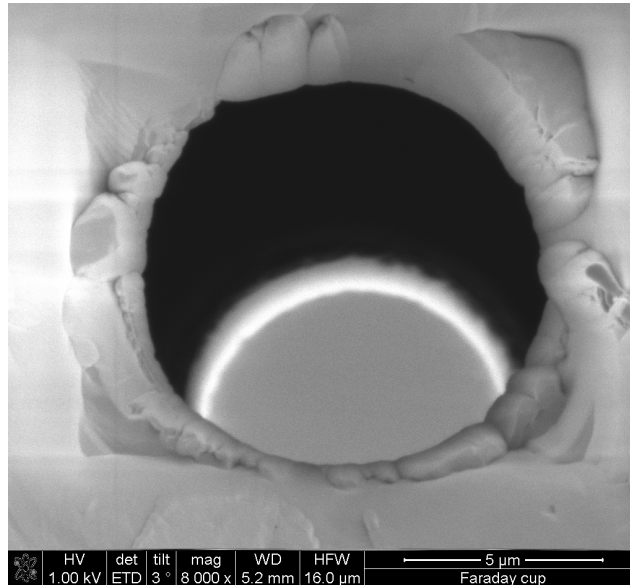


Figure 7.3 A look trough the titanium aperture and trough the milled aluminium aperture (tilt angle 3°, SE image, 1 kV, FIB Nanolab Nova 200, FEI)

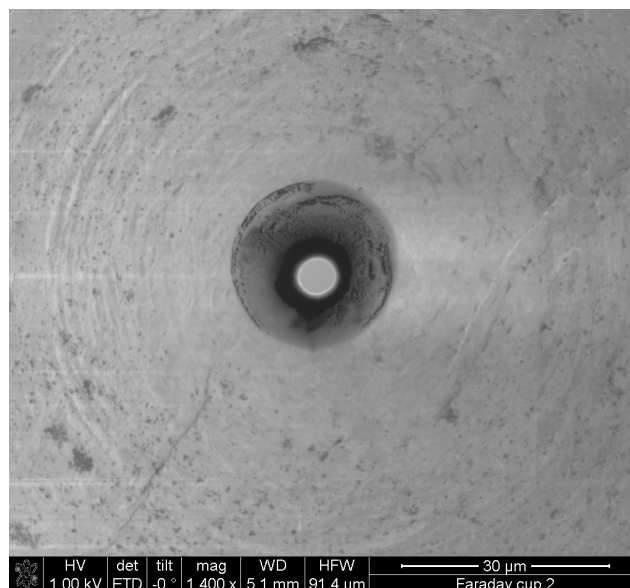


Figure 7.4 A look trough the titanium aperture and trough the milled aluminium aperture (tilt angle 0°, SE image, 1 kV, FIB Nanolab Nova 200, FEI)

It can clearly be seen that the two apertures are perfectly aligned. By adjusting the two apertures at the construction they can very easily shift, which can be seen in figure 7.5. The previously aligned apertures show a little mismatch of about 4 μm.

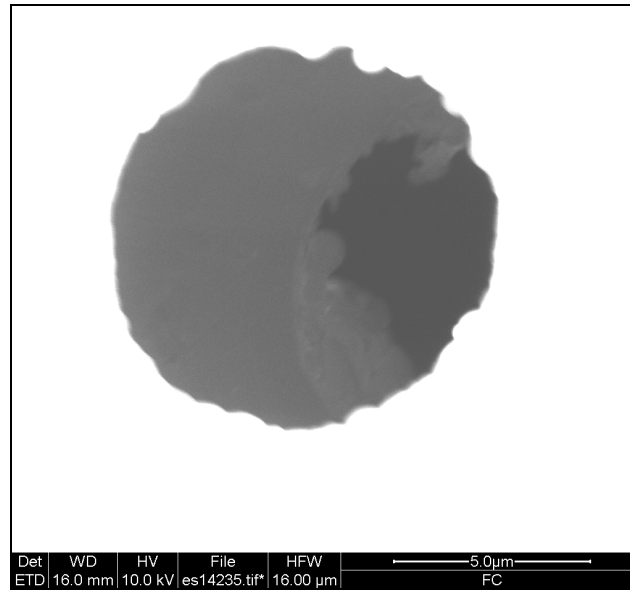


Figure 7.5 The misalignment after the complementation of the Faraday cup
(SE image, 10 kV, ESEM Quanta 600, FEI)

However, this misalignment does not prevent measuring. The acceptance angle of the Faraday cup is further decreased, which improves the measurement accuracy. The influence of the acceptance angle on the results is discussed in detail in chapter 7.5.

7.2 The Method

With the presented device it is possible to measure the fraction of the unscattered electrons under low vacuum conditions, considering the acceptance angle of the device. Formula 3.2 shows the analytical expression for the presented problem.

The unscattered electron current can be measured in high vacuum and the fraction of unscattered electrons at a defined chamber pressure and working distance can be determined in low vacuum. Especially the Beam Gas Path Length (BGPL), the distance the electrons travel inside the gaseous environment, is difficult to consider. The working distance is different from the BGPL because of the construction of the microscope (see figure 3.9) and it depends on the pressure gradient. Latest publications on this topic have shown very impressive the isodensity contours of the imaging gas around a PLA (Danilatos, 2009) (see figure 7.6).

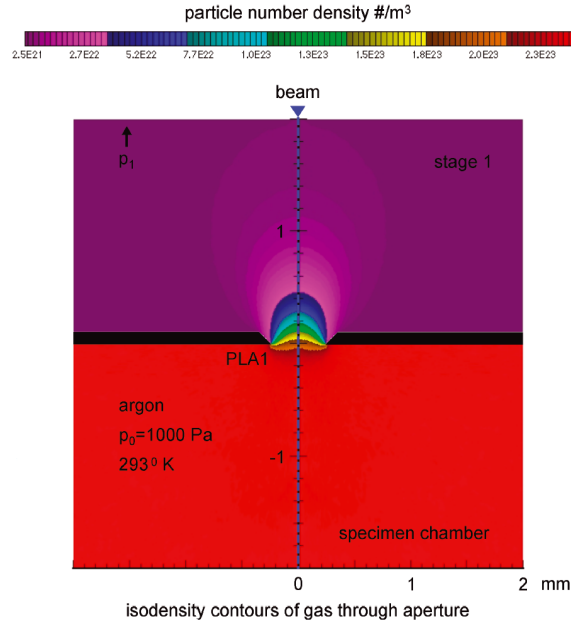


Figure 7.6 Isodensity contours of argon around a PLA (Danilatos, 2009)

Under consideration of these findings the working distance (WD) can not be used as BGPL to calculate the total scattering cross-section.

However, by varying the WD, or more specifically the BGPL, the total scattering cross-section can be calculated without the need of this parameter.

Figure 7.7 shows the average number of collisions as function of the working distance for different chamber pressures. This linear dependency can be used for further calculations (formula 7.1):

$$m = \frac{\sigma_T \cdot p \cdot (WD + \Delta(p))}{k_B \cdot T} = k_\sigma \cdot (WD + \Delta(p, type)) + d_\sigma \quad 7.1$$

where T is the temperature of the imaging gas, $\Delta(p)$ a pressure depending parameter, k_σ the slope of the best fit straight line (see figure 7.7) and d_σ the y-intercept of the best fit straight line (see figure 7.7).

In this case the BGPL is replaced by the working distance plus a pressure dependant parameter. In other words the variation of the average number of collisions with varying BGPL can be expressed by the slope of the best fit straight line (formula 7.2).

$$k_\sigma = \frac{\Delta m}{\Delta BGPL} \quad 7.2$$

Therefore, the total scattering cross-section can be calculated by using formula 7.3.

$$\sigma = \frac{m \cdot k_B \cdot T}{p \cdot BGPL} = \frac{k_\sigma \cdot k_B \cdot T}{p} \quad 7.3$$

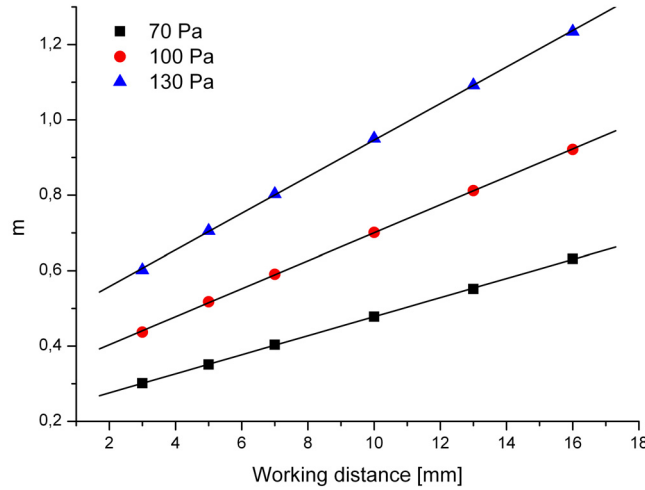


Figure 7.7 Average number of collisions per electron with the imaging gas as a function of the working distance [mm] (imaging gas: H₂O, 15 keV)

7.2.1 Temperature and Pressure Conditions

The two remaining parameters which must be confirmed are the temperature and the pressure in the specimen chamber. The total scattering cross-section is direct proportional to the temperature. Therefore, knowledge of the exact temperature is important to increase the measurement accuracy. Because of the expansion of the imaging gas into the vacuum, adiabatic cooling of the gas could be possible. He and Joy (2003) measured the temperature of the imaging gas with a sensitive thermocouple close to the inlet gas jet. They measured a temperature drop down during the pumping process, but after pressure stabilisation, the temperature recovered the starting value. The gas rapidly achieves thermal equilibrium with the chamber walls and therefore the imaging gas can be assumed to be at room temperature. Following these results the temperature of the imaging gas was assumed to be at room temperature for the calculations of the total scattering cross-sections.

The pressure of the imaging gas is a result of the inlet of gas from a selected source and the pumping of the gas caused by the differential pumping system (see figure 3.4). Therefore, the equilibrium of the pressure in the specimen chamber must be checked separately. In environmental scanning electron microscopy a lot of different gases are used and the most pressure measuring devices are gas type depending. In the ESEM Quanta 600 itself are two

pressure measuring devices. An Edwards Active Gauge AIM-S-NW 25 for high vacuum measuring and a Pfeiffer CMR 262 (see figure 7.8) for the ESEM and low vacuum regime.



Figure 7.8 Pfeiffer CMR 262, positioned between specimen chamber and turbomolecular pump

In comparison with the Edwards device, the Pfeiffer measures the pressure independently from the gas type, because it is a capacitance gauge. The momentum of the gas atoms or molecules is used and not the ionisation energy. This device can be used for pressure regions between 10^{-2} to 110 mbar with an accuracy of 0.2 % of the result (Dondorf, 2004). Therefore, it is an ideal device for these investigations. However, it is positioned just below the chamber, above the turbomolecular pump and therefore the pressure equilibrium inside the chamber must be checked.

For these investigations an Inficon Triple Gauge TM BCG450 was used and mounted at different positions at the chamber. It is not that accurate ($1 \cdot 10^{-8}$ to 50 mbar, accuracy 15% of the result (Inficon, 2005)) but it can be easily mounted at different positions. In figure 7.9 and figure 7.10 the two different positions are shown.

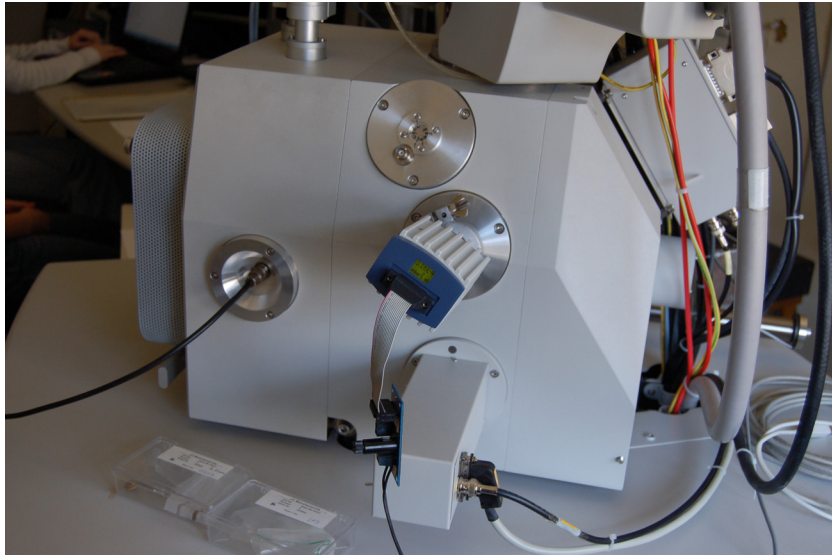


Figure 7.9 Inficon device mounted at the right-back side of the chamber (Position 1)



Figure 7.10 Inficon device mounted at the left-front side of the chamber (Position 2)

The Inficon measuring device is gas type depended and the result for different gases are shown in figure 7.11. It can clearly be seen that the Inficon device is calibrated for nitrogen gas, the measured values agree very well for both devices. The high amount of nitrogen in ambient air can also be seen, the two curves are nearly identical.

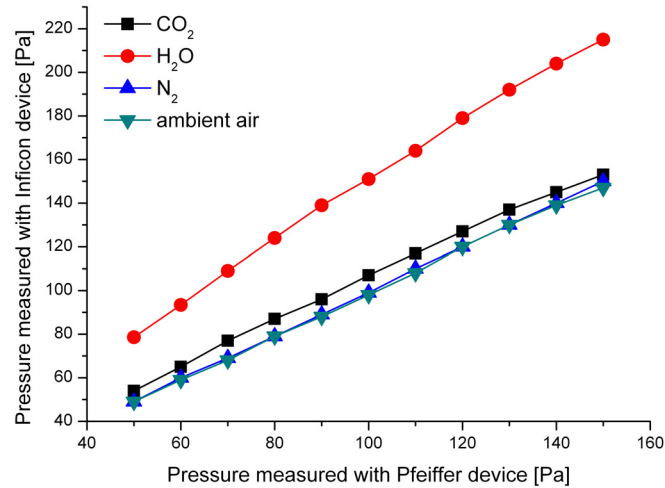


Figure 7.11 Gas type dependent pressure measurements (Pfeiffer (independent) vs Inficon (dependent))

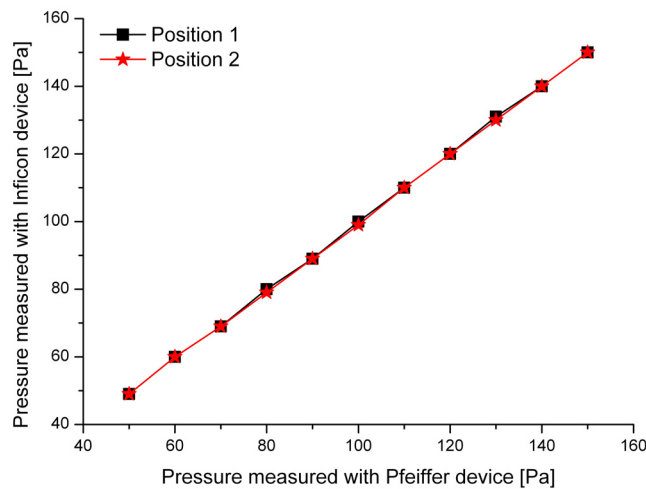


Figure 7.12 Pressure measured with the Inficon device vs. the Pfeiffer device

Nevertheless this device is adequate to check if there is pressure equilibrium inside the chamber (see figure 7.12).

In all three cases the values are nearly identical which indicates that the gas inside the specimen chamber is in equilibrium and that the displayed value from the Pfeiffer device can be used to calculate the total scattering cross-section. These results are in good agreement with theoretical considerations (Danilatos, 1994). The main part of the gas inside the chamber is at rest, because the volume of the chamber is very big compared to the diameter of the PLA and the gas inlet.

7.3 Experimental Results

The measurements were performed at the ESEM Quanta 600 (see figure 7.13). The field emission gun of this microscope is inevitable for a stable electron source during measurements. The primary beam current was read out before and after each measuring cycle to check the stability of the source.



Figure 7.13 The ESEM Quanta 600 (FEI) at the Institute for Electron Microscopy and Fine Structure Research

As described in chapter 3.2.2 the microscope can be used in LVSEM or ESEM mode. For using the microscope as an ESEM, the GSED must be inserted. This detector considers the indispensable second PLA. Because of the complex gas flow inside the chamber, especially nearby the PLA, the following measurements were performed in low vacuum mode to simplify the gas flow.

As described, the total scattering cross-section can be determined by measuring the primary beam current under high vacuum conditions and the unscattered fraction in gaseous environment. The working distance, or more specifically the BGPL must be changed to obtain the slope of the best fit straight line. For every measurement several working distances (3, 5, 7, 10, 13, 16 mm) and pressures (70, 100, 130 Pa) were investigated for statistical reasons. The acceleration voltage was varied between 5 keV and 30 keV in 5 keV steps. For these measurements the primary beam current was set to exact 1 nA by varying the beam cross-over. After each measuring cycle the stability of the primary beam current was checked in high vacuum.

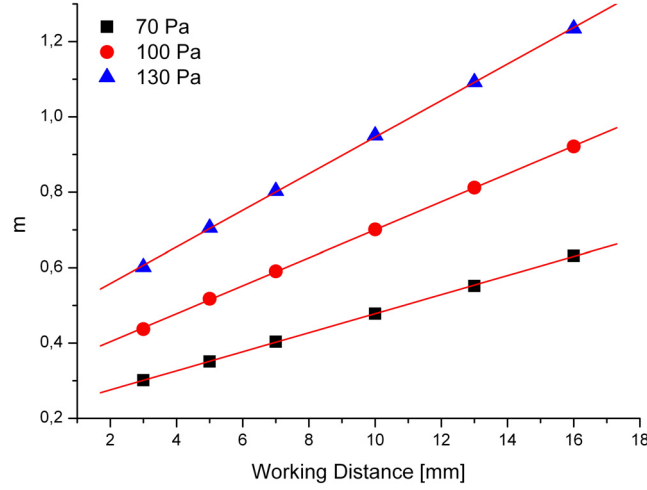


Figure 7.14 Average number of collisions per electron with the imaging gas as a function of the working distance (imaging gas: H₂O, 15 keV)

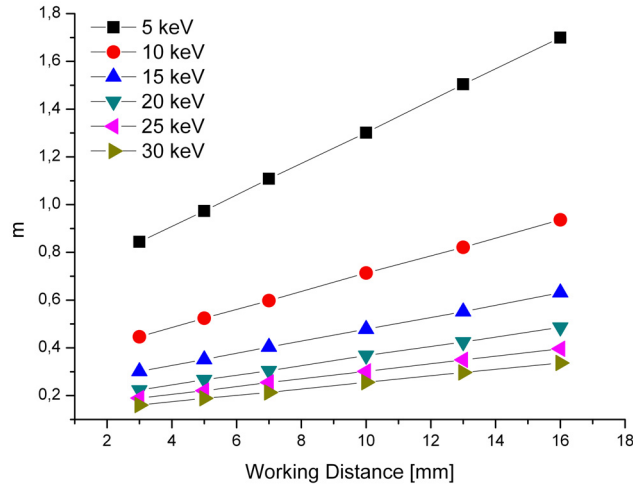


Figure 7.15 Average number of collisions per electron with the imaging gas as a function of working distance (imaging gas: H₂O, 70 Pa)

In figure 7.14 and figure 7.15 examples of such results can be seen. The average number of collisions m goes linearly with increasing working distance and with increasing pressure. The correlation coefficient of these linear regressions in figure 7.14 is nearly 1 (>0.999), which indicates the high accuracy of this method. It is not necessary to calculate the difference between working distance and BGPL because only the slope of the best fit straight line is essential.

Based on these investigations the total scattering cross-section can be calculated by using formula 6.4 (see figure 7.16). Averaging over the particular values for the different pressures

further increases the accuracy. The data points can be fitted with the expression presented by Joshipura and Vinodkumar (1996) (formula 7.4):

$$\sigma = A \cdot (E \cdot 1000)^{-B} \quad 7.4$$

where A and B are gas depending parameters.

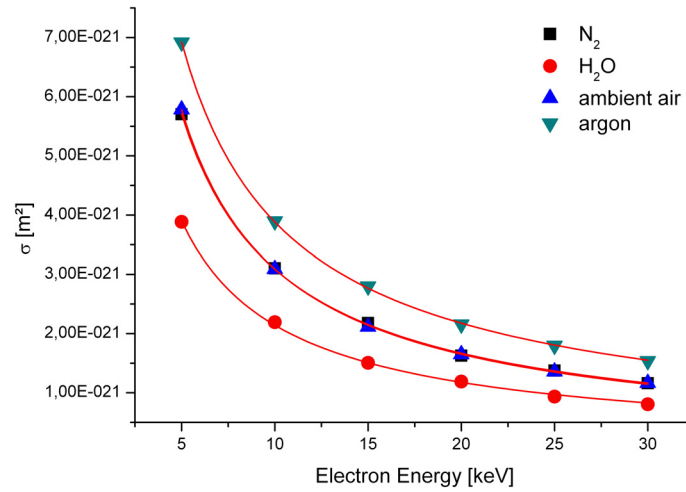


Figure 7.16 Total scattering cross-section as a function of electron energy

Table 3 Experimental results

E [keV]	$\sigma(\text{N}_2)$ [m^2]	$\sigma(\text{H}_2\text{O})$ [m^2]	$\sigma(\text{ambient air})$ [m^2]	$\sigma(\text{argon})$ [m^2]
5	5.70E-21	3.88E-21	5.78E-21	6.92E-21
10	3.10E-21	2.19E-21	3.08E-21	3.89E-21
15	2.17E-21	1.50E-21	2.11E-21	2.80E-21
20	1.62E-21	1.18E-21	1.64E-21	2.16E-21
25	1.37E-21	9.34E-22	1.35E-21	1.80E-21
30	1.16E-21	8.05E-22	1.16E-21	1.54E-21

Table 4 Experimental results of formula 6.4

	A [m ² /keV]	B	χ^2	R ²
H ₂ O	1.568E-20	0.864	0.119E-46	0.99929
N ₂	2.381E-20	0.887	6.651E-46	0.99982
ambient air	2.475E-20	0.904	3.031E-46	0.99992
argon	2.654E-20	0.835	4.954E-46	0.9999

The experimental results are shown in table 3. The theoretically derived curve progression fits very well and the gas depending parameters A and B are shown in table 4. The error square values are small and the correlation coefficients near by one which indicates the high measurement accuracy of the presented method. However, these values are only considering the statistical errors, the influence of the systematically errors are discussed in chapter 7.5.

7.4 Theoretical Results

The theoretical concept of Danilatos (1988), which is presented in chapter 6, was used to calculate the total scattering cross-section of nitrogen gas, water vapour, ambient air and argon.

Matlab was used to program the described algorithm and for the numerical integrations, which are necessary for formula 5.5.

Because of the strong angular dependency of the differential cross-sections, it was necessary to divide the different angle regions into different step sizes (see appendix 11.5.3 and 11.5.4). The first regime is from $1 \cdot 10^{-6}$ rad to $5 \cdot 10^{-5}$ rad with an iteration step size of $1 \cdot 10^{-6}$, the second part from $5 \cdot 10^{-5}$ rad to 0.17 rad with a step size of $1 \cdot 10^{-5}$ rad and the rest till π with a step size $1 \cdot 10^{-3}$ rad.

With these parameters the computation time is acceptable (3 to 8 minutes). The lowest integration limit is $1 \cdot 10^{-6}$ rad, which is small enough according to Danilatos, the results do not vary any more by decreasing this value. At 0.17 rad the inelastic differential cross-section formula becomes invalid. By further decreasing the iteration step sizes the results do not vary any more which indicate that the parameters are set correctly. The necessary parameters for the calculations are shown in table 1.

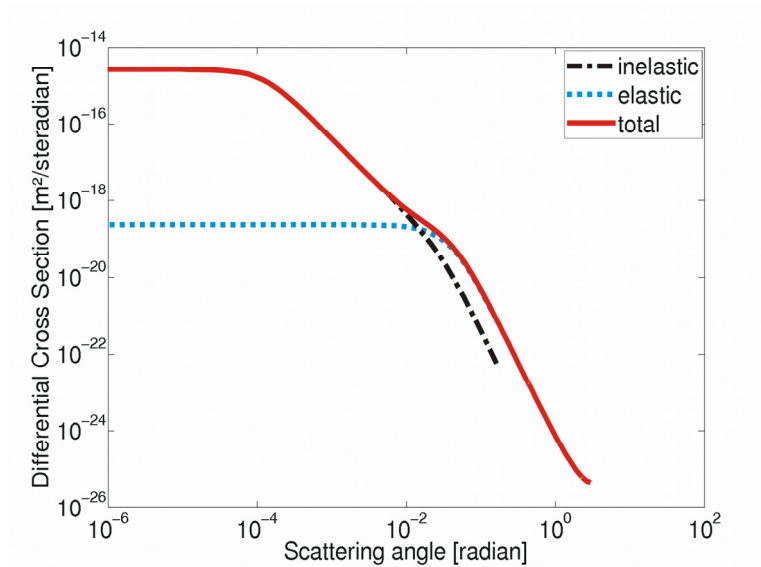


Figure 7.17 Elastic, inelastic and total differential cross-section of argon (calculated with the program TSCS of monatomic gases (see chapter 11.5.3))

The inelastic, the elastic and the total differential cross-section of argon can be seen in figure 7.17. At low scattering angles the inelastic differential cross-section is much bigger than the elastic cross-section. By increasing the scattering angle above 10^{-2} rad the elastic scattering dominates and the invalidity of the inelastic theory above 0.17 rad carries no weight concerning the results. In figure 7.18 both calculated cross-sections can be seen, using the theory of Danilatos (1988) and the analytical expression by Kessler 1963. Both curve progressions are identical which indicates that the basic concept is correct.

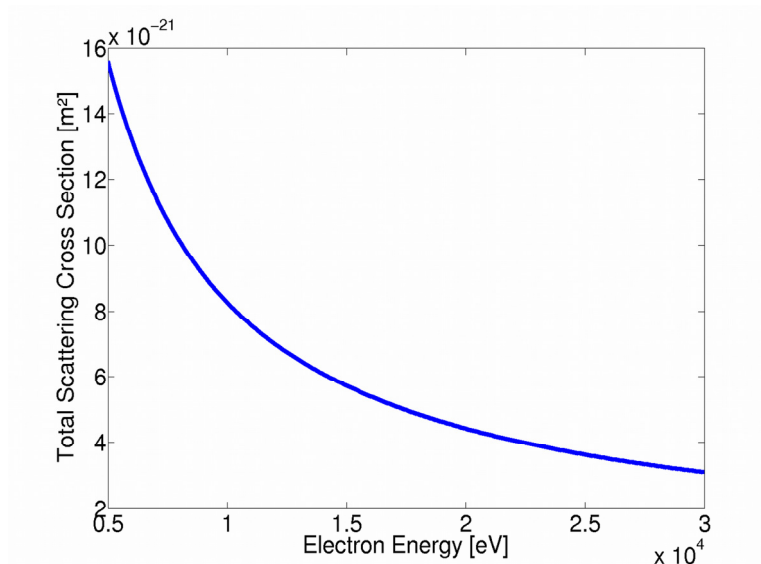


Figure 7.18 Total scattering cross-section of argon (Danilatos, 1988; + Kessler 1963) (calculated with the program TSCS of monatomic gases (see chapter 11.5.3))

In figure 7.19 the calculated total cross-sections for argon, water vapour, nitrogen gas and ambient air can be seen. The curve progression of nitrogen gas is nearly identical with ambient air, which is not surprising because of the high amount of nitrogen in ambient air ($N_2 \sim 78.1\%$; $O_2 \sim 20.9\%$; $Ar \sim 0.93\%$ (Linde et al., 1994)).

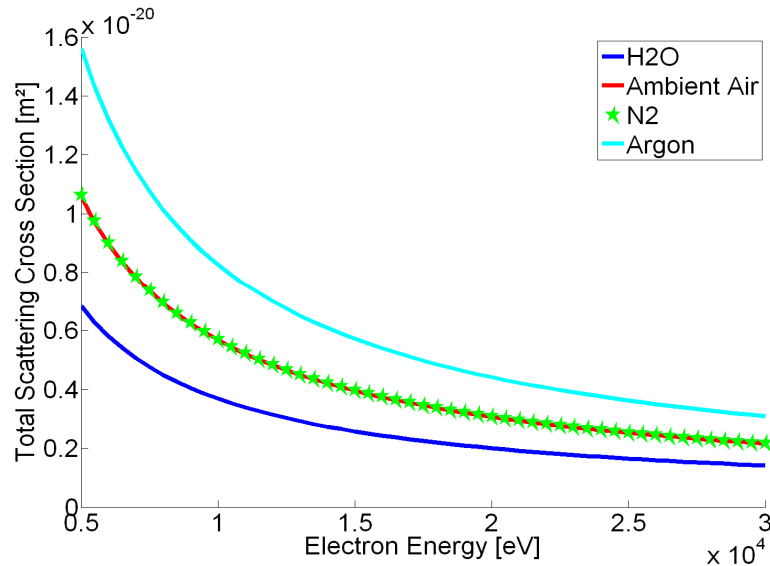


Figure 7.19 Total scattering cross-section of water vapour, ambient air, nitrogen gas and argon as a function of electron energy (calculated with the program TSCS of monatomic gases (see chapter 11.5.3) and with the program TSCS of molecular gases (see chapter 11.5.4))

7.5 Comparison of Experimental Data with Calculations

The comparison between theoretical and experimental results for water vapour shows a significant difference (see figure 7.20). There are two possible reasons for these differences. On the one hand the experimental measurements are affected by the acceptance angle of the Faraday cup which results in an underestimation of the total scattering cross-section. On the other hand the assumptions and simplifications of the calculations influence the theoretical results (see chapter 6).

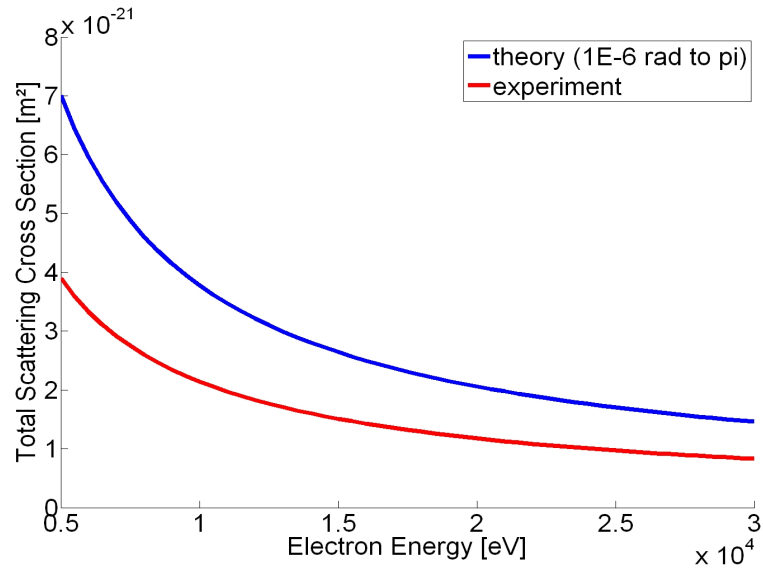


Figure 7.20 Comparison of experimental and theoretical determined total scattering cross-section of water vapour

The main parameter which complicates the comparison is the acceptance angle of the Faraday cup. A fraction of electrons which undergo a scattering event are measured as unscattered electrons because of the diameter of the Faraday cup, although the diameter is only $10\text{ }\mu\text{m}$ (figure 7.21).

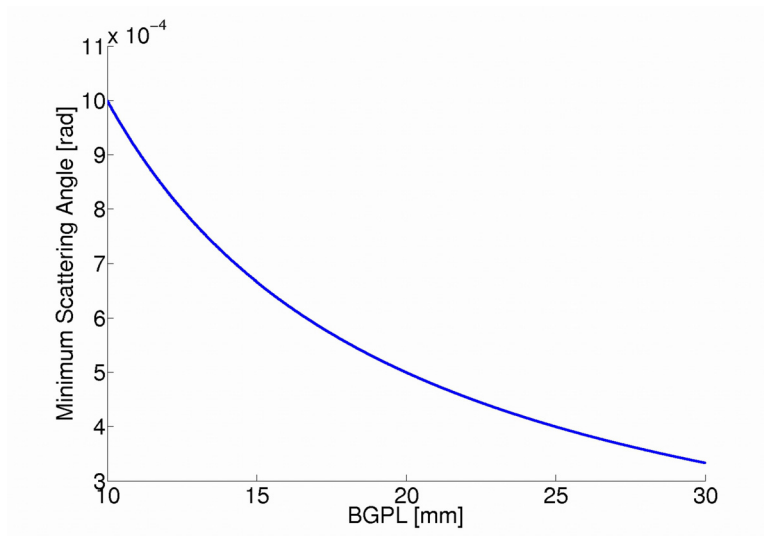


Figure 7.21 Minimum acceptance angle of the Faraday cup [rad] as a function of the BGPL [mm] by using $10\text{ }\mu\text{m}$ apertures

In first approximation the variation of the integration limits can be used for the comparison of the results (Rattenberger et al., 2009) (figure 7.22-figure 7.25).

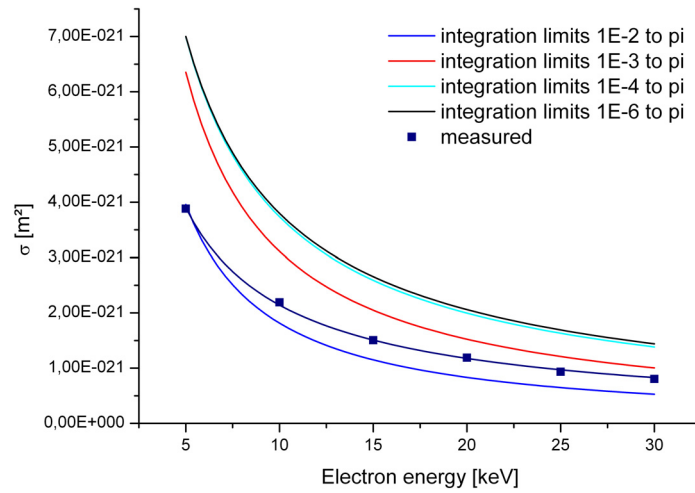


Figure 7.22 Variation of the integration limits for water vapour (calculated with the program TSCS of molecular gases (see chapter 11.5.4))

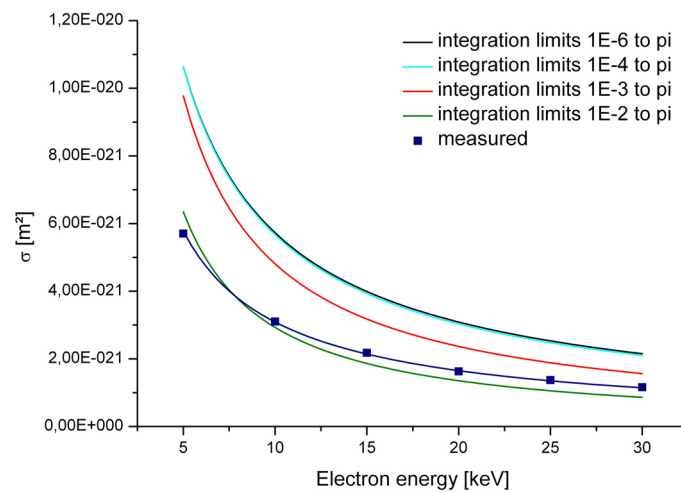


Figure 7.23 Variation of the integration limits for nitrogen gas (calculated with the program TSCS of molecular gases (see chapter 11.5.4))

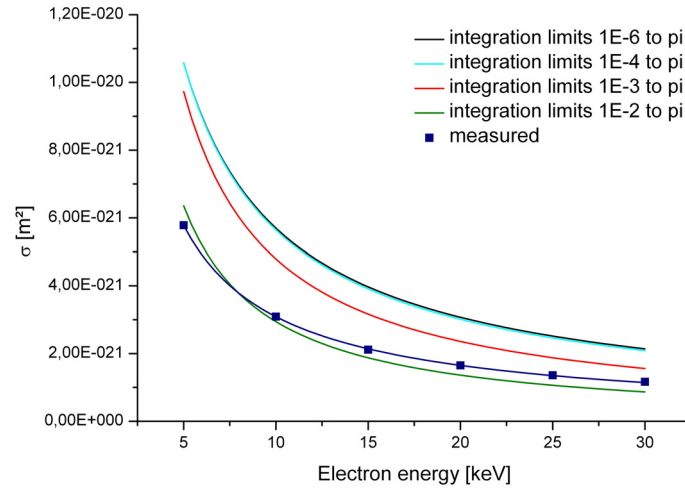


Figure 7.24 Variation of the integration limits for ambient air (calculated with the program TSCS of molecular gases (see chapter 11.5.4))

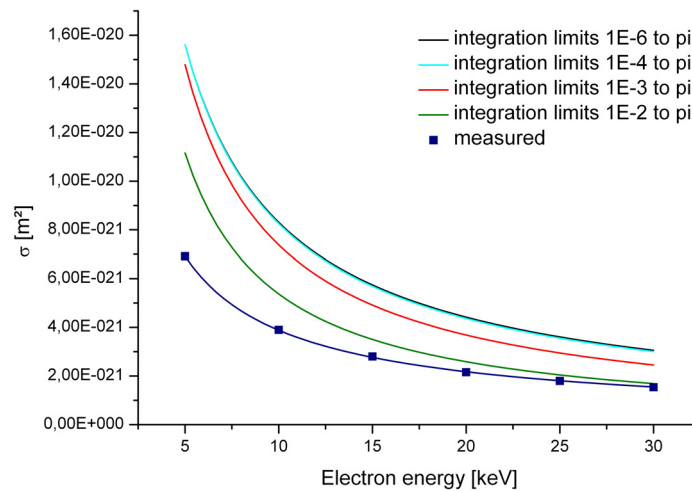


Figure 7.25 Variation of the integration limits for argon (calculated with the program TSCS of monatomic gases (see chapter 11.5.3))

It can clearly be seen that the choice of the lower integration limit plays an important role. A main part of the scattering events result in low angle deflection of the electron trajectories. However, not only the absolute value is influenced but also the curve progression between theory and experiment is different. By decreasing the electron energy the calculated total scattering cross-section increases stronger than the measured total scattering cross-section. Another difference is that the curve progression between monatomic and molecular gases varies. Due to the fact that the experimental conditions are similar for all measurements, the reasons for these differences are certainly the assumptions and simplifications used for the calculations (see chapter 6.1.2). This new scientific insight clarifies the possibility that this

method allows to draw back conclusions to the theory of calculation and to develop an improved understatement.

Direct comparisons between the experimental results and the calculations are difficult. The electrons undergo scattering events all along the BGPL and therefore scattering into a relatively large angle near by the Faraday cup will worsen the results.

Another factor which is important for these comparisons is that the fraction of scattering into small angles is electron energy dependent. The inelastic differential cross-section is responsible for scattering into low angles. This cross-section is decreased by decreasing electron energy, but the angle in which the drop of the total scattering cross-section starts is shifted to higher angles, which lead to an increase in the total scattering cross-section. Because of this shift the increase of the lower integration limit leads not only to a reduction of the total scattering cross-section, the curve progression is also influenced. This effect can be seen in figure 7.26 and figure 7.27.

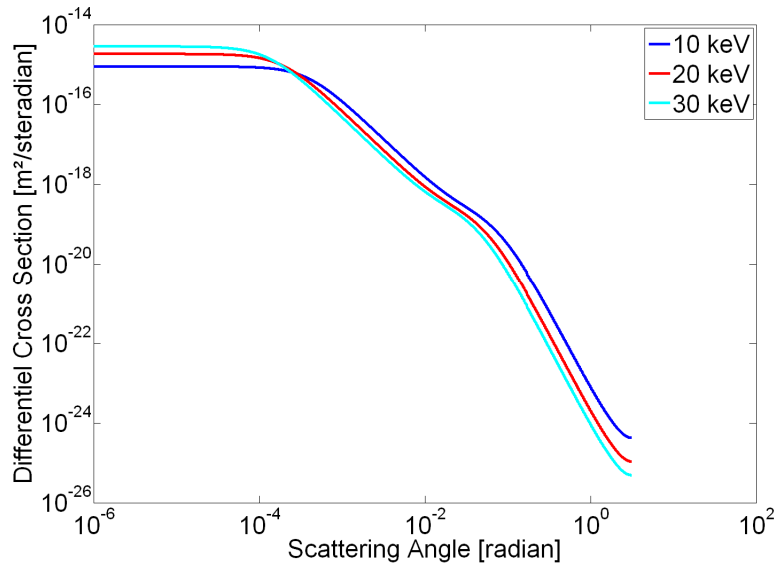


Figure 7.26 Total differential cross-section of argon
(calculated with the program TSCS of monatomic gases (see chapter 11.5.3))

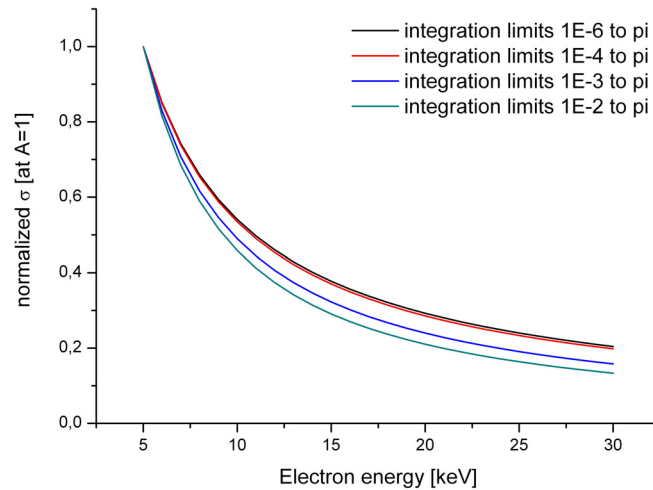


Figure 7.27 Normalised total scattering cross-section of H₂O
(calculated with the program TSCS of molecular gases (see chapter 11.5.4))

Another important fact which must be considered is the correct choice of the ionisation potential and the effective atom radius which is very important for the theoretical calculations. In the publication of Danilatos (1988) the first ionisation potential was used and formula 5.8 for the determination of the effective atom radius. Alternatively, the mean excitation energy J_{mean} and formula 7.6 for the effective atom radius can be used (Reimer, 1998).

$$J_{\text{mean}} = 9.76 \cdot Z + 58.8 \cdot Z^{-0.19} \quad \text{for } Z > 6 \quad 7.5$$

$$J_{\text{mean}} = 11.5 \cdot Z \quad \text{for } Z \leq 6$$

$$R = a_H \cdot Z^{-1/3} \quad 7.6$$

Table 5 Comparison of the ionisation potential and the effective atom radius

	J [eV] (table 1)	J_{mean} [eV] (formula 7.5)	R [m] (formula 6.6)	R [m] (formula 7.6)
H	13.60	11.5	3.74E-11	5.29E-11
N	14.53	108.95	2.88E-11	2.76E-11
O	13.62	117.69	2.58E-11	2.65E-11
Ar	15.76	209.63	2.63E-11	2.02E-11

By variation of the parameters (see table 5) the influence of the mean excitation potential and the effective atom radius can be seen (see figure 7.28-figure 7.31).

Version 1: first ionisation potential (table 5); radius (see formula 6.6)

Version 2: mean ionisation potential (see formula 7.5); radius (see formula 6.6)

Version 3: mean ionisation potential (see formula 7.5); radius (see formula 7.6)

Version 4: first ionisation potential (table 5); radius (see formula 7.6)

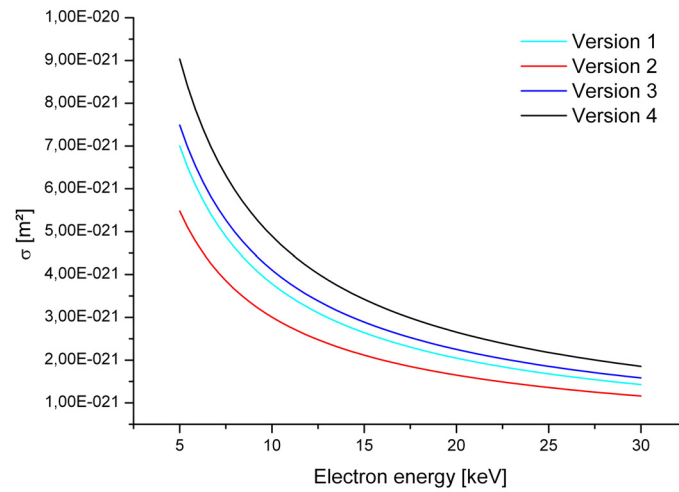


Figure 7.28 Total scattering cross-section as a function of electron energy of H₂O (calculated with the program TSCS of molecular gases (see chapter 11.5.4))

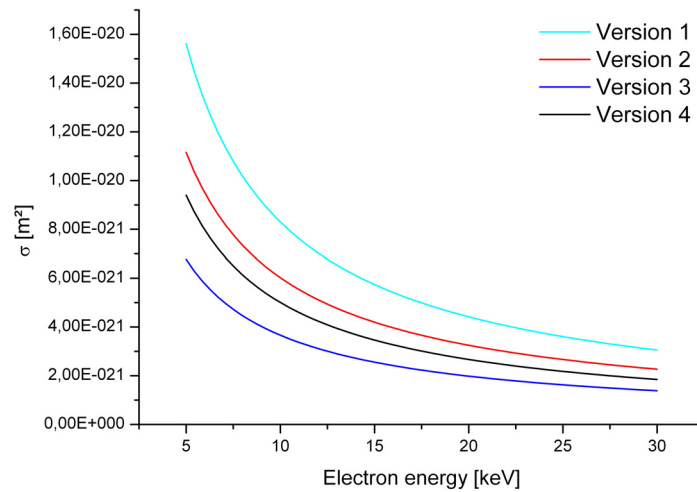


Figure 7.29 Total scattering cross-section as a function of electron energy of argon (calculated with the program TSCS of monatomic gases (see chapter 11.5.3))

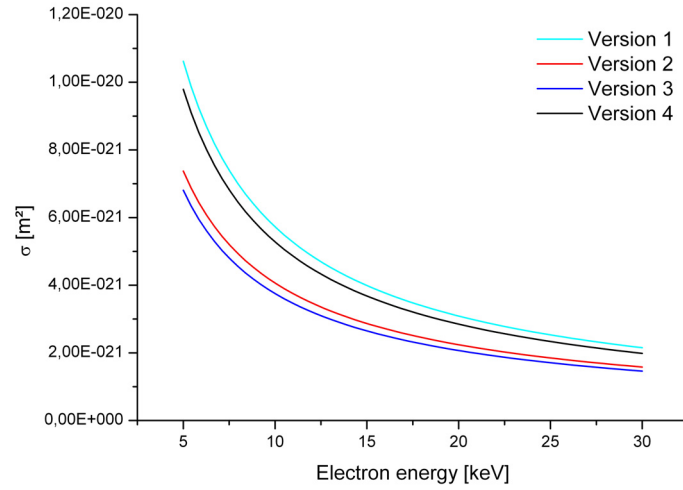


Figure 7.30 Total scattering cross-section as a function of electron energy of N₂ (calculated with the program TSCS of molecular gases (see chapter 11.5.4))

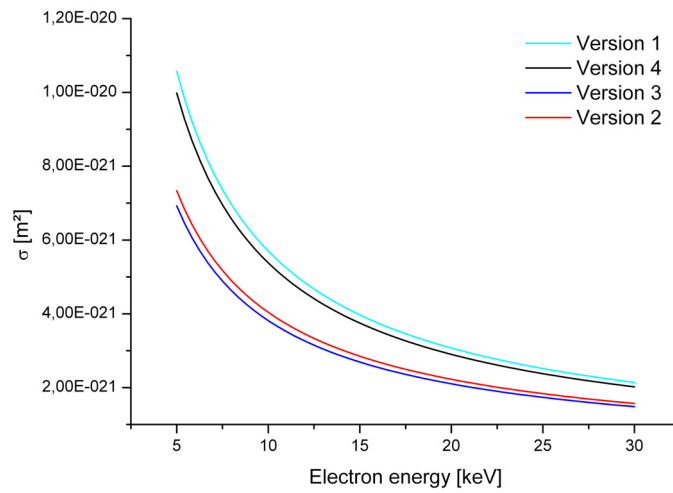


Figure 7.31 Total scattering cross-section as a function of electron energy of ambient air (calculated with the program TSCS of molecular gases (see chapter 11.5.4))

Table 6 Parameter A and B for H₂O, argon, ambient air and N₂

	H ₂ O		argon		ambient air		N ₂	
	A [m ²]	B	A [m ²]	B	A [m ²]	B	A [m ²]	B
V1	2.92E-20	0.887	6.75E-20	0.910	4.44E-20	0.892	4.46E-20	0.891
V2	2.21E-20	0.865	4.66E-20	0.889	2.93E-20	0.860	2.94E-20	0.860
V3	3.02E-20	0.867	2.82E-20	0.886	2.76E-20	0.860	2.71E-20	0.859
V4	3.74E-20	0.883	4.05E-20	0.908	4.19E-20	0.892	4.11E-20	0.891
measured	1.57E-20	0.864	2.65E-20	0.835	2.48E-20	0.904	2.38E-20	0.887

Again, the Faraday cup complicates the comparison because both parameters A and B are influenced by the acceptance angle (formula 7.4).

The parameter A, which is mainly responsible for the absolute value of the total cross-section, is influenced because a lot of the electrons which scatter are detected as unscattered (see table 6). The parameter B, which is responsible for the decrease of the cross-section with increasing electron energy, is influenced because of the scattering angle dependency on the electron energy (see figure 7.26 and figure 7.27).

However, comparison between the different theoretical curves and the experimental determined values can be seen in figure 7.32 and figure 7.33. The parameter A (formula 7.4) is set to 1 and then the different curve progressions are normalised to the highest value at 5 keV. From this point of view it seems that the results for the mean excitation energy represent the scattering conditions in a better way. However, the influence of the acceptance angle is difficult to consider and therefore no clear conclusion can be found.

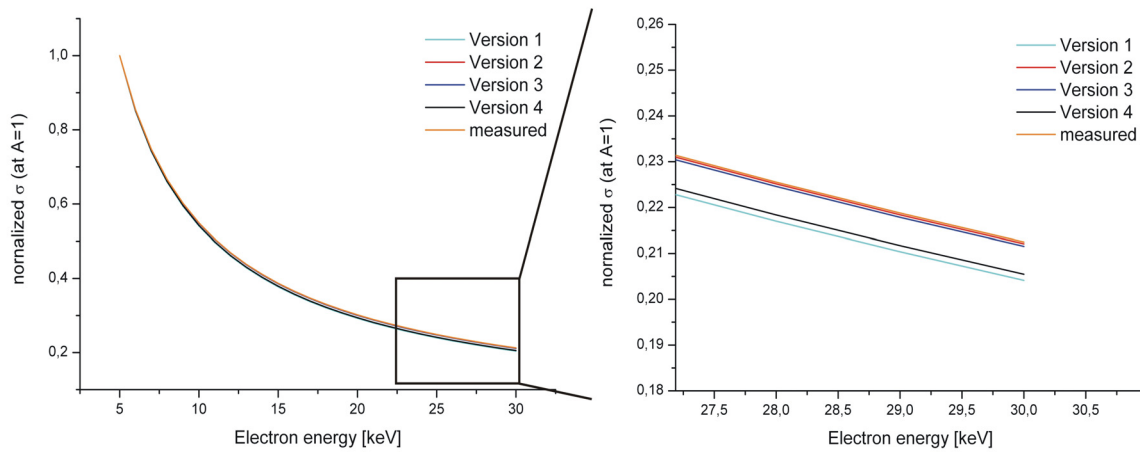


Figure 7.32 Normalised σ_T (at $A=1$) of H_2O

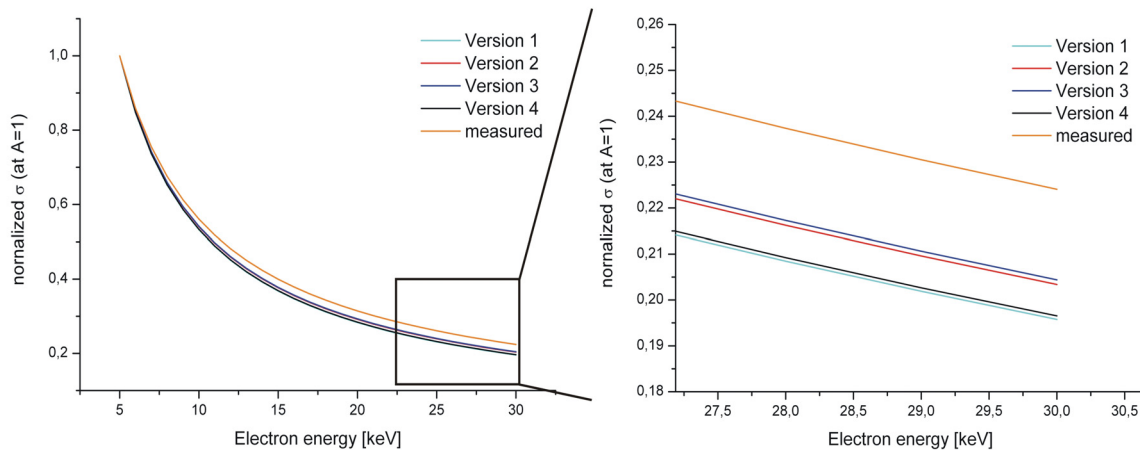


Figure 7.33 Normalised σ_T (at $A=1$) of argon

8 Determination of the Effective BGPL

The distance an electron travels inside the gaseous environment of the chamber is called effective Beam Gas Path Length (BGPL) (Rattenberger et. al., 2008; Rattenberger et. al., 2009). This distance is not equal to the working distance. It depends on the used detector (see figure 3.9) and is pressure gradient dependent. Because of the transition region between high vacuum in the electron column and the low vacuum in the sample chamber, there is even a signal loss before the electron beam passes the PLA. For the following results the concept of the effective BGPL is used. The pressure gradient in the transition region is normalised to the chamber pressure and the scattering of the primary beam electrons starts above the PLA in a pressure regime considered equal to the chamber pressure.

Danilatos et al. (2010) reported Monte Carlo simulations of the beam transmission under low vacuum and ESEM conditions for the ESEM Quanta 600 from FEI and the LEO Supra 35VP from Zeiss. It should be announced that the parameter θ , or stagnation gas thickness, is identical with the term BGPL used in this work and previous publications (Rattenberger et. al., 2009).

The gas flow inside the ESEM Quanta 600 is mainly influenced by the design of the bullet (Danilatos et al., 2010). This component is screwed at the end of the electron column and serves as possible mounting for the GSED. The principal constituents of this bullet are the pressure limiting aperture and the four slotted holes below the PLA which serve as openings for the differential pumping system. In low vacuum and ESEM mode a rotary pump evacuates this area, which guarantees good vacuum conditions in the electron column. The PLA consists of three 400 μm platinum apertures separated by two distance pieces (200 μm thick and 1250 μm diameter). The whole construction can be seen in figure 8.1.

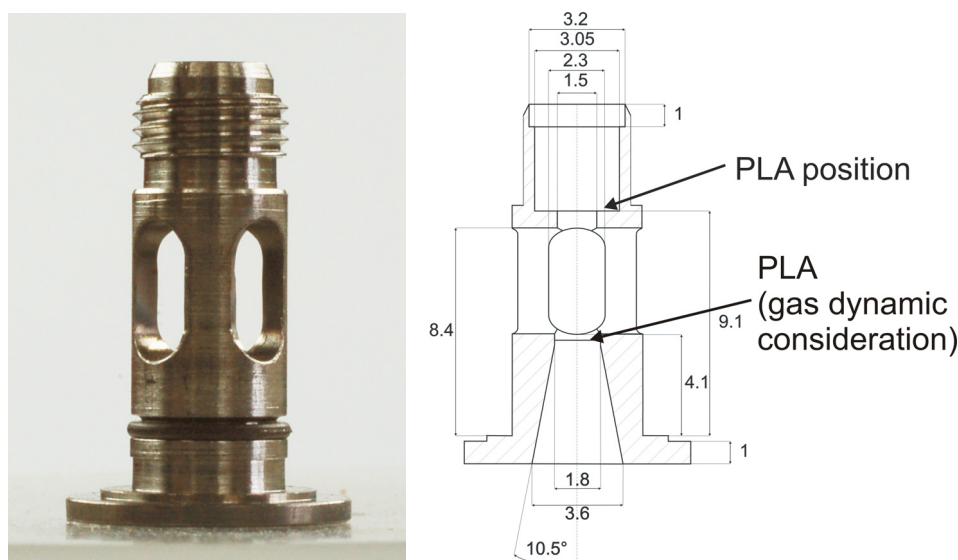


Figure 8.1 Photograph of the ESEM Quanta 600 bullet (left), technical drawing of the bullet (right)

Caused by gas dynamic considerations the 1.8 mm hole below the openings acts as a second PLA (Danilatos et al., 2010) (see figure 8.1). The outcome of this is that in contrast to the general information of the manufacturer, the microscope has two PLAs even under low vacuum conditions.

For a better comparison between the Monte Carlo simulations done by Danilatos et al. (2010) and the experimental results by Rattenberger et al. (2009), backpressure measurements at the slotted holes were necessary. The Inficon device (see chapter 7.2.1) was used to measure this pressure as a function of the chamber pressure using (nitrogen gas). A T-piece was mounted between the column and the backing valve which is closed in high vacuum mode and opened in low vacuum and ESEM mode. With this construction it is possible to measure the backpressure P_1 between the two PLAs (see figure 8.2).

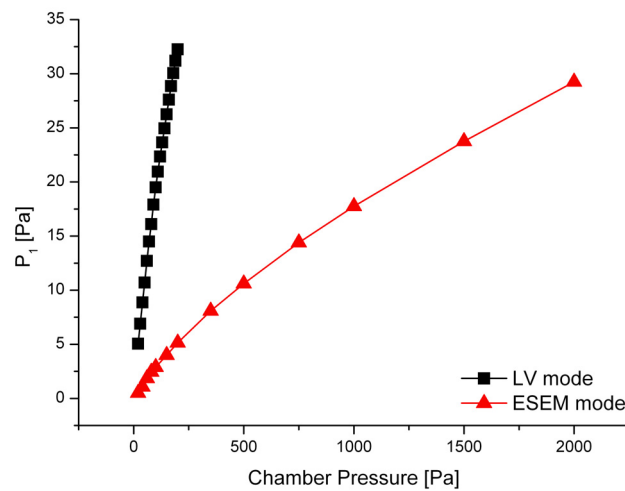


Figure 8.2 Pressure at the slotted holes (P_1) as a function of the chamber pressure in low vacuum and ESEM mode.

It can clearly be seen that the gas flow under low vacuum conditions worsens the microscope capabilities in a fundamental way in comparison with the ESEM mode. The 1.8 mm hole acts as a PLA but the diameter is relatively large in comparison with the 400 μm aperture used in the GSED. The initial value at 20 Pa is one order of magnitude higher in low vacuum mode (0.5 Pa versus 5 Pa) and increases stronger with increasing chamber pressure.

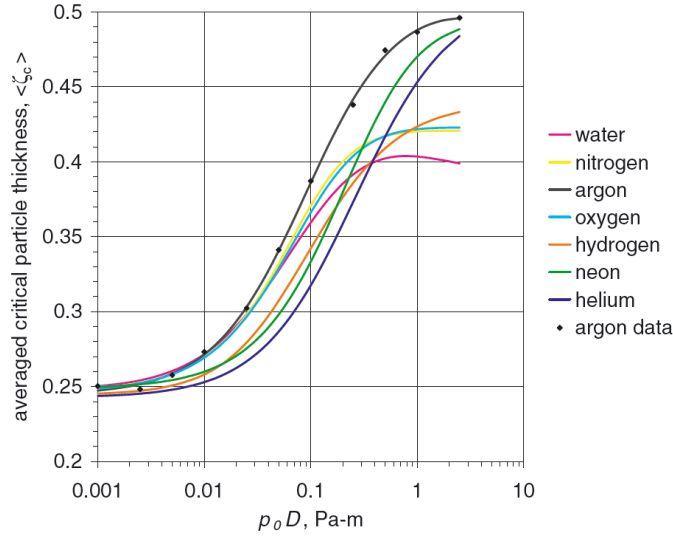


Figure 8.3 Averaged critical particle thickness as a function of $p_0 D$ (Danilatos, 2009)

The physical limit for operating an ESEM is described with the critical particle thickness (Danilatos, 2009). It is defined as the limiting operation case, at 0 mm distance between PLA and specimen. In figure 8.3 the critical particle thickness ξ_c can be seen as a function of the chamber pressure multiplied with the PLA diameter D for several imaging gases.

$$\xi_c = \frac{1}{n \cdot D} \cdot \int_0^\infty n(z) dz \quad 8.1$$

Danilatos (2009) presents the transmission-wise ranking of some different imaging gases: helium>hydrogen>neon>water>oxygen>nitrogen>argon.

In reality this physical limit can not be achieved using the ESEM Quanta 600. Under low vacuum conditions the distance between PLA and specimen is at least 4.1 mm caused by geometrical reasons (see figure 8.1) and the gas flow upwards the PLA is not designed in a perfect way which enhances the scattering above the PLA.

Therefore, the minimum normalised particle thickness $\xi_{\min}$ can be described as:

$$\xi_{\min} = \xi_{fore} + \xi_c + \xi_{aft} \quad 8.2$$

where ξ_{fore} is the particle thickness before the PLA and ξ_{aft} is the particle thickness after the PLA caused by the backpressure.

The BGPL is directly dependant on the particle thickness and the diameter of the PLA:

$$BGPL_c = \xi_c \cdot D \quad 8.3$$

and therefore the minimum normalised beam gas path length $BGPL_{min}$ can be described as:

$$BGPL_{min} = BGPL_{fore} + BGPL_c + BGPL_{aft} \quad 8.4$$

where $BGPL_{fore}$ is the beam gas path length before the PLA and $BGPL_{aft}$ is the beam gas path length after the PLA caused by the backpressure.

For an optimum beam transfer the primary beam loss before and after the PLA should be zero (see formula 8.5).

$$BGPL_{fore} = BGPL_{aft} = 0 \quad 8.5$$

8.1 Experimental Results

With the described Faraday cup it is possible to measure the influence of the pressure gradient on the effective BGPL. In principle there are two methods to determine the effective BGPL.

The first opportunity is to use the x-axis intercept of the best fit straight line of figure 8.4 and formula 7.1. The average number of interactions per electron as a function of the WD must be visualised and the x-intercept gives the difference between the working distance and the BGPL for a defined chamber pressure.

The influence of the pressure gradient on the BGPL can be seen in figure 8.4. With increasing chamber pressure the effective BGPL increases.

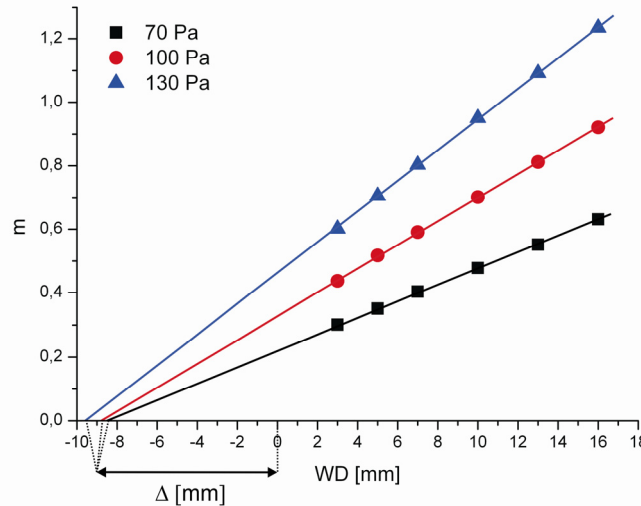


Figure 8.4 Average number of interaction as a function of the WD [mm]

The second opportunity to calculate the effective BGPL is by using the already calculated total scattering cross-section (formula 8.6). The working distance is set to a certain value (for example 10 mm) and the chamber pressure is varied between 40 and 130 Pa in 10 Pa steps.

$$BGPL = \frac{m \cdot k_B \cdot T}{p \cdot \sigma_T}$$

8.6

The measurement accuracy of this method is higher because the influence of the acceptance angle can be neglected. The underestimated average number of interactions m reduces (see formula 7.3 and 8.6).

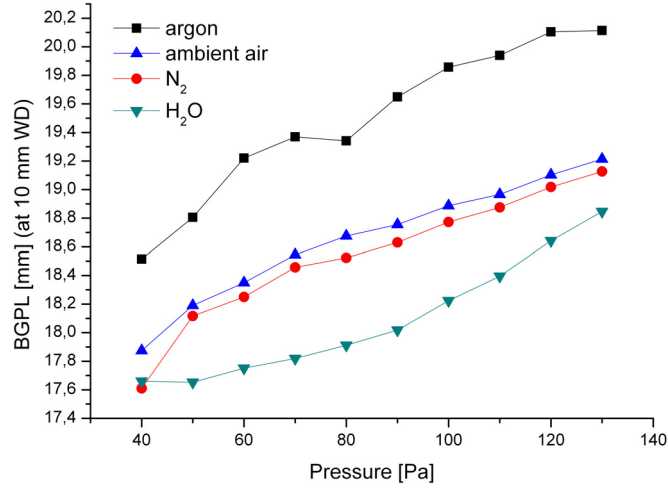


Figure 8.5 Effective BGPL as a function of the chamber pressure [Pa]

The results can be seen in figure 8.5. The BGPL increases with increasing chamber pressure. The different curve progressions for different imaging gases confirm the gas type dependency presented by Danilatos (2009). Argon has a higher effective BGPL, respectively particle thickness (see formula 7.3), than nitrogen or water vapour.

9 Low Voltage Backscatter Imaging

9.1 Introduction

The latest generation of high end scanning electron microscopes enables investigations at very low acceleration voltages or electron landing energies. This method gives scientists the opportunity to analyse their samples in the original condition without the necessity of coating with an additional electrically conducting layer (see figure 3.2) (Reimer, 1993; Joy and Joy, 1996; Frank et al., 2001). By using low electron energies signals from the surface are detected and not a convolution of information from different depths (see figure 9.1). The signal diffusion and the interaction volume are strongly reduced which improves the spatial and lateral resolution of SE images (see figure 9.2). By decreasing the acceleration voltage, the SE yield increases and the partially interfering edge effect is suppressed (Reimer, 1993).

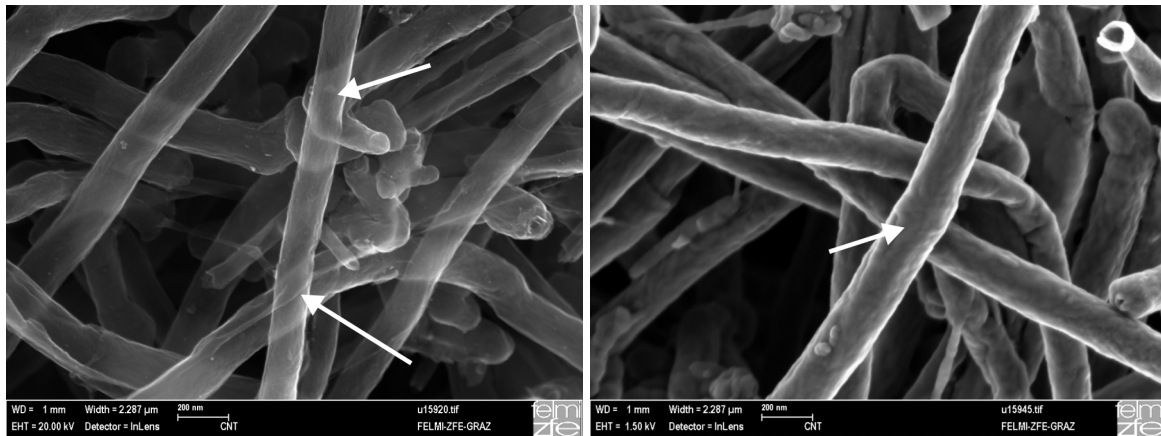


Figure 9.1 SE image of carbon nanotubes (20 keV (left), 1.5 keV (right))

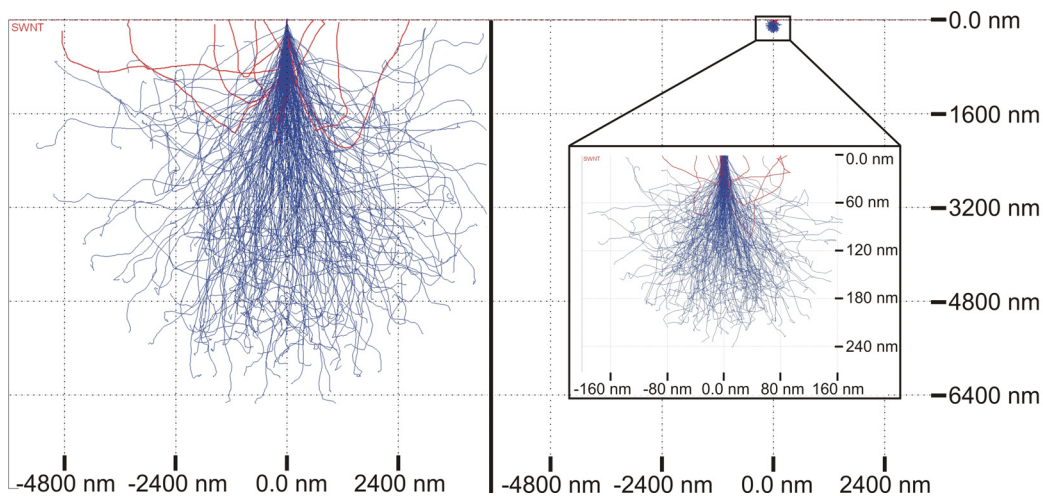


Figure 9.2 Interaction volume in carbon (20 keV (left), 3 keV (right))

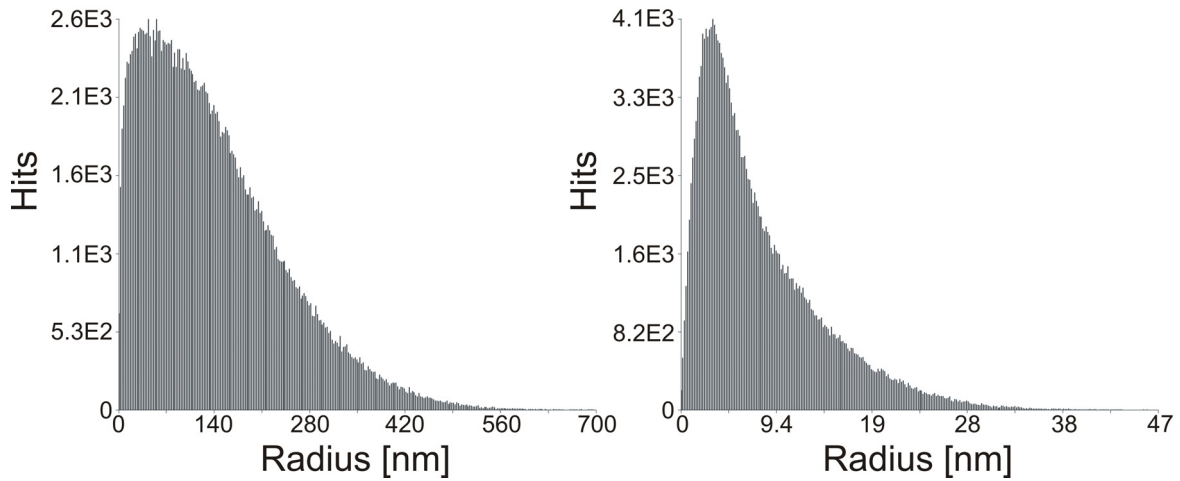


Figure 9.3 Surface radius of BSE (Au sample: left 20 keV; right 3 keV)

On the one hand the decrease in signal diffusion improves the lateral resolution of backscatter images in a direct way. The distance between focused electron probe and emission point of the backscatter electrons is strongly reduced with decreasing electron energy (see figure 9.3).

On the other hand we have to mention that the electron probe size increases caused by chromatic aberration (see chapter 2.3.2).

It is already known that below 1 kV acceleration voltage new contrasts in secondary electron and backscatter electron images appear (Takeuchi et al., 2009; Frank et al., 2000; Reimer, 1993). Surface effects can be investigated but they complicate or avoid the image acquisition or interpretation. Therefore, fundamental research at model-like samples is necessary for the understanding of contrast mechanisms at low acceleration voltages. For the correct interpretation of low voltage images an improved understanding of the secondary electron yield and the backscatter coefficient is necessary.

Specialised detection systems offer the possibility to classify and image high angle and low angle backscatter electrons as well as BSEs with high and low energy loss (Jaksch et al., 2005). These options further complicate the image interpretation (Jaksch, 2008). However, not only backscatter electrons can be dispersed concerning their energy, also secondary electrons can be portioned in consideration of their energy and show different contrasts (Takeuchi et al., 2009). In summary, it can be stated that detailed knowledge of the contrast mechanism and of the detection system is necessary for a correct interpretation of low voltages images.

9.2 The Principle of the Detector

At low electron energies the gun brightness decreases, while the influence of external electric fields (proportional to the time of flight through the column), the chromatic aberration and the

diffraction error increase (Frank, 2002). Therefore, the electron beam should be formed, transported and focused at high energies.

Without the use of a monochromated or Cs-corrected electron column there are two different possibilities to achieve very low electron energies at acceptable aberrations. In both cases the electron beam stays at high energies inside the electron column and a retarding field decelerates the electrons before landing on the specimen.

1. The deceleration can be achieved by using a combined magnetic-electrostatic end lens (see chapter 2.2.2.2) (Frosien et al., 1989). The electron beam is decelerated to its final energy in the gap of the electrostatic lens. This design leads to much smaller spherical and chromatic aberration coefficient in comparison with a conventional system (Weimer and Martin, 1994). The Zeiss Ultra 55 which was used for the following investigations has a combined magnetic-electrostatic end lens and the detection system is described in detail in chapter 2.2.2.2.
2. The other possibility to decelerate the electron beam is by using a cathode lens system. The electrons are decelerated inside the specimen chamber by an axial electrostatic field which is directly applied to the specimen (see figure 9.4). By using this lens system the resolution remains within 2-5 times its value at tens of keV (Frank et al., 2000). The disadvantage of this method is that only specimens with flat surfaces can be investigated which can tolerate a strong electric field.

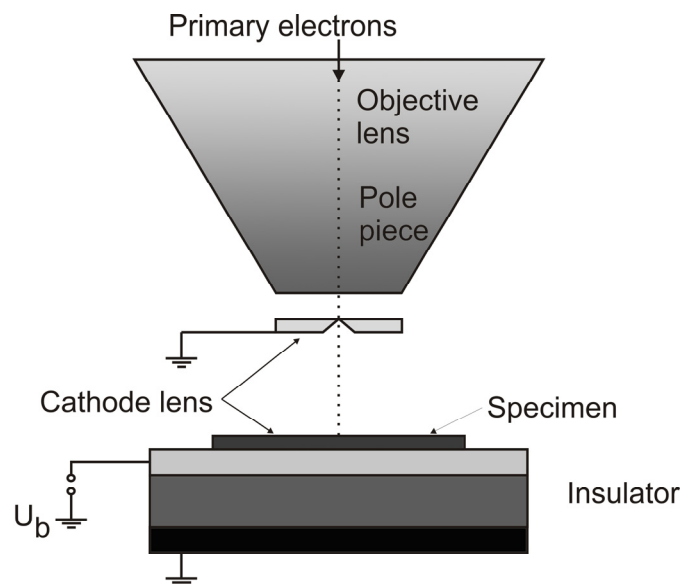


Figure 9.4 Schematic drawing of a cathode lens system for low voltage SEM

The resulting electron energy is achieved by the acceleration voltage of the electron gun subtracted by the deceleration field through the specimen. This retarding field is the reason why in this case the term landing energy is used to describe the energy of the primary electrons. The secondary and backscatter electrons leaving the specimen are

strongly accelerated in the direction of the optical axis. On their way they hit a yttrium-aluminium-garnet (YAG) single crystal and can be detected. To distinguish between secondary and backscatter electrons and to visualise the angular distribution of backscatter electrons a multi-channel detection system can be used (Müllerova and Konvalina, 2006).

The Zeiss Ultra 55 (see figure 9.5) has two low voltage detectors, the Inlens secondary electron detector and the energy selective backscatter detector (EsB). Both detectors are located in the electron column and the primary electron beam passes the detectors by apertures.

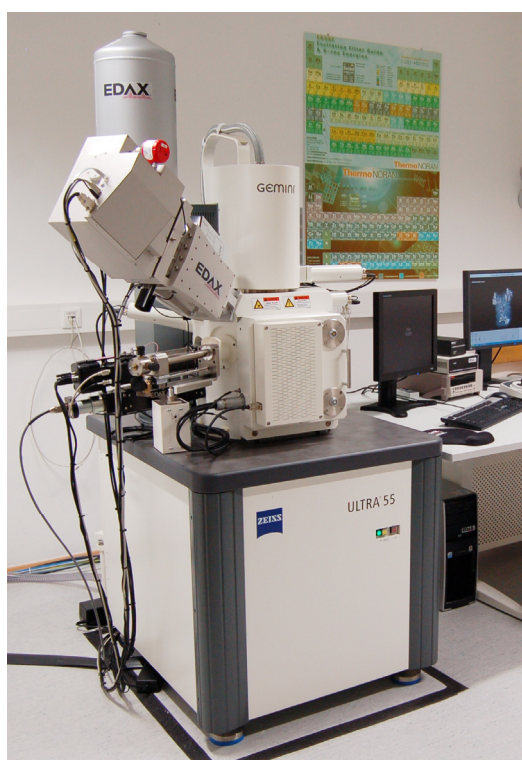


Figure 9.5 The Zeiss Ultra 55 at the Institute for Electron Microscopy and Fine Structure Research

The primary electrons are decelerated within the electron column and therefore no additional retarding field inside the specimen chamber is needed. The secondary and backscatter electrons follow the reverse path inside the column and are there detected by the Inlens or EsB detector (see figure 9.6). Because of their different energies they follow their way up the column on different trajectories which enable the discrimination between backscattered and secondary electrons. The diameter of the Inlens detector aperture is chosen in a way to allow backscattered electrons to pass (see figure 9.6).

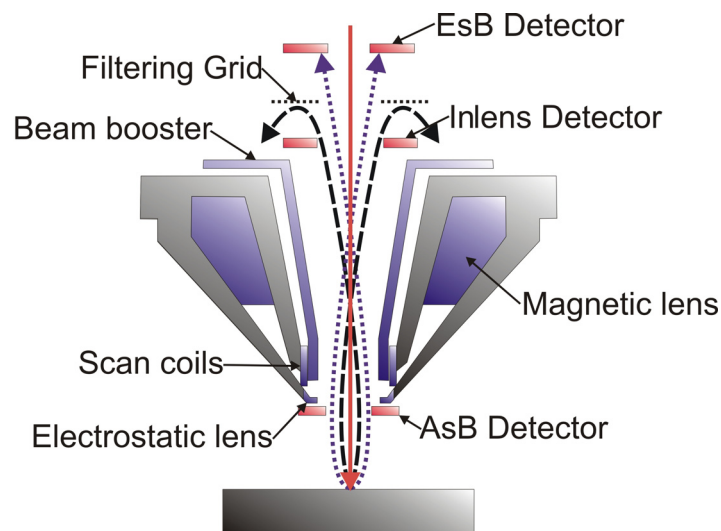


Figure 9.6 Schematic drawing of Zeiss Ultra 55 column (Ackermann, 2004)

The characteristic properties of the Inlens detector are a very good signal to noise ratio at low electron energies and the absence of the typical shadow effect, because of the in-lens symmetric design (Reimer, 1998).

The filtering grid between the two detectors allows the user to filter out the backscattered electrons which have a significant energy loss (Ackermann, 2004; Jaksch, 2008). This grid can be negatively biased by the user up to a voltage of -1500 V. Therefore, it is possible to cut away unwanted secondary electrons and multiple inelastic scattered backscatter electrons. Based on these conditions the EsB detector is capable to detect low loss, high angle backscatter electrons down to an acceleration voltage of 100 V.

There is a lack of literature concerning the collection angle of this low voltage backscatter electron detector and there is no information available from the microscope manufacturer. From this it follows that the collection angle can only be estimated by means of its geometry and the influence of the electrostatic lens on electron trajectories must be neglected. Considering these assumption the collection angle at 2 mm working distance is about 20 degree.

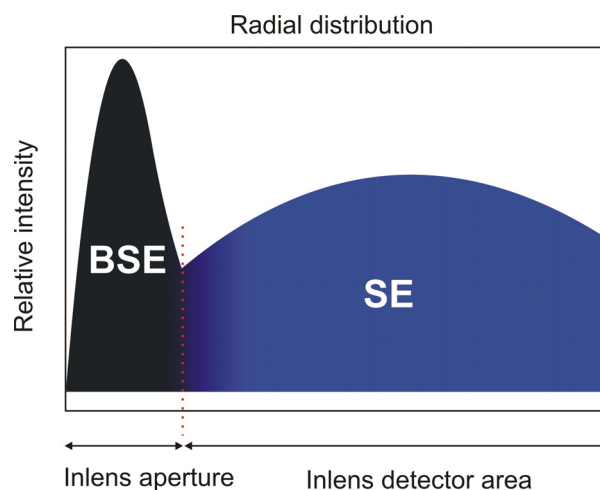


Figure 9.7 Discrimination between SEs and BSEs

By raising the bias of the filtering grid the amount of secondary electrons can be decreased and the lateral resolution of backscatter images is improved by decreasing the penetration depth of the detected backscattered electrons. The multiple scattered BSEs with higher energy losses are deflected by the filtering grid and not detected. Therefore, low voltage backscatter imaging with the EsB detector is a very surface sensitive method because of the low acceleration voltage and because of the possibility to detect low loss backscattered electrons. Figure 9.8 shows how the EsB grid voltage influences the secondary electron amount in EsB images. At 0 V grid bias a superposition of SE and BSE signal is detected. The topography of the sample can be seen as well as weak material contrast from the inorganic particle in the zeolite matrix. By increasing the grid voltage topographic details vanish and the material contrast is strongly increased. At 1000 V grid voltage the linescan clarifies that the SE amount is negligible and the material contrast can clearly be seen without interfering topographic information.

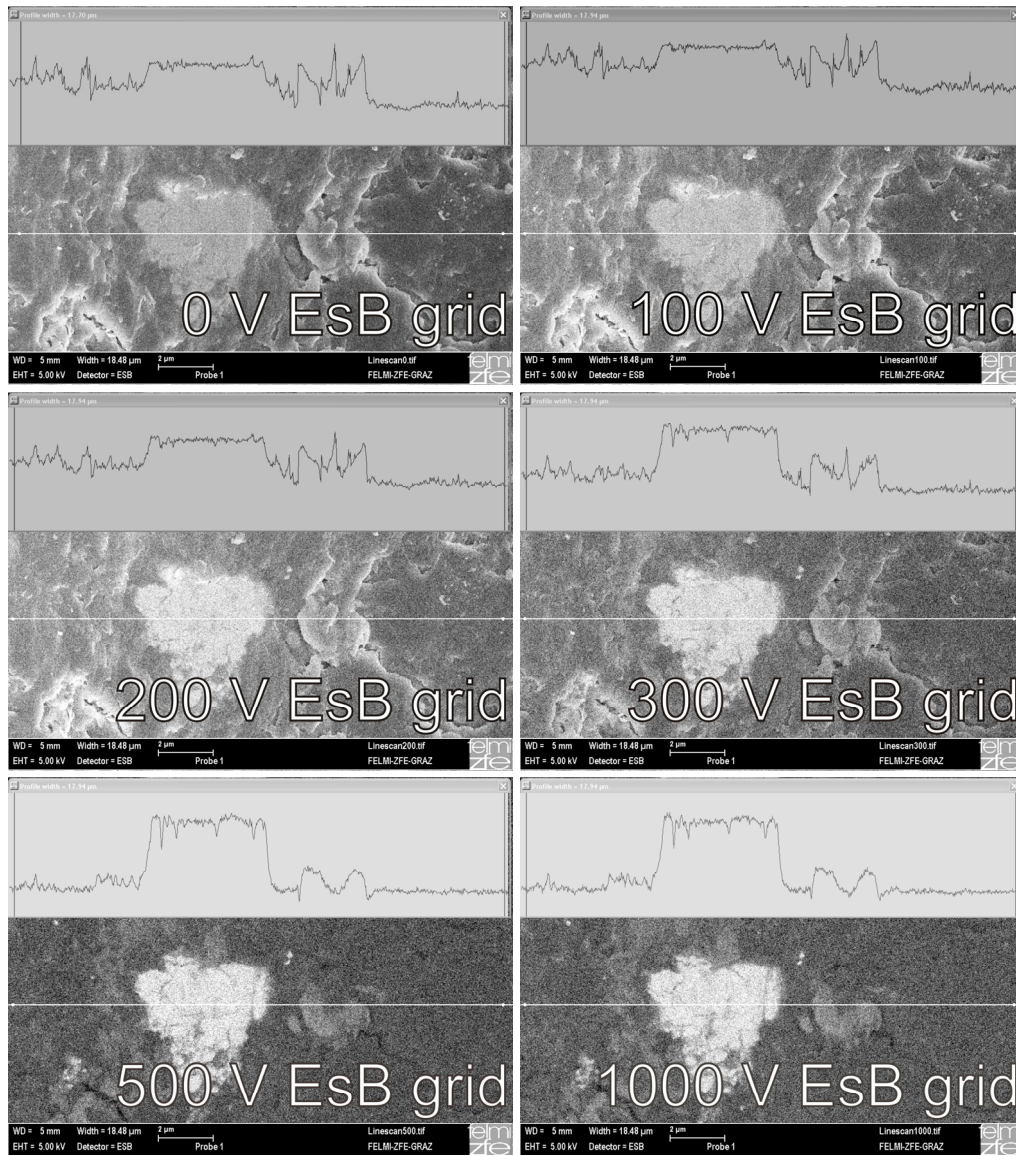


Figure 9.8 Influence of the filtering grid bias on the SE amount in EsB images of inorganic particles in zeolite at 5 keV electron energy (0 V, 100 V, 200 V, 300 V, 500 V, 1000 V)

9.3 Monte Carlo Simulation

In standard works the elastic scattering of electrons in solids is often described by the Rutherford scattering theory (Goldstein et al., 2003). This scattering theory is based on classical mechanics and the electron is treated as a small particle with mass and electric charge (see chapter 4.1.1). From the Rutherford theory we get a nearly monotonic increase of the backscatter coefficient with increasing atomic number (see figure 5.10) (Reimer, 1998). However, this scattering theory neglects the spin orbit coupling of the electrons and shows therefore very often strong variations to the Mott scattering theory which is based on quantum physics. The differences are very high considering high angle scattering at high atomic

number atoms and low electron energies (see chapter 4.1.3). In these cases nonlinear material contrast can be observed. That means chemical compounds of higher atomic numbers appear darker in a backscatter image than lighter elements.

Contrast nonlinearities complicate the interpretation of low voltage backscatter images, in particular because there is no chance to analyse the sample via energy dispersive X-ray spectrometry. Increasing the electron energy is very often not possible due to sample charging or radiation damage. As a consequence the energy is too low to produce a useful amount of X-rays or the electron energy is too low to excite the needed characteristic line. Therefore, different chemical compositions must be interpreted by using backscatter images and in this case the intuitive image consideration is impeded which necessitates Monte Carlo simulations for validation.

The computing power of modern personal computers enables to calculate Monte Carlo simulations very easily which clarifies the interaction volumes and backscatter coefficients. However, especially when taking charging, contamination or inhomogeneities into account, even Monte Carlo simulations get problems, because the complexity of the real sample is unknown or too complex to simulate.

In the last few years the Monte Carlo simulation program Casino 2.42 (Drouin et al., 2007) achieved wide distribution because of the convenient handling and the possibility so simulate grain boundaries, surface layers and self designated chemical compositions. The user can adapt the simulations with experimental microscope parameters (e.g. electron energy and spot size) and can choose between different total cross-sections, partial cross-sections, effective ionisation potentials as well as simulation parameters like random number generators.

In this work the program CASINO 2.42 was used to simulate interaction volumes and backscatter coefficients for the comparisons with the experimental results.

9.4 Material Contrast

To simplify the way of looking at the problem a model-like sample was used. The substrate is a silicon wafer followed by a 50 nm evaporated carbon layer and a 50 nm sputtered gold layer (see figure 9.9). Even for 1 keV electron energy the escape depth of backscattered electrons is not larger than 10 nm which guarantees the material contrast from the highly pure element (see figure 9.10). Parts of the gold layer were removed and the contrast characteristics between these two elements were measured with low voltage backscatter images between 5000 eV and 100 eV.

By adjusting the filtering grid voltage a tradeoff between signal intensity and signal quality is necessary. By increasing the grid voltage the signal to noise ratio declines but the amount of unwanted secondary electrons and multiple scattered backscatter electrons decreases. There is no literature concerning the filtering grid voltage available and therefore the grid voltage was estimated to 90 % of the acceleration voltage.



Figure 9.9 Schematic drawing of the carbon/gold layer system

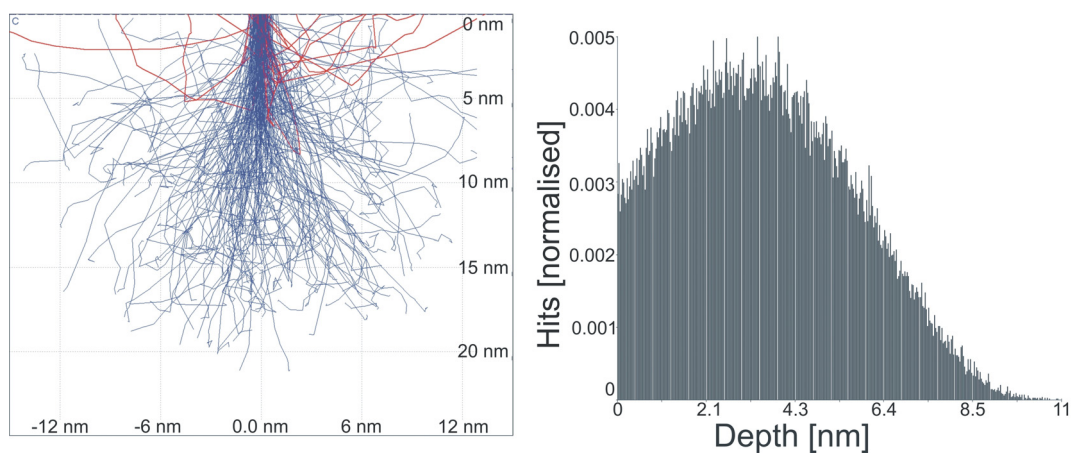
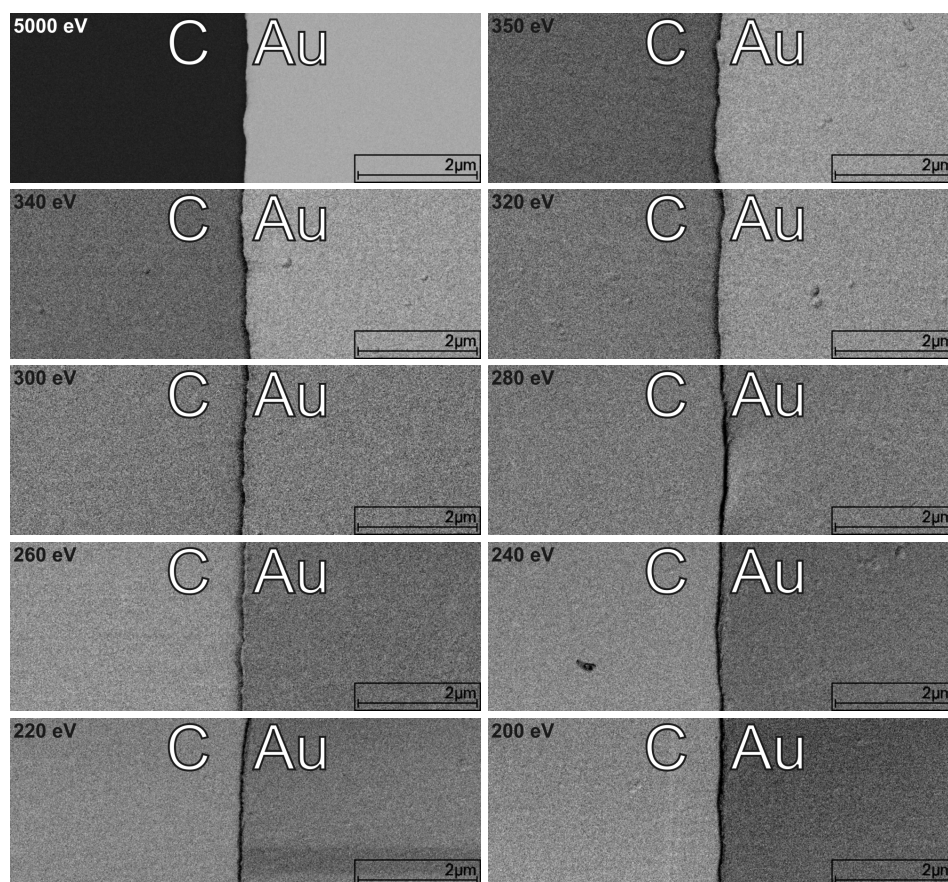


Figure 9.10 Interaction volume of 1 keV electrons in a carbon substrate (backscattered electrons (red)) (left) and escape depth of the backscattered electrons



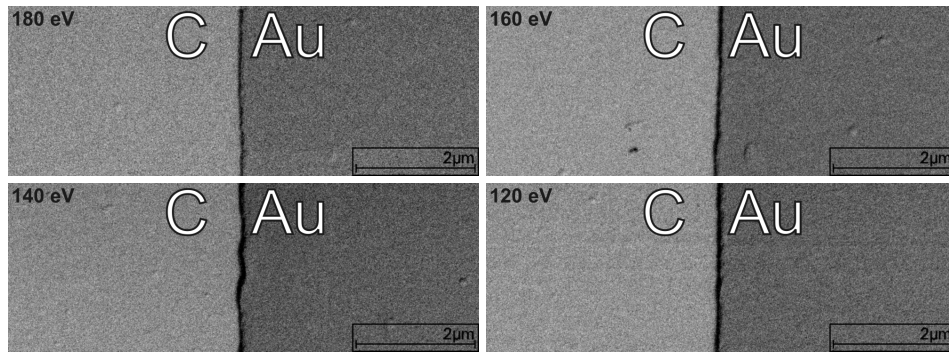


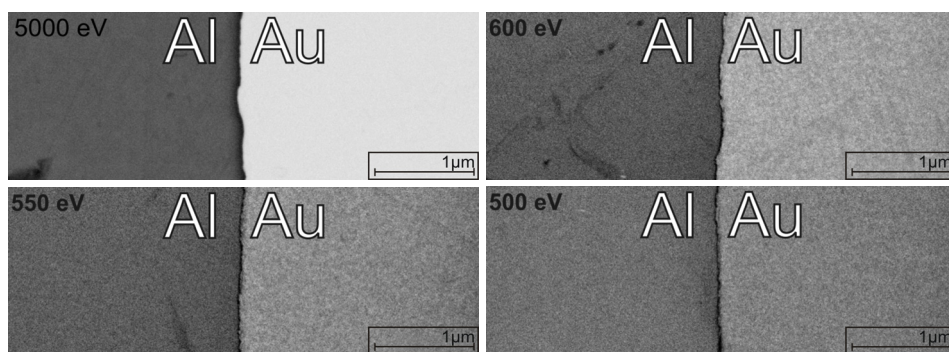
Figure 9.11 50 nm gold layer (right) on 50 nm carbon layer (left) measured with acceleration voltages between 5000 eV and 120 eV

At around 290 eV a contrast inversion of this sample can be detected. A nonlinear material contrast at a difference of 73 atomic numbers can be seen clearly (see figure 9.11) (Rattenberger et al., 2010).

Another example for nonlinear material contrast is a sputtered gold layer on a polished aluminium plate (see figure 9.12). Again parts of the sputtered gold layer were removed to achieve a good material contrast and a sharp changeover of the chemical composition. In figure 9.13 the contrast crossover can be seen. At around 410 eV aluminium becomes brighter in the backscatter image and a nonlinear material contrast can be observed



Figure 9.12 Schematic drawing of the gold layer and the polished aluminium plate



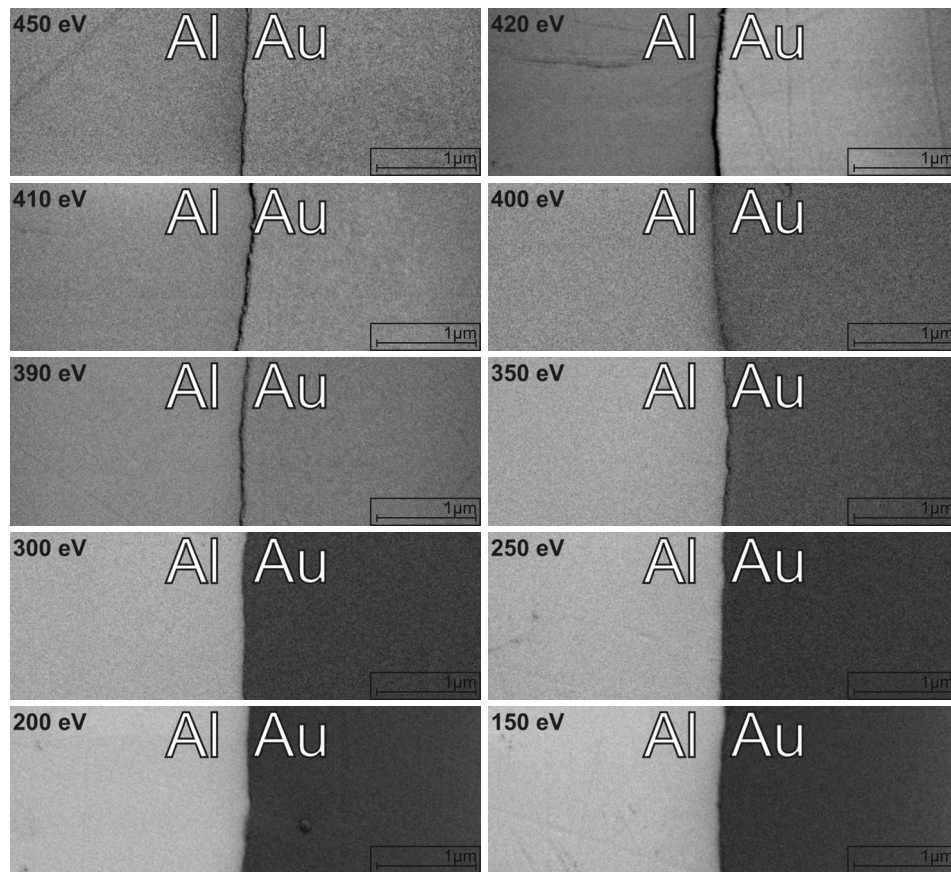


Figure 9.13 50 nm gold layer (right) on a polished aluminium plate (left) measured with acceleration voltages between 5000 eV and 150 eV

Another example for nonlinear material contrast inversion is silver and gold. A “Gasentladungsapparatur” was used to sputter the silver and gold layers alternately on a silicon wafer. The layers were sputtered at 1.8 kV and $9 \cdot 10^{-2}$ Pa argon pressure (Jakobic et al., 1978). The focus ion beam microscope (FIB Nanolab Nova 200, FEI) was used to lift a block out of the multilayer system (Schaffer and Wagner, 2008) (see figure 9.14 and figure 9.15) (FIB parameter: 0.5 - 7 nA, 30 keV ion energy).

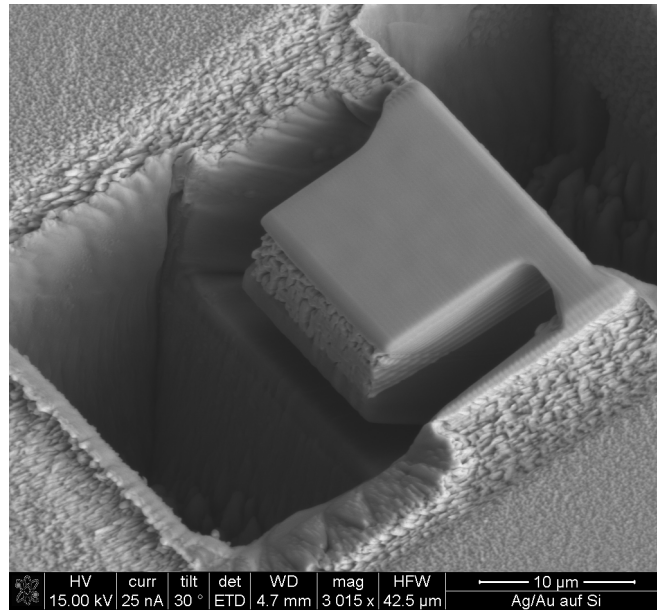


Figure 9.14 Block lift out of the silver/gold multilayer system

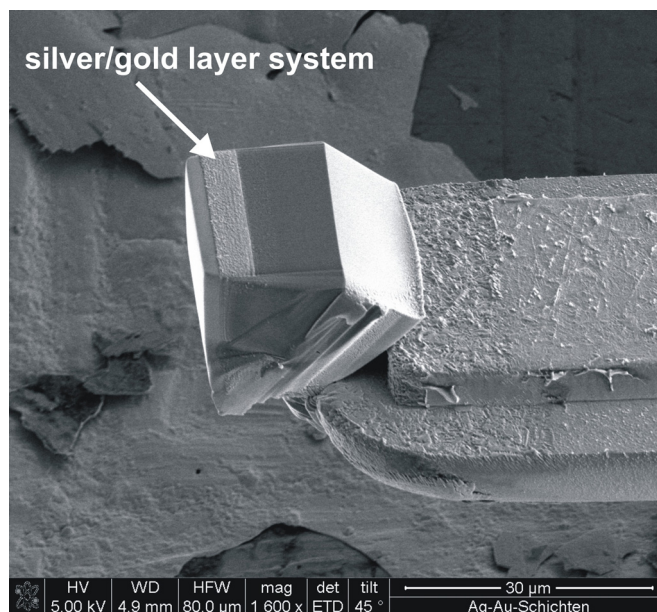


Figure 9.15 Mounted silver/gold multilayer block on a TEM grid

After the lift out, the block was mounted on a TEM grid and transferred into the Zeiss Ultra 55 for the low voltage imaging. As substrate a silicon wafer was used which can be seen as dark area at the right side of the backscatter images (see figure 9.16).

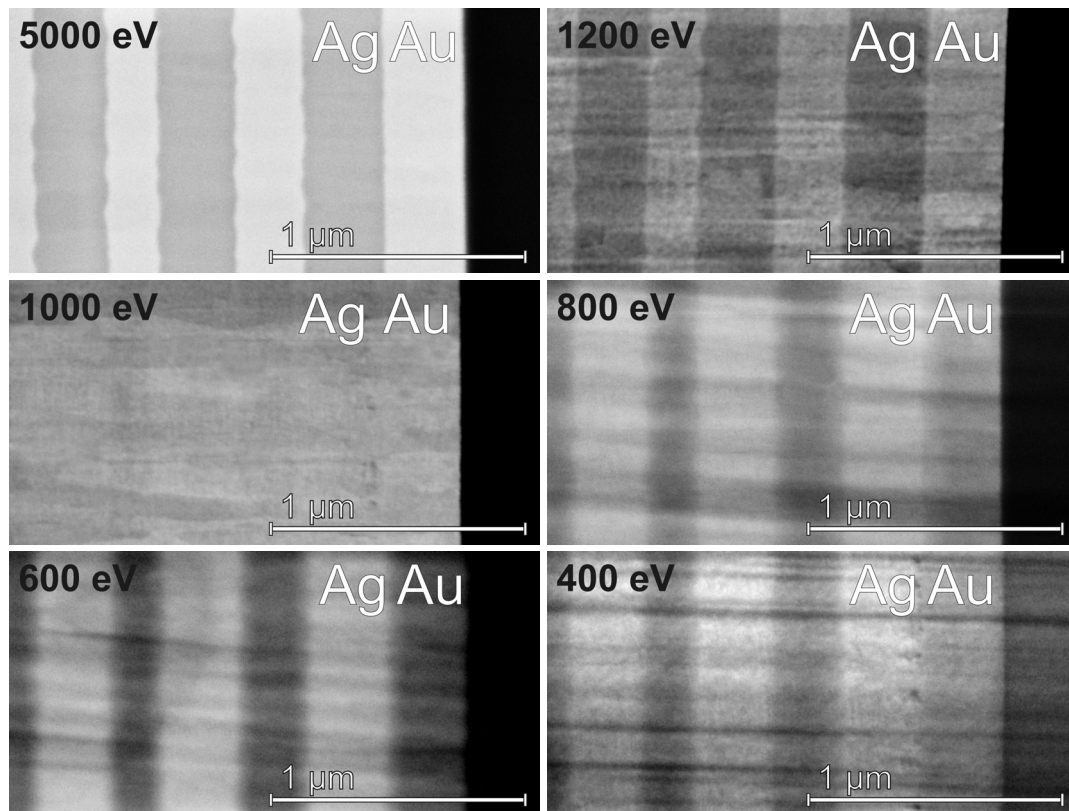


Figure 9.16 Low voltage backscatter images of a sputtered silver and gold multilayer system measured with acceleration voltages between 5000 eV and 400 eV

Redeposition of the milled material is responsible for the curtain effect, which is the reason for the streaks in the low voltage images (Orloff et al., 2002). This topographic information is evidence that unwanted secondary electrons were detected.

However, again an inversion in the material contrast can be seen. At about 1000 eV the contrast disappears and below this energy the sputtered silver layers appear brighter than the gold layers in the backscatter image.

Another example for material contrast inversion can be seen in figure 9.17. A 50 nm gold and a 50 nm iron layer was sputtered on a silicon wafer and parts of the iron layer were removed.

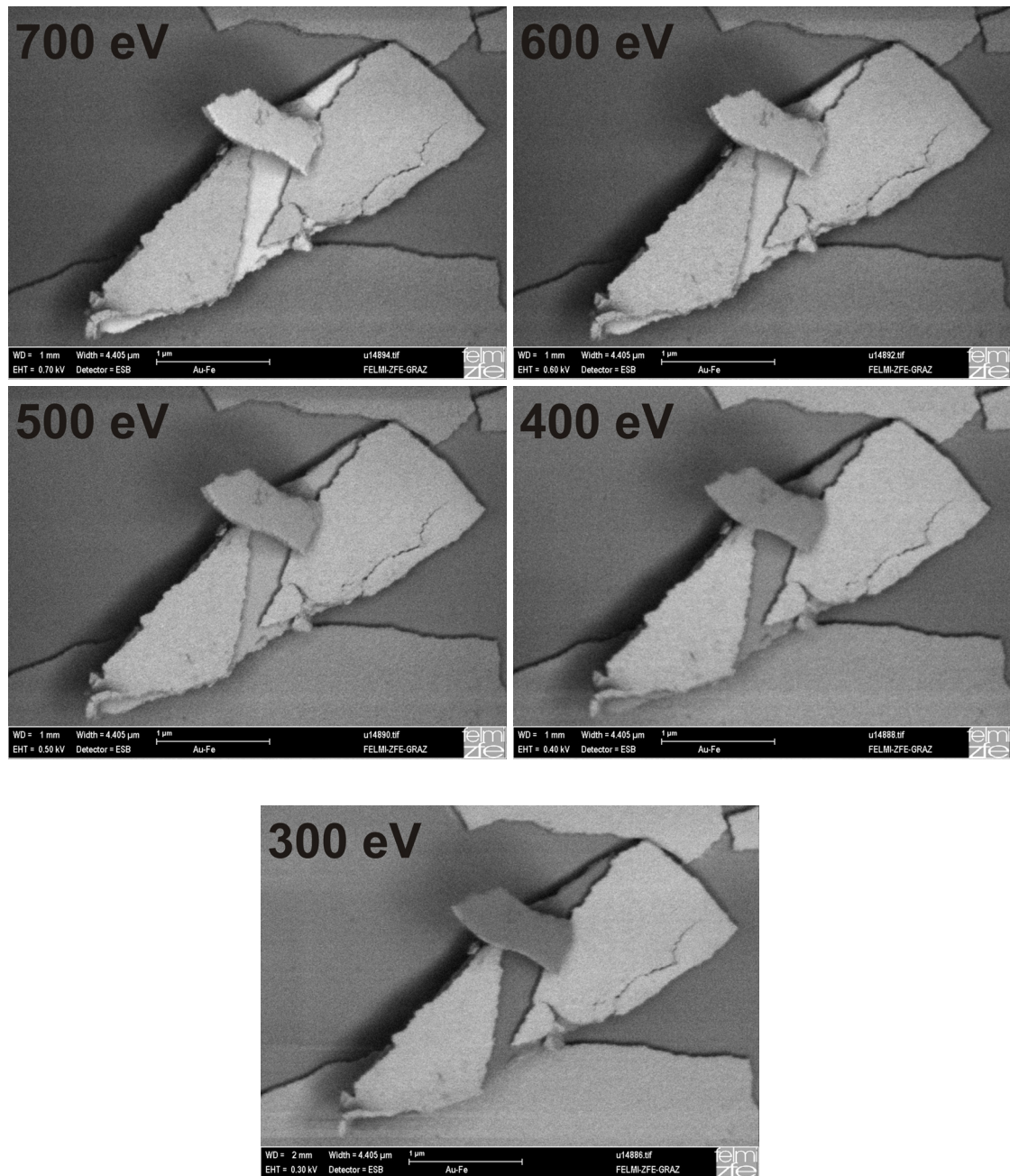


Figure 9.17 Low voltage backscatter images of a sputtered iron/gold system on a silicon wafer measured with acceleration voltages between 700 eV and 300 eV

In the 700 eV backscatter image the gold layer in the middle appears much brighter than the left and right situated iron layer. By decreasing the electron energy the contrast declines and between 600 and 500 eV the contrast inverts.

9.5 Comparison and Interpretation

To visualise the complex differential cross-section of the investigated samples the program MOTT was developed (source code see chapter 11.5.1). In figures 8.18 to 8.21 the Mott differential cross-section per atom as a function of the electron energy for carbon, gold, aluminium and silver can be seen. The complexity of the orbital structure of gold and silver is reflected which results in deep minima and maxima. By contrast, carbon and aluminium do not show these strong angular dependencies.

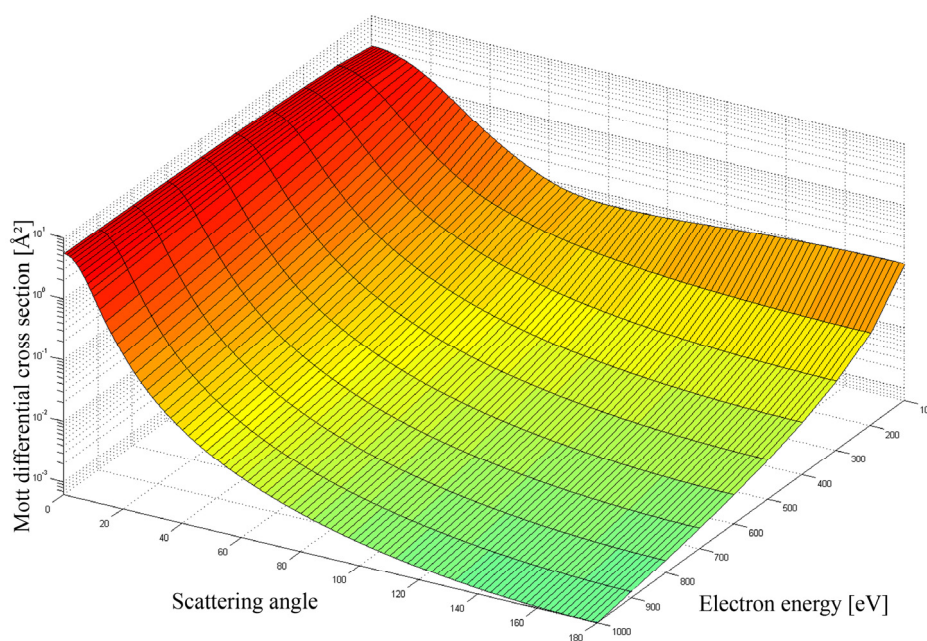


Figure 9.18 Elastic differential cross-section of Carbon
(calculated with the program MOTT (see chapter 11.5.1))

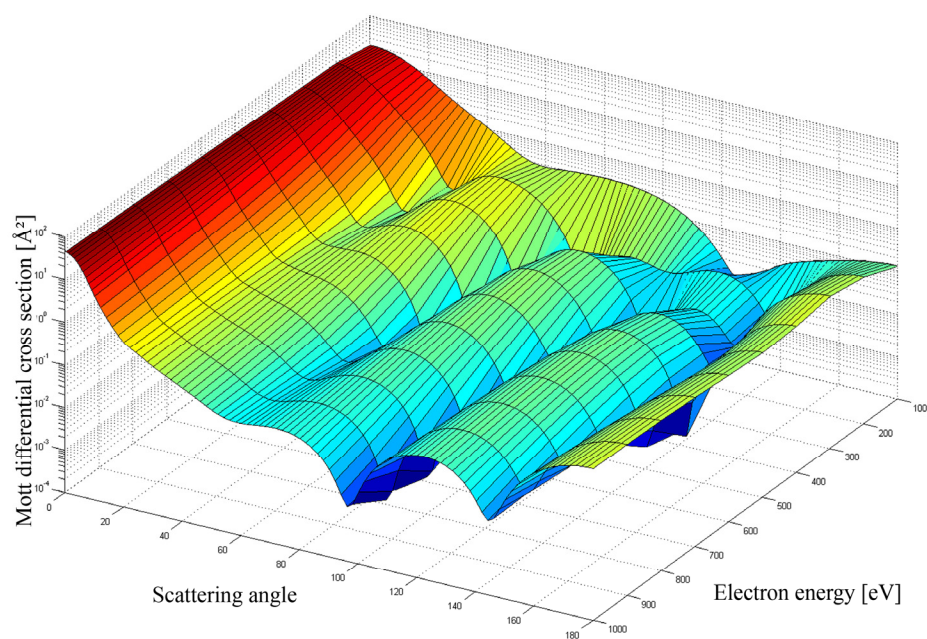


Figure 9.19 Elastic differential cross-section of gold
(calculated with the program MOTT (see chapter 11.5.1))

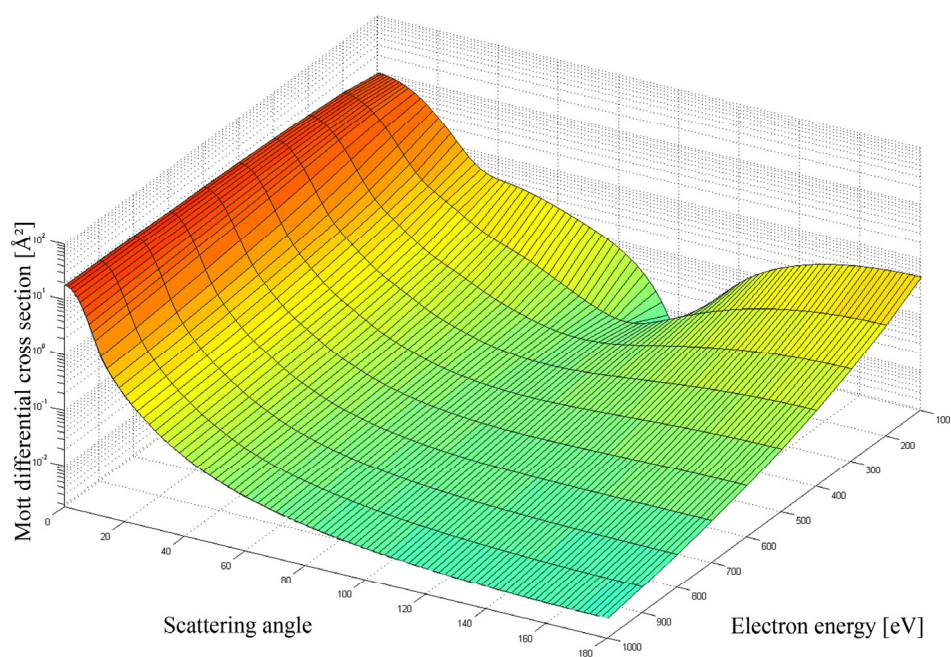


Figure 9.20 Elastic differential cross-section of aluminium
(calculated with the program MOTT (see chapter 11.5.1))

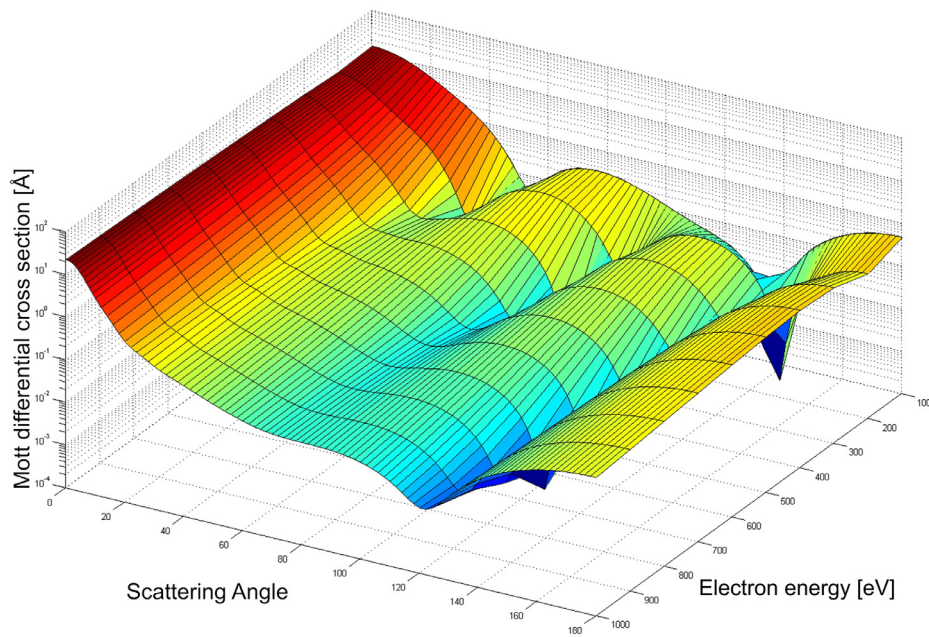


Figure 9.21 Elastic differential cross-section of silver
(calculated with the program MOTT (see chapter 11.5.1))

However, these differential cross-sections themselves can not explain the contrast inversion observed at low voltage backscatter images. The dense packing of the atoms is neglected and therefore Monte Carlo simulations are necessary for the correct comparison. In figure 9.22 the possible contrast inversions considering the differential cross-section per atom can be seen (source code see chapter 11.5.2). The differential cross-section was integrated between 90 and 180 degree using equation 6.3. It can be seen that for carbon and gold no contrast inversion is predicted using the differential cross-section per atom.

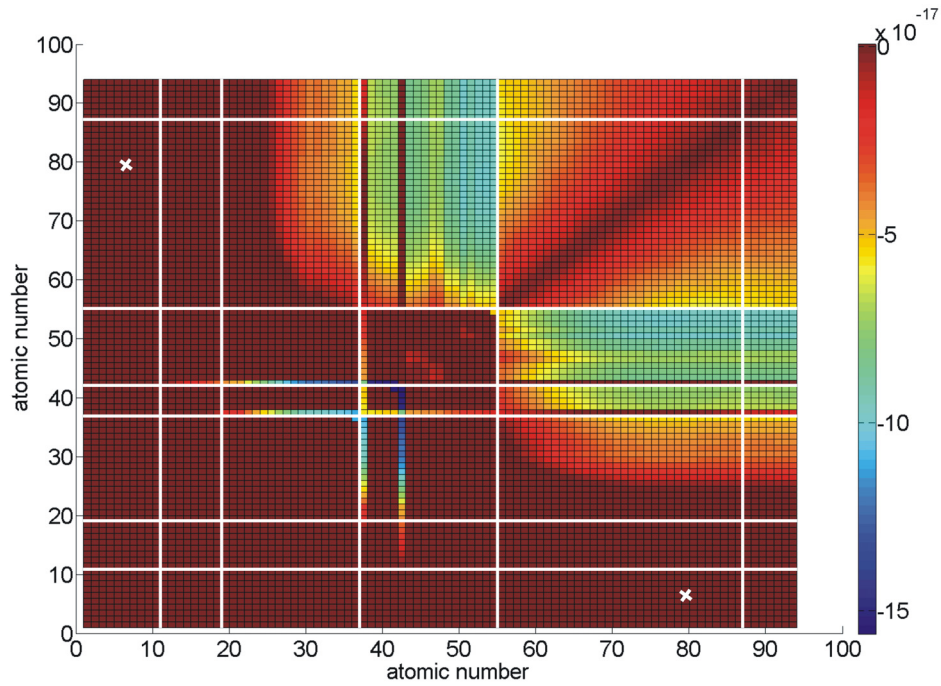


Figure 9.22 Possible contrast inversions considering the Mott differential cross-section per atom (calculated with the program MOTT (inversion) (see chapter 11.5.2))

For the Monte Carlo simulations the program Casino 2.42 was used. 2 million electrons were simulated to achieve a good statistic and the calculated backscatter coefficients were used for the comparison with the experimental results. For the simulation the standard density values of the program were used and for the total and partial cross-sections the different available options were compared. For the effective ionisation cross-section the Casnati option and for the ionisation potential the Joy and Luo option was used (Casnati et al., 1982; Joy and Luo, 1989). The results of the backscatter coefficients as a function of electron energy are shown in figures 8.23 to 8.25 for carbon and gold.

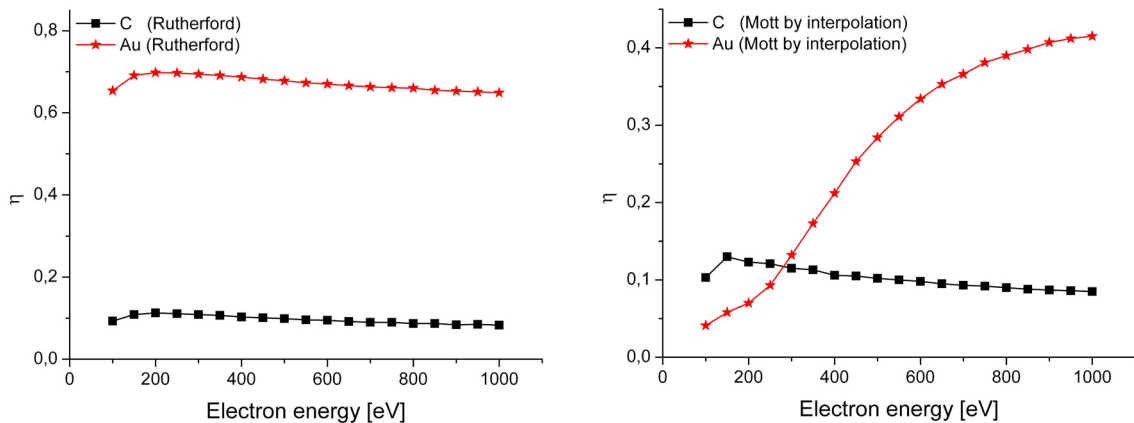


Figure 9.23 Backscatter coefficient of carbon and gold (calculated with Casino 2.42)
Rutherford (left); Mott by interpolation (right))

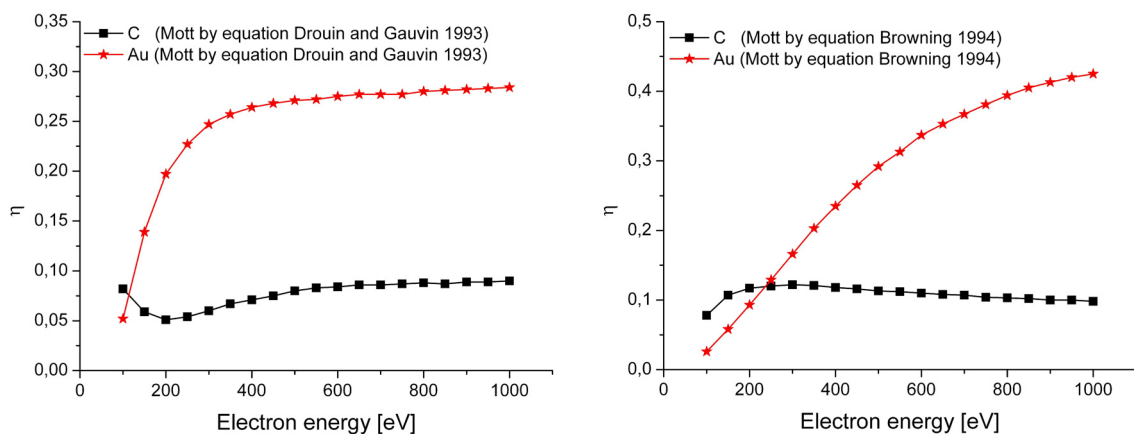


Figure 9.24 Backscatter coefficient of carbon and gold (calculated with Casino 2.42) (Mott by equation (Drouin and Gauvin, 1993) (left); Mott by equation (Browning et al., 1994) (right))

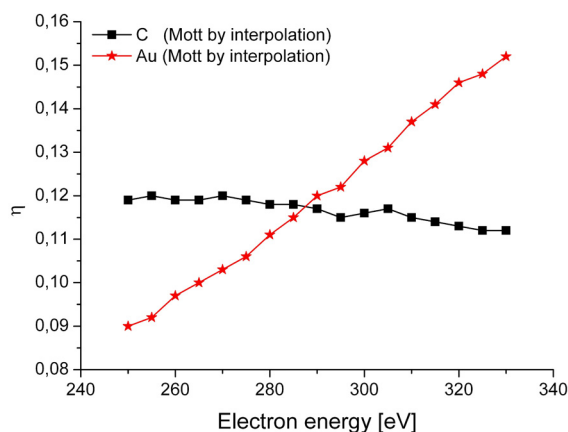


Figure 9.25 Backscatter coefficient of carbon and gold (Mott by interpolation)

Using the Mott by interpolation option the cross-over of the backscatter coefficient is at about 285 eV, using the equation by Browning option at 240 eV, using the equation by Drouin and Gauvin (1993) at 115 eV. Therefore, the Mott by interpolation option agrees at best with the experimental results. Calculating the backscatter coefficients with the Rutherford scattering theory gives completely different results. The simplifications of this model are too extensive to represent the real interaction processes at low electron energies and no contrast inversion is predicted.

For the aluminium/gold layer system the Monte Carlo simulations using the Mott by interpolation option indicate a cross-over at about 410 eV which is again in good comparison with the experimental results (~ 410 eV). Using Browning's equation gives a cross-over energy at 420 eV and using Drouin and Gauvin's equation an energy of 115 eV. The backscatter coefficients as a function of electron energy can be seen in figure 9.26 and figure 9.27.

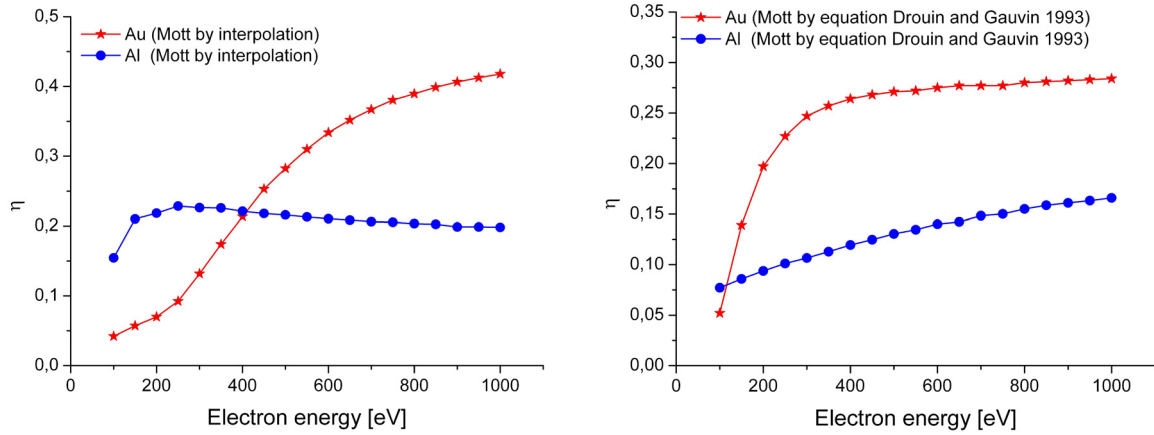


Figure 9.26 Backscatter coefficient of aluminium and gold (Mott by interpolation (left); Mott by equation (Drouin and Gauvin, 1993) (right))

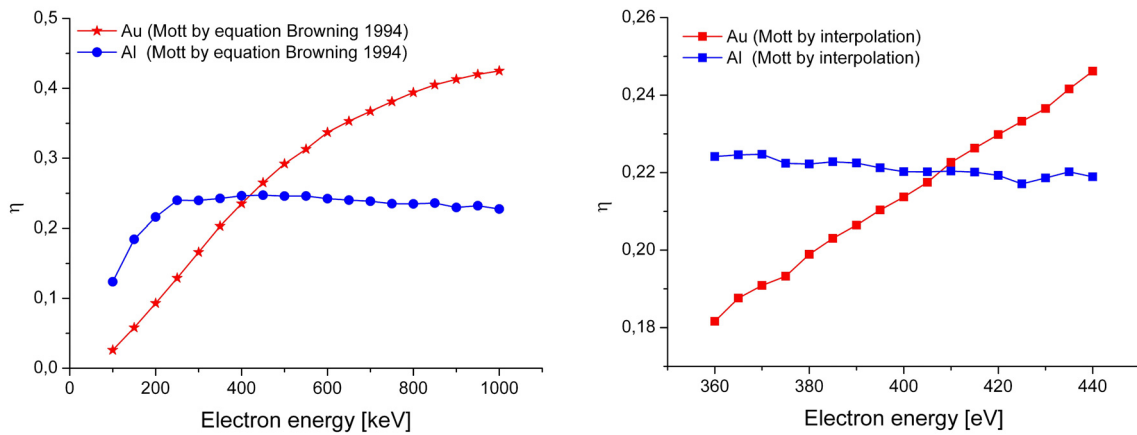


Figure 9.27 Backscatter coefficient of aluminium and gold (Mott by equation (Browning et al., 1994) (left); Mott by interpolation (right))

In the case of the silver/gold sputtered multilayer system the results vary. From the Monte Carlo simulation using Mott by interpolation the contrast inversion should occur at about 730 eV, but the low voltage backscatter images show the contrast disappearance marginal below 1000 eV. The result for using Browning's equation is 840 eV and for Drouin's and Gauvin's equation 340 eV (figure 9.28 and figure 9.29).

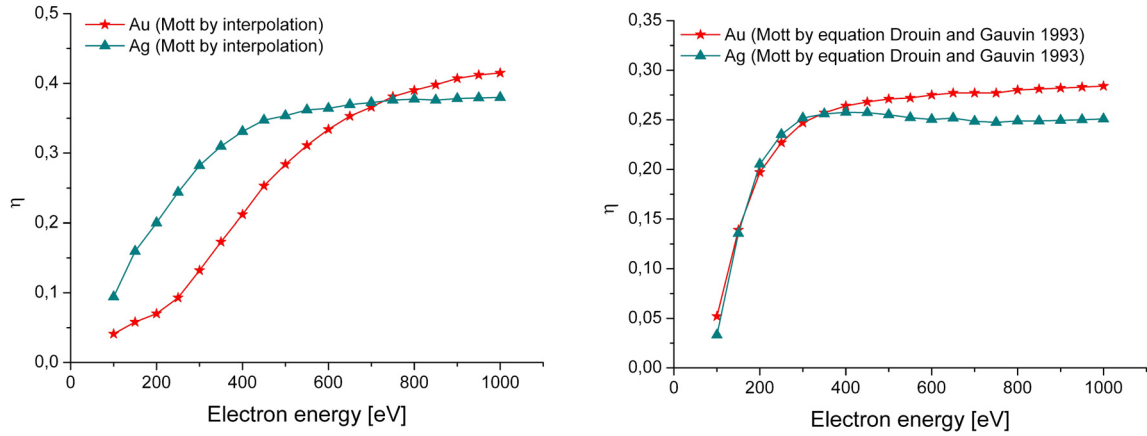


Figure 9.28 Backscatter coefficient of silver and gold (Mott by interpolation (left); Mott by equation (Drouin and Gauvin, 1993) (right))

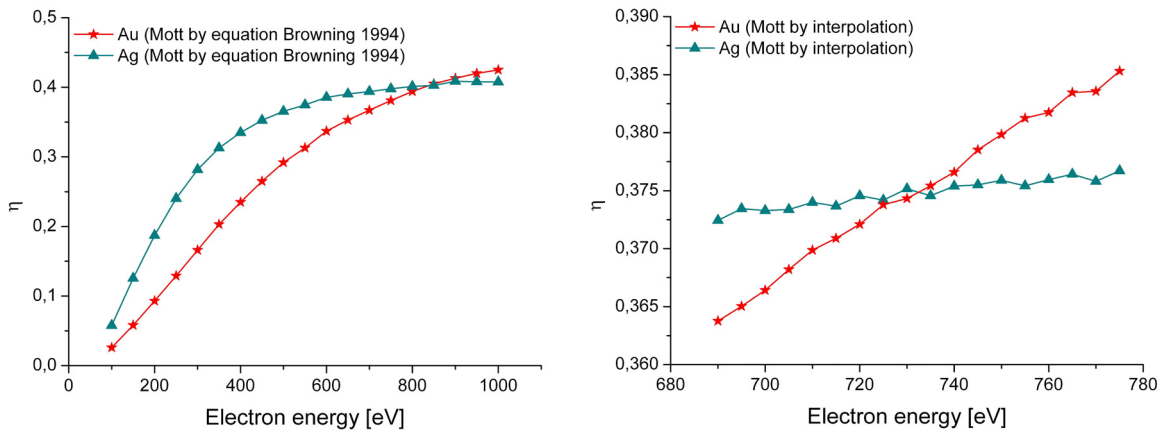


Figure 9.29 Backscatter coefficient of silver and gold (Mott by equation (Browning et al., 1994) (left); Mott by interpolation (right))

For the iron/gold layer system the experimental results are in good comparison with the Monte Carlo simulations. Again the Mott by interpolation option (565 eV) agrees very well with the experimental results (about 550 eV). The equation by Drouin and Gauvin predicts no inversion in material contrast and Browning's equation a cross-over energy of 600 eV.

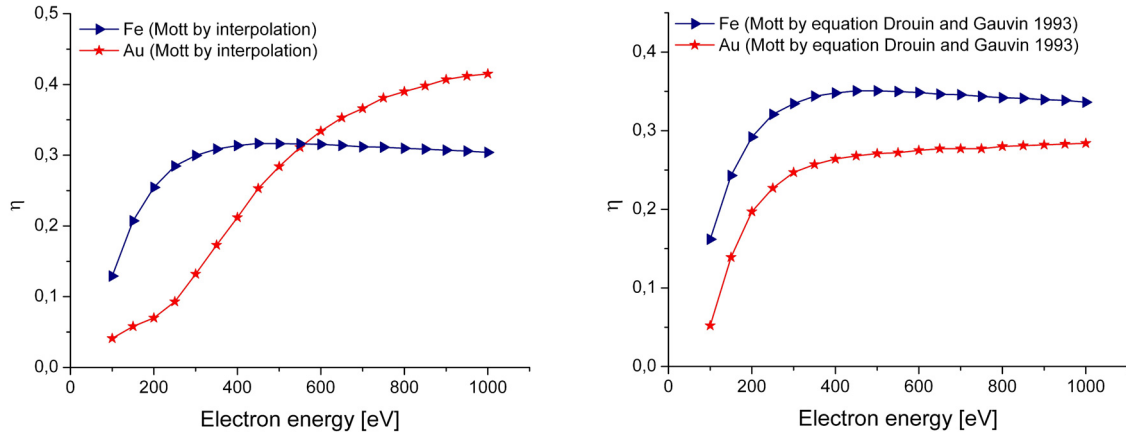


Figure 9.30 Backscatter coefficient of iron and gold (Mott by interpolation (left); Mott by equation (Drouin and Gauvin, 1993) (right))

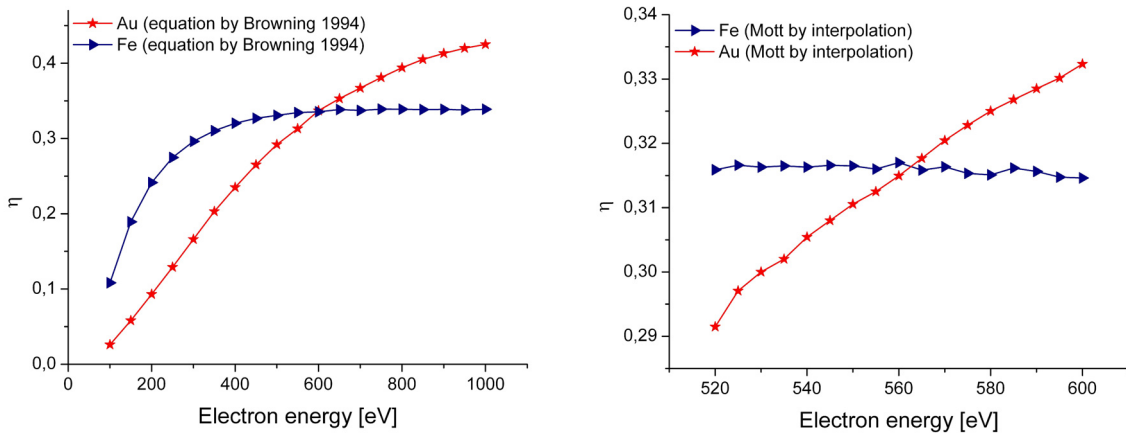


Figure 9.31 Backscatter coefficient of iron and gold (Mott by equation (Browning et al., 1994) (left); Mott by interpolation (right))

The summary of the experimental and theoretical contrast cross-over energies can be seen in table 7. The carbon/gold, iron/gold and the aluminium/gold systems are in good comparison with the Mott by interpolation results. The experimental result for silver/gold is significant higher than the theoretically predicted cross-over energy.

Table 7 Comparison of the experimental and theoretical contrast cross-over energies for carbon-gold, aluminium-gold, silver-gold and iron-gold

	Experiment [eV]	Mott by interpolation [eV]	Browning's equation [eV]	Drouin and Gauvin's equation [eV]
C-Au	about 290	285	240	115
Al-Au	about 410	410	420	115
Ag-Au	about 1000	730	840	340
Fe-Au	about 550	565	600	-

For Monte Carlo simulations the differential cross-section can be calculate by using a database which is calculated by numerical integration or by using empirical formulae. The influence of these different approaches can be seen in table 7. Especially the formula by Drouin and Gauvin (1993) does not fit very well with the experimental results. The backscatter coefficients for iron and gold do not intersect above 100 eV and therefore no contrast inversion is predicted. Browning et al. (1994) used a procedure to fit the backscatter coefficients calculated by the interpolated Mott cross-sections which is the reason for the comparable results. However, the interpolated Mott cross-section is more accurate than any empirical formula (Drouin et al., 1997) and for the carbon/gold, aluminium/gold, and iron/ gold system these values agree best with experimental results. This indicates that the established basic model is correct and that the contrast progression in low voltage backscatter images can be measured with high accuracy.

Besides the influence of the cross-section are several other possible reasons for the differences between simulation and experiment.

The Monte Carlo simulation program neglects the detector specification. It calculates the electrons which leave the specimen surface without consideration of their energy or escape angle and the detector in combination with the lens system collects the low loss, high-angle backscatter electrons. Especially concerning the Mott differential cross-section deep minima and maxima in the angular dependency occur, which can be attributed to the angular momentum of the incident electrons on their trajectories around the nuclei (Reimer, 1993). Wagner et. al. (2005) confirms and clarifies this strong angular dependency of the backscattered electrons experimentally for bulk samples.

Other differences between experiment and simulation are oxide and amorphous layers, inhomogeneities, and contaminations which can complicate the comparison. In the case of aluminium and silver, oxide layers can very easily influence the experimental results. Even

very thin oxide layers in the range of several nanometres influence the emission of secondary electrons and the elastic backscattering of electrons because of the small interaction volume.

Figure 9.32 shows a Monte Carlo simulation of the interaction volume at 150 eV electron energy. By simulating a 1 nm thick oxide layer on aluminium nearly the whole volume of the backscatter electrons is restricted inside this layer. Just a small fraction of the backscattered electrons has an escape depth larger than 1 nm. Again this simulation neglects the energy filtering of the backscattered electrons in the Zeiss Ultra 55 and only the different chemical composition is considered, not the influence of charging on SEs and BSEs (Cazaux, 1999).

The influence of the sample and the detection system can be seen by comparing the presented results with previously published findings. The contrast inversion of aluminium/gold was investigated using a cathode lens system under ultra high vacuum conditions by Frank et. al (2000). They presented a cross-over energy of about 300 eV in comparison to 410 eV. Possible reasons for the differences are the thickness of the oxide layer on aluminium and the different detection system.

Besides the special detection system, the sample preparation is of vital importance, because of the surface sensitivity of this method. Classical mechanical polishing was not used because of the possibility of luting. Instead, the silver/gold multilayer system was prepared in the FEI Nova 200 FIB (FIB parameter: 0.5 - 7 nA, 30 keV ion energy), which can also influence the contrast formation by implementation of gallium atoms. Redeposition and especially amorphous layers (Orloff et al., 2002) caused by ion irradiation can also influence the results, which could be an explanation for the differences between the simulations and the experimental results.

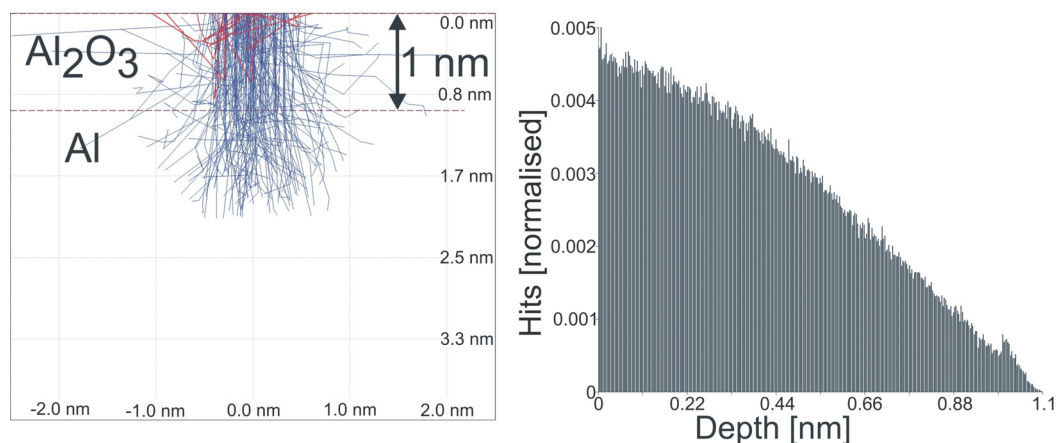


Figure 9.32 Simulated interaction volume of 150 eV electrons (backscattered electrons (red)) (left); Escape depth of backscattered electrons (calculated with Casino 2.42); sample: 1 nm Al_2O_3 layer above an aluminium substrate

9.6 Hydrocarbon Contamination

Hydrocarbon contamination inside the specimen chamber and on the sample itself results in image artefacts which complicate or avoid image acquisition. Areas previously exposed to the focused electron beam can normally be seen as black squares. This electron induced deposited layer can be thick enough that no signal from the sample can be achieved (Reimer, 1993). Because of the surface sensitivity of low voltage electron microscopy these artefacts complicates the image acquisition in a fundamental way. The Monte Carlo simulation of a 1 nm thick carbon layer on a gold substrate clarifies this problem (see figure 9.33). Nearly the whole interaction volume of the 150 eV electrons is inside of the contamination layer and no signal from the sample itself can be achieved.

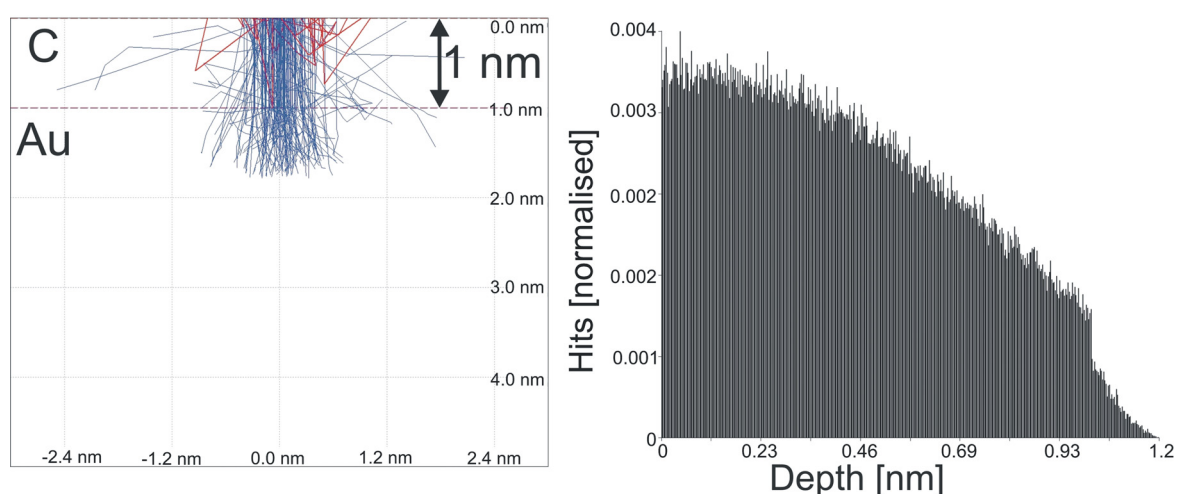


Figure 9.33 Simulated interaction volume of 150 eV electrons in a 1 nm carbon layer ($\delta = 1 \text{ g/cm}^3$) above a gold substrate (backscattered electrons (red)) (left); Escape depth of backscattered electrons (right) (calculated with Casino 2.42)

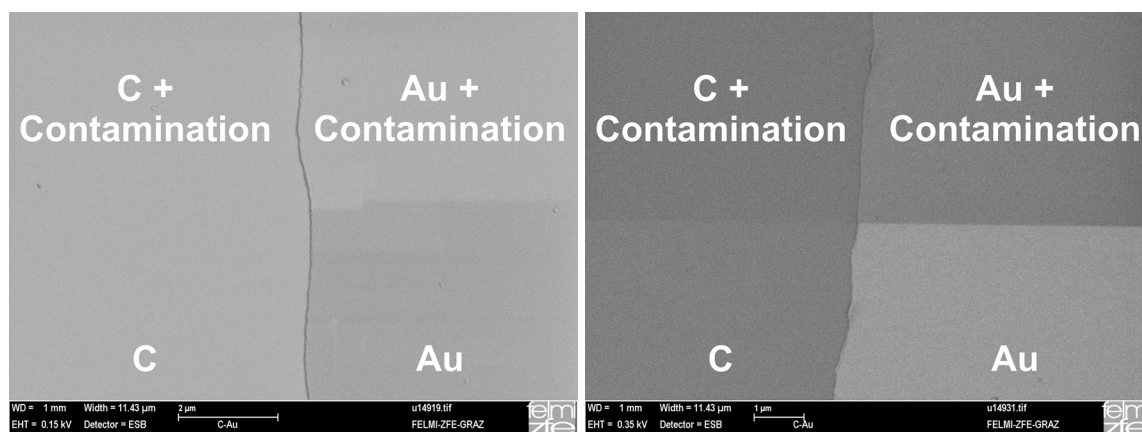


Figure 9.34 150 eV (left) and 350 eV (right) backscatter electron image (upper half was previously irradiated by the primary electron beam)

In figure 9.34 the corresponding low voltage backscatter images can be seen. The upper half was previously irradiated by the electron beam and the lower half was a fresh and clean area. The upper half of the 150 eV low voltage backscatter image at the left side shows that the contamination layer is too thick to investigate the sample. No material contrast can be seen because the escape depth of the backscattered electrons is smaller than the contamination thickness. In the 350 eV backscatter image on the right side the difference between the gold and carbon layer are visible, although the contrast is strongly degraded in comparison with the clean area.

To access the deposited morphologies and volumes of the contamination layers atomic force microscopy (AFM) measurements were performed. They were performed with a Dimension 3100 microscope equipped with a hybrid closed loop scan head and a Nanoscope IVa controller (Digital Instruments, VEECO). All measurements were done in TappingMode™ with different Olympus cantilever (2 – 40 N/m) depending on the sample requirements. As substrate for the contamination a clean silicon wafer was used. The choice of the substrate influences the results in a fundamental way because the majority of the contamination originates from the sample itself (Reimer, 1993).

The SEM was used under standard conditions which are usually used for high resolution images (SEM parameters: 5 keV; 0.26 nA; cycle time 80 s). Three different field of views were previously contaminated and the shape, the volume and the height of the contamination layers compared (see figures 8.35 to 8.40).

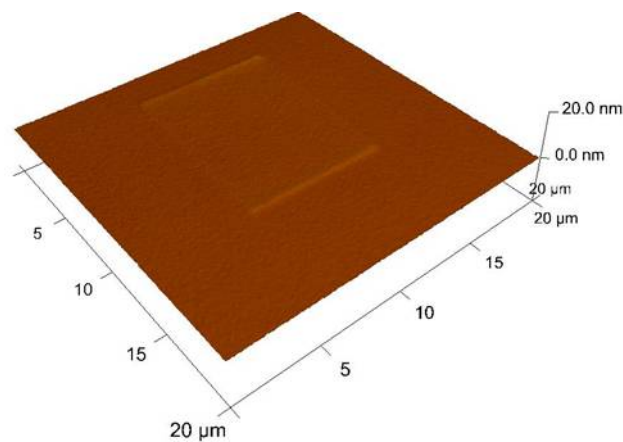


Figure 9.35 AFM height profile of hydrocarbon contamination on a silicon wafer
 AFM parameter: tapping mode, field of view: 20 μm; SEM parameters: 5 keV; 0.26 nA; cycle time: 80 s; field of view: 11.43 μm; pitch: 5.58 nm

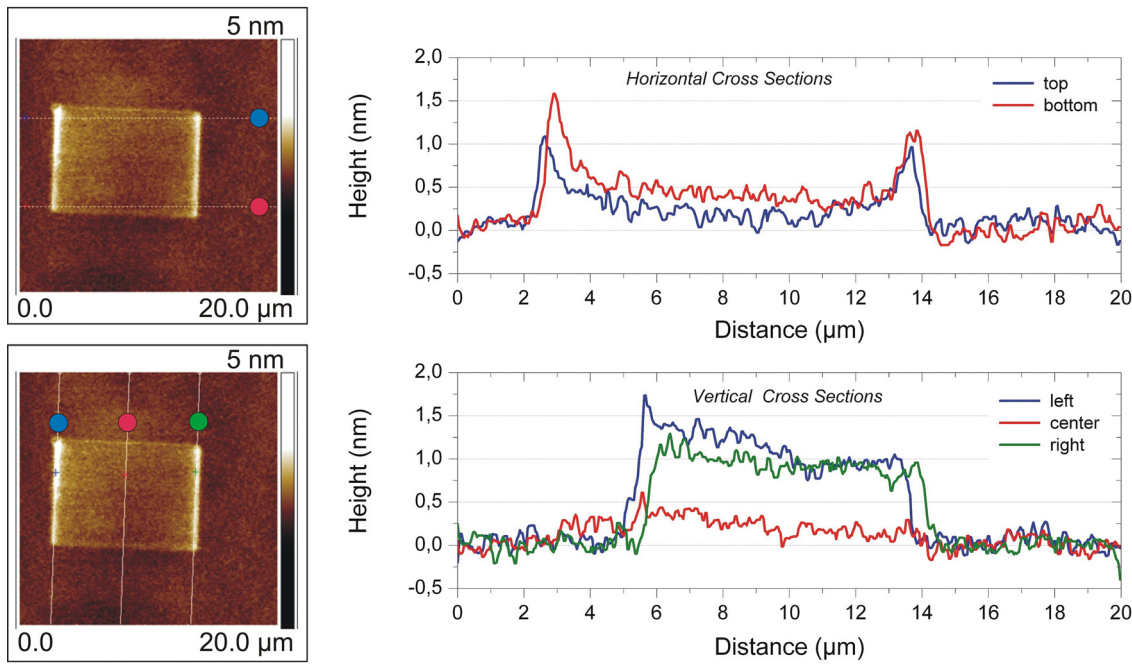


Figure 9.36 AFM line scans of hydrocarbon contamination on a silicon wafer;
AFM parameter: tapping mode, field of view: 20 μm ; SEM parameters: 5 keV; 0.26 nA; cycle
time: 80 s; field of view: 11.43 μm ; pitch: 5.58 nm

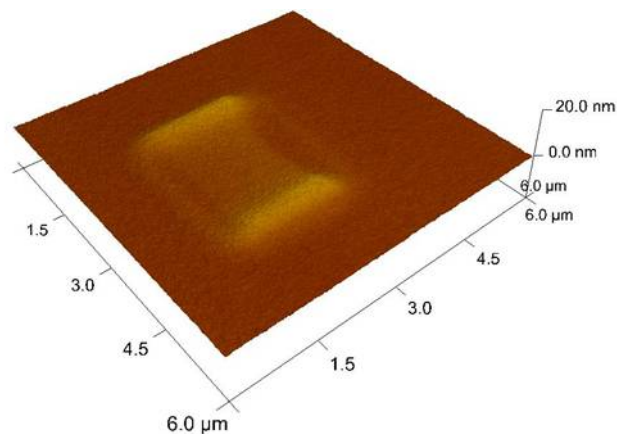


Figure 9.37 AFM height profile of hydrocarbon contamination on a silicon wafer
AFM parameter: tapping mode, field of view: 6 μm ; SEM parameters: 5 keV; 0.26 nA; cycle
time: 80 s; field of view: 2.29 μm ; pitch: 1.12 nm

The height profile measurements show the influence of the dwell time on the hydrocarbon contamination (see figure 9.36). At the boundary points at the left and right side of the image the contamination thickness is nearly three times higher than in the middle. The comparison with Monte Carlo simulations (see figure 9.34) show, that even the relatively small contamination layer in the middle of the sample (about 0.5 nm) can significantly decrease the contrast in low voltage images.

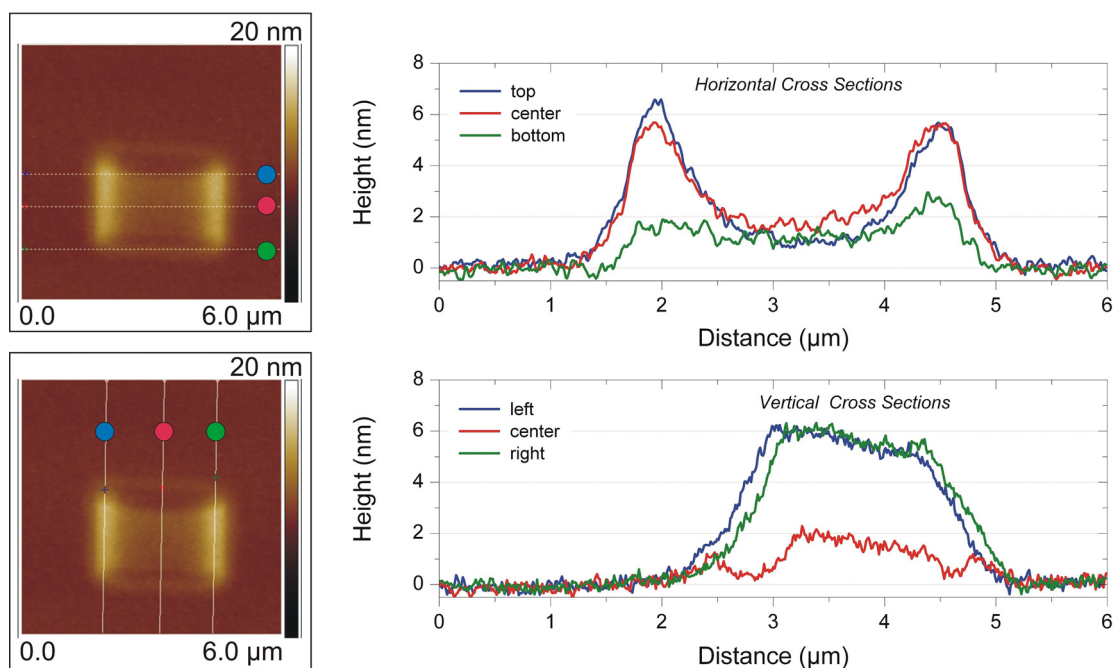


Figure 9.38 AFM line scans of hydrocarbon contamination on a silicon wafer;
AFM parameter: tapping mode, field of view: 6 μm ; SEM parameters: 5 keV; 0.26 nA; cycle
time: 80 s; field of view: 2.29 μm ; pitch: 1.12 nm

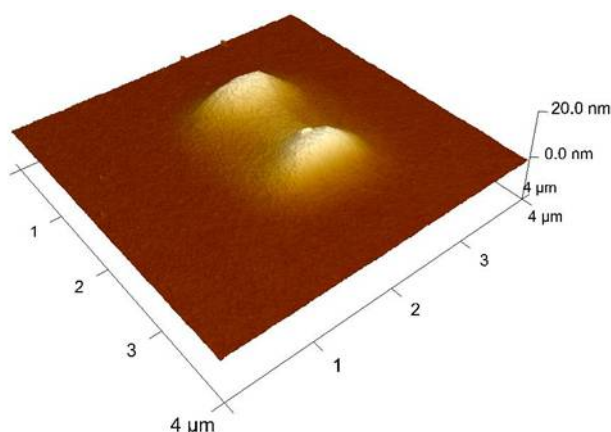


Figure 9.39 AFM height profile of hydrocarbon contamination on a silicon wafer
AFM parameter: tapping mode, field of view: 4 μm ; SEM parameters: 5 keV; 0.26 nA; cycle
time: 80 s; field of view: 1.14 μm ; pitch: 0.56 nm

By decreasing the field of view, the hydrocarbon contamination increases (see figure 9.38). The contamination thickness in the middle of the image is very large and low voltage imaging is impossible in these previously irradiated areas. The whole interaction volume of SEs and BSEs is inside this volume and no information from the sample itself can be achieved.

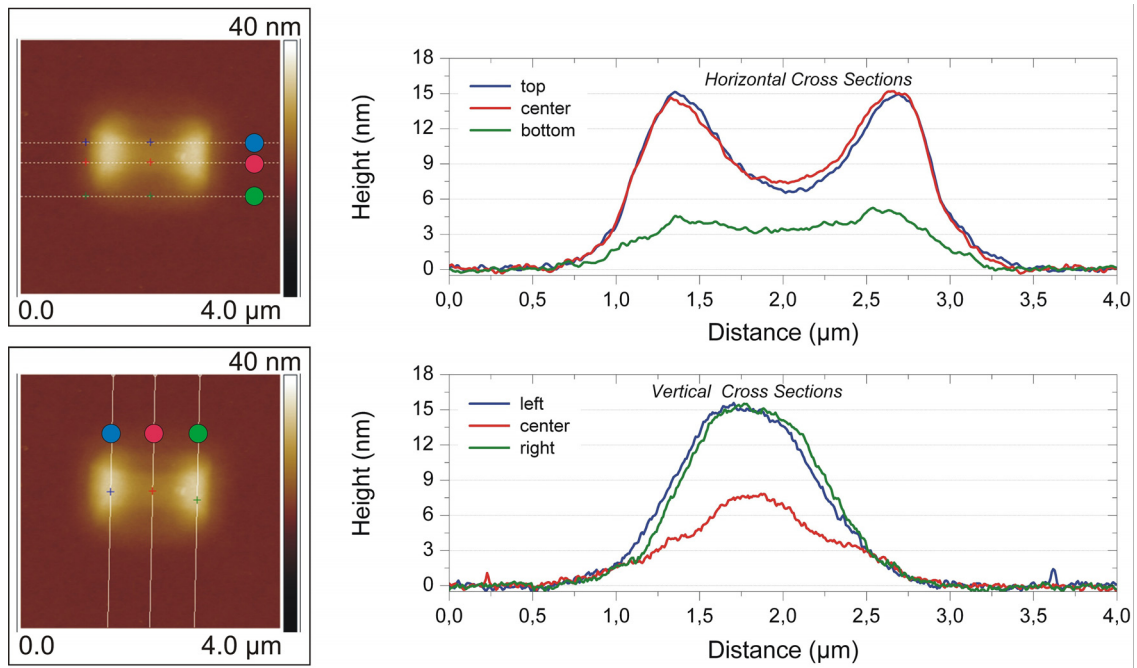


Figure 9.40 AFM line scans of hydrocarbon contamination on a silicon wafer;
 AFM parameter: tapping mode, field of view: 4 μm ; SEM parameters: 5 keV; 0.26 nA; cycle
 time: 80 s; field of view: 1.14 μm ; pitch: 0.56 nm

By increasing the magnification the dose rate of the electron probe is further increased. The effect of the scan generator and the associated higher dwell time at the left and right side influences the whole contaminated area.

Table 8 Comparison of the contamination volumes

Image dimension	horizontal [nm]	11432	2286	1143
	vertical [nm]	8541	1708	854
Pitch [nm]		5.6	1.1	0.6
Volume	total [nm^3]	31	22	25
	inside image [nm^3]	29	10	8
	outside image [nm^3]	2	12	17

The contaminated volumes for all three different magnifications can be seen in table 8. The image area is one hundred times smaller for figure 9.39 in comparison with figure 9.35 and at the same time the total contaminated volumes only slightly decrease from 31 nm^2 to 25 nm^2 which clarifies the influence of the scan pitch.

These measurements show that previously irradiated areas at this silicon wafer can not be investigated by low electron energies due to hydrocarbon contamination. For image acquisition new and clean areas must be used to investigate the sample and not the contamination.

9.7 Decontamination

Hydrocarbon contamination is a common challenge in electron microscopy. Concerning the trend to low voltage applications, this problem gains centre stage. There are different approaches to decrease the contamination.

Mainly responsible for hydrocarbon contamination is the sample itself and therefore a proper handling before and during a sample exchange is of vital importance (Reimer, 1993).

To optimise the cleanliness of the specimen chamber and the sample several methods are available. The pumping system of SEMs remove contamination, but because of the low vapour pressure of hydrocarbons this method is not very effective.

Heating the chamber increases the vapour pressure which improves the pumping of hydrocarbons. However, for practical reasons this method is still too slow.

Another common used method is by using a plasma cleaner. Highly reactive oxygen radicals are used to oxidise the hydrocarbons. These products such as CO, CO₂ or H₂O are volatile and can be removed by the vacuum system (Roediger et al., 2009).

Recently another method for cleaning the vacuum chamber and the specimen becomes more and more important. An O₂ gas jet is pointed towards the specimen and the electron beam partially ionises the gas molecules and generates ozone. This highly reactive ozone oxidises the hydrocarbons to volatile species.

9.7.1 Proof of Concept

The method of decontaminating the specimen by using an O₂ gas jet was tested for low voltage electron microscopy.

The designed decontamination device can be seen in figure 9.41. The gas bottle is attached at the specimen door and the gas nozzle is mounted at the stage to optimise the amount of O₂ molecules nearby the electron probe.

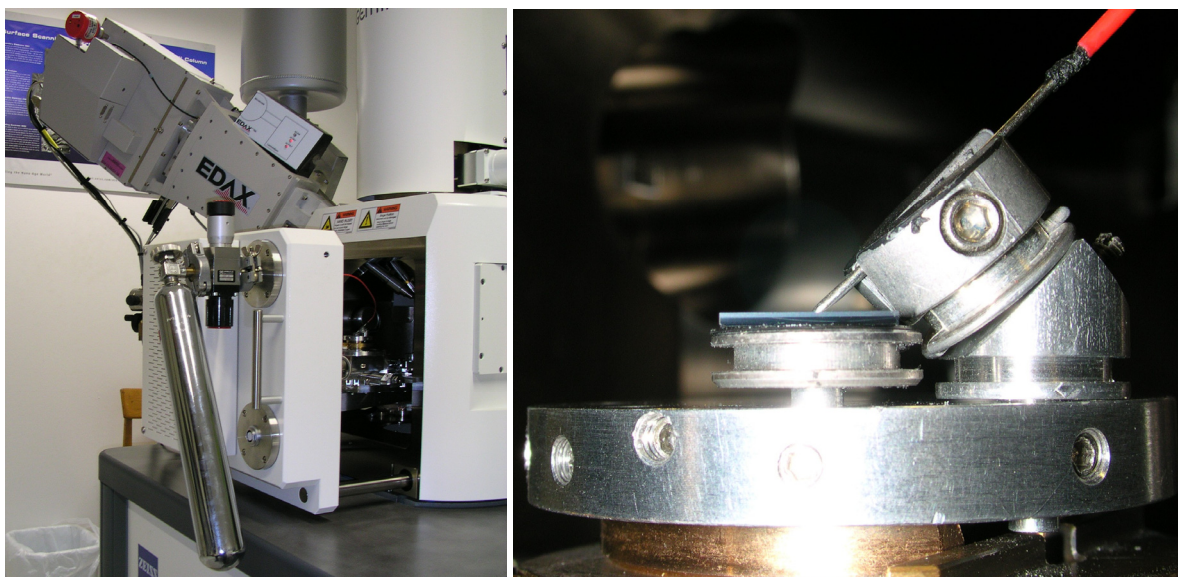


Figure 9.41 Decontamination device mounted on the Zeiss Ultra 55 (left), gas nozzle mounted on the specimen holder (right)

To control the performance of this device WDX and EDX measurements on a silicon wafer and on an aluminium sample were realised.

In the WDX spectrum almost no carbon signal can be detected during the use of the decontamination device in contrast to the EDX measurements. In this case the carbon signal is strongly reduced but not eliminated (see figure 9.42).

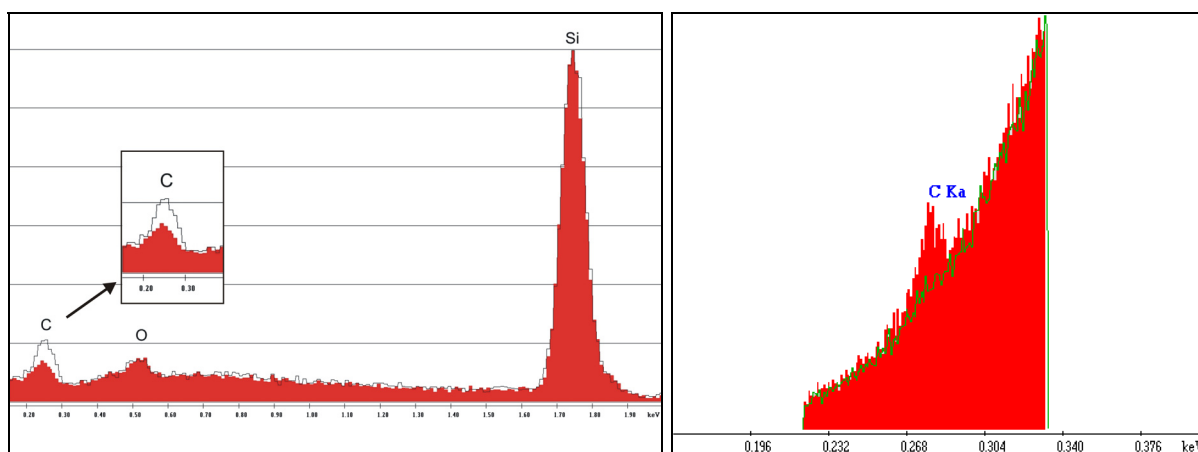


Figure 9.42 EDX spectrum with and without O₂ gas jet (left, silicon wafer, 3 keV), WDX spectrum with and without O₂ gas jet (right, aluminium sample, 20 keV)

In figure 9.43 an area previously cleaned with the O₂ jet was investigated in comparison with an uncleaned and unused area. During these measurements the decontamination device was switched off and it can clearly be seen that both spectra are nearly identical. At the right side

of figure 9.43 these two areas were compared during usage of the decontamination device. In this case the normal area shows a little bit less carbon contamination.

To sum up, the decontamination device reduces the carbon amount, but does not completely remove the whole contamination. For successful decreasing the hydrocarbon contamination the device must be switched on during measurements, otherwise the electron probe deposits a carbonic layer which will be measured an addition to the sample signal.

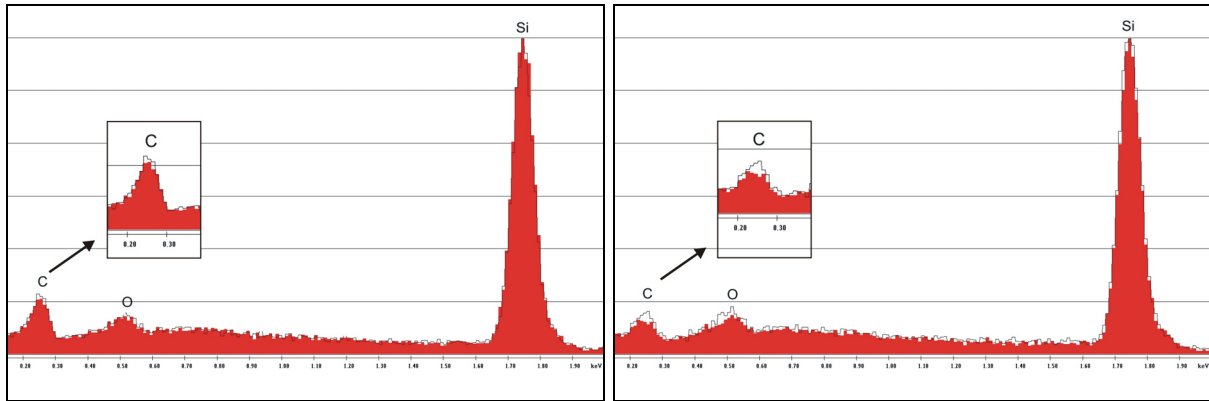


Figure 9.43 EDX spectra of normal and previously cleaned area without O₂ jet (left) EDX spectra of normal and previously cleaned with O₂ jet (right) (silicon wafer, 3keV)

The influence of the decontamination device on low electron energies microscopy can be seen in figure 9.44. The 500 eV electron micrographs show a significant decrease in hydrocarbon contamination.

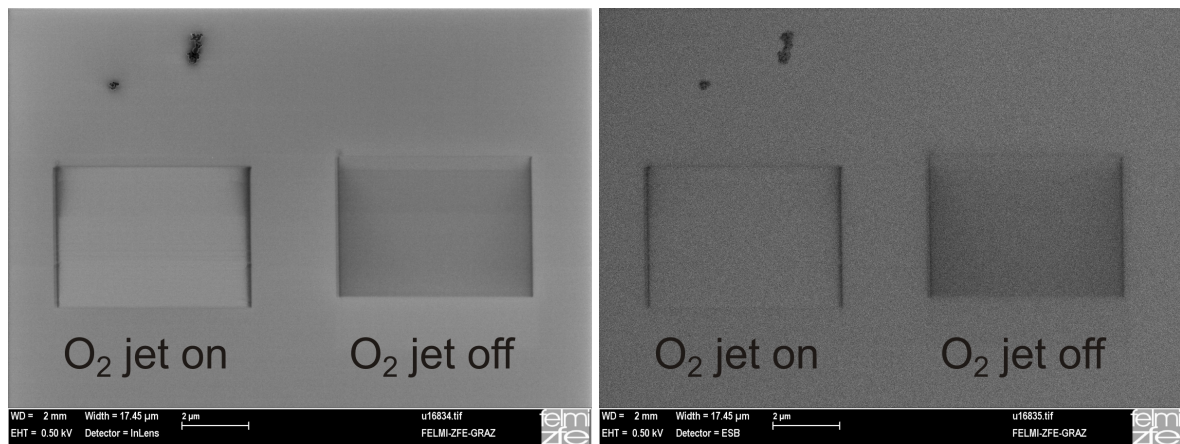


Figure 9.44 SE (left) and BSE (right) image of previously irradiated areas

The presented results document that the designed decontamination device decreases the hydrocarbon contamination. However, it is well known in literature that the carbonic layer can not be removed completely using an O₂ gas jet (Roediger et al., 2009). Further investigations and innovative ideas are necessary for a complete elimination of electron induced hydrocarbon contamination.

10 Conclusion

In Environmental Scanning Electron Microscopy (ESEM) the scattering of primary beam electrons inside the gaseous environment of the specimen chamber influences the signal to noise ratio and the interpretation of X-ray spectrometry results in a fundamental way.

The total scattering cross-section, which describes the probability that a scattering event between an electron and a gas molecule occurs, is gas type and electron energy depending. In literature there is a lack of information concerning this physical parameter for electron microscopy relevant energy ranges. This results in systematic errors in X-ray spectrometry correction procedures for low vacuum and environmental scanning electron microscopy.

In this thesis, a method is presented to determine the total scattering cross-section experimentally. Results for nitrogen, argon, water vapour and ambient air are presented for electron energies between 5 and 30 keV. For numerical integration several Matlab programs were developed to calculate the cross-sections theoretically. These results are compared with the experimental findings which draw suitable conclusions for optimising the experiment. The influence of simplifications and assumptions on the calculated results is discussed in detail.

The effective distance the electron beam travels inside the gaseous environment is called effective beam gas path length. Minimising the beam gas path length can increase the signal to noise ratio and enables very high pressure regimes in environmental scanning electron microscopy. However, modern microscopes are still far away from physical limits which mean optimum performance. The tremendous benefit of modern field emission guns is wasted caused by insufficient beam transfer. In this thesis, two different methods are presented to measure the effective beam gas path length. The methods can be adapted for every available microscope which allows to compare and to evaluate the different microscope designs and their effect on the primary beam transfer through the imaging gas. Results are presented for nitrogen, argon, water vapour and ambient air.

New technologies in scanning electron microscopy enable the opportunity to investigate the samples at very low electron energies (< 1 keV). The interaction volume and the signal diffusion is strongly decreased which improves the lateral resolution. Radiation damages and charging artefacts are strongly decreased and electrically insulating samples can be investigated without additional sputtering or evaporating the sample with a conductive layer.

The interpretation of low voltage electron micrographs is complicated by new contrast mechanisms and insufficiently known detection principles.

Therefore, model-like samples are investigated using low voltage backscatter images. The contrast inversions of carbon/gold, aluminium/gold, silver/gold and iron/gold layer systems are shown and the contrast progression compared with Monte Carlo simulations. In all these examples non-linear material contrast which prevents the intuitive image interpretation could be observed.

Different elastic scattering theories are compared and their effect on backscatter coefficients as a function of electron energies is discussed. It is well known that the Rutherford scattering theory does not describe the electron solid interaction at low electron energies in a correct way and therefore simulations using the Mott scattering theory are essential. Empirical formulae for the elastic differential cross-section are compared with interpolated values from a database. The interpolated values are more accurate than any empirical formula to describe the contrast mechanisms.

The experimentally determined cross-over energies are in good comparison with the Monte Carlo simulations using the interpolated cross-sections. This indicates that the established basic model is correct and that contrast progressions in low voltage electron backscatter micrographs can be determined with high accuracy.

Hydrocarbon contamination in electron microscopy is a common problem. In low voltage electron microscopy this electron induced deposition can complicate or even prevent the image acquisition. Atomic force microscopy was used to measure the layer thickness and these results are compared with Monte Carlo simulations.

A home-made decontamination device was tested to decrease the layer thickness. An O₂ gas jet is directed towards the electron probe inside the specimen chamber, which leads to ozone generation. This highly reactive ozone oxidises the hydrocarbons to volatile species which successfully reduces the contamination. It can be shown that the decontamination device successfully reduces the layer thickness, but low voltage electron microscopy is an extremely surface sensitive method and even very thin carbonic layers can complicate the image acquisition.

11 Appendix

11.1 List of Formula Symbols

U	acceleration voltage	$[V]$
$I_G(PE, BSE, SE)$	amount attributed to PE, BSE and SE	$[A]$
I_{amp}	amplified electron current	$[A]$
ζ	angle between surface normal and backscatter emission	
$\frac{d\eta}{d\Omega}$	angular distribution of backscattered electrons	
α_L	aperture limiting semi angle	$[rad]$
P_l^1	associated Legendre polynomial	
Z	atomic number	
A	atomic weight	
m	average number of interactions between primary beam electrons and imaging gas molecules or atoms	
χ	azimuth angle	$[rad]$
η	backscatter coefficient	
η_1	backscatter coefficient of material 1	
η_2	backscatter coefficient of material 2	
α	beam aperture	$[rad]$
I	beam current	$[A]$
d_A	beam diameter in the plane of least confusion (axial astigmatism)	$[m]$
d_C	beam diameter in the plane of least confusion (chromatic aberration)	$[m]$
d_D	beam diameter in the plane of least confusion (diffraction error)	$[m]$
d_S	beam diameter in the plane of least confusion (spherical aberration)	$[m]$
$BGPL$	beam gas path length	$[m]$
$BGPL_{fore}$	beam gas path length before the PLA	$[m]$
$BGPL_{aft}$	beam gas path length after the PLA caused by the backpressure	$[m]$
a_H	Bohr radius; $a_H = 52.9 pm$	
k_B	Boltzmann constant; $k = 1,38 \cdot 10^{-23} J / K$	
β	brightness	$[A/(cm^2 \cdot sr)]$
C_C	chromatic aberration coefficient	$[m]$
C	contrast (between 0 and 1)	

ξ_c	critical particle thickness	
ΔS	cross-sectional area of the beam	$[cm^2]$
$j = \frac{\Delta I}{\Delta S}$	current density	$[A / cm^2]$
ds	detector specimen gap	$[m]$
D	diameter of the PLA	$[m]$
Δf_A	different focus length sagittal to meridional	$[m]$
$\frac{d\sigma_{Ru}}{d\Omega}$	differential cross-section at an unscreened nucleus	$[m^2 / sr]$
$\frac{d\sigma_{Rs}}{d\Omega}$	differential cross-section at a screened nucleus	$[m^2 / sr]$
$\frac{d\sigma}{d\Omega}$	differential cross-section	$[m^2 / sr]$
$\frac{d\sigma_e}{d\Omega}$	elastic differential cross-section	$[m^2 / sr]$
$k_{strength}$	magnetic lens strength parameter	
$2 \cdot b$	distance between neighbouring atoms	$[m]$
L	distance between PLA and specimen	$[m]$
r_{nj}	distance between scattering centres n and j	$[m]$
r	distance electron nucleus	$[m]$
R	effective atom radius (screening radius)	$[m]$
V_{eff}	effective screening potential	$[eV]$
Φ_{eff}	effective work function	$[J]$
$f_e(0)$	elastic scattering amplitude at 0 rad	$[m]$
e_0	electric constant; $e_0 = 8,85 \cdot 10^{-12} (A^2 \cdot s^4) / (kg \cdot m^3)$	
$\vec{E}_{efs}$	electric field strength	$[V / m]$
E	electron energy	$[eV]$
ΔE	electron energy spread	$[eV]$
m_e	electron mass	$[kg]$
E_0	electron rest energy; $E_0 = 511 \text{ keV}$	
m_0	electron rest mass; $m_0 = 9.11 \cdot 10^{-31} \text{ kg}$	
$\vec{v}$	electron velocity	$[m / s]$
v	electron velocity	$[m / s]$
λ	electron wavelength	$[m]$

e	elementary charge; $e = 1.6 \cdot 10^{-19} \text{ C}$	
ζ	emission angle relative to the surface normal	
J_c	emission current density	$[A / cm^2]$
T_C	emission temperature	$[K]$
J	first ionisation potential	$[eV]$
f	focal length	$[m]$
f_P	fraction of the beam which is not scattered	
f_{P1}	fraction of unscattered electrons at pressure P1	
f_{P2}	fraction of unscattered electrons at pressure P2	
A	gas depending parameter	$[m^2]$
B	gas depending parameter	
$2 \cdot a$	half width of the bell shaped magnetic field	$[m]$
$\vec{p}$	impulse vector of the electron	$[(kg \cdot m) / s]$
$\frac{d\sigma_i}{d\Omega}$	inelastic differential cross-section	$[m^2 / sr]$
σ_j	individual scattering cross-section j	$[m^2]$
I_P	intensity contributed to the unscattered beam	
I_M	intensity contributed to the scattered beam	
I_M	intensity contributed to the scattered beam	
I_1	intensity at pressure P1	
I_2	intensity at pressure P2	
$I_G(SE)$	ion current attributed to SE amplification	$[A]$
Δx	layer thickness of low loss electrons	$[m]$
$\vec{F}$	Lorentz force	$[N]$
$\vec{B}$	magnetic field strength	$[T]$
k	magnetic lens strength parameter	
A_c	material constant	$[A / (cm^2 \cdot K^2)]$
B_z	magnetic field component in z direction	$[T]$
$S_{\max}$	maximum intensity	
B_z	maximum of the symmetric bell shaped magnetic field	$[T]$
r_m	maximum range of interaction of the incident electron	$[m]$
ε	mean energy loss to produce one SE	$[eV]$
dE_m	mean energy loss	$[eV]$
E_{i_mean}	mean energy per excitation (for silicon 3.6 eV)	$[eV]$

J_{mean}	mean ionisation (excitation) energy	$[eV]$
Λ	mean free path	$[m]$
n_{mean}	mean number of electron hole pairs	
$BGPL_{min}$	minimum normalised beam gas path length	$[m]$
ξ_{min}	minimum normalised particle thickness	
d_0	minimum distance between scattering centres	$[m]$
S_{min}	minimum intensity	
$\frac{d\sigma_M}{d\Omega}$	Mott differential cross-section	$[m^2 / sr]$
n_B	number of primary beam electrons	
n_{SE}	number of secondary electrons	
P_l	ordinary Legendre polynomial	
n	particle density	$[particles / m^3]$
ξ_{fore}	particle thickness before the PLA	
ξ_{aft}	particle thickness after the PLA caused by the backpressure	
η	phase shift	
p	pressure	$[Pa]$
$\Delta(p)$	pressure dependent difference between WD and BGPL	$[m]$
I_0	primary beam current	$[A]$
$r(\theta)$	ratio Mott to Rutherford differential cross-section	
I	remaining unscattered beam current	$[A]$
$f(\theta)$	scattering amplitude	$[m]$
θ	scattering angle	$[rad]$
θ_0	screening angle	$[rad]$
i_{SE}	secondary electron current	$[A]$
t_{SE}	secondary electron escape depth	$[m]$
δ	secondary electron yield	
$\delta(0, Z)$	secondary electron yield (normal incident and atomic number Z)	
k_σ	slope of the best fit straight line	$[m^{-1}]$
$\Delta\Omega = \pi \cdot \alpha^2$	solid angle	$[rad]$
C_s	spherical aberration coefficient	$[m]$
$g_1(\theta, \chi)$	spin flip amplitude	$[m]$
ϕ	surface tilt angle	
T	temperature	$[K]$

σ	total electron yield	
σ_T	total scattering cross-section	$[m^2]$
σ_{eT}	total elastic scattering cross-section	$[m^2]$
$\alpha_{Townsend}$	Townsend's first ionisation coefficient	$[m^{-1}]$
$\vec{r}$	vector electron nucleus	$[m]$
$V(r)$	Wehntzel potential	$[eV]$
Φ_W	work function	$[J]$
WD	working distance	$[m]$
d_σ	y-intercept of the best fit straight line	

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11.4 List of Abbreviations

AES	Auger Electron Spectrometry
AFM	Atomic Force Microscopy
BSE	Backscatter Electrons
CL	Cathodo-Luminescence
EDXS	Energy Dispersive X-ray Spectrometry
ESEM	Environmental Scanning Electron Microscope
ETD	Everhart Thornley Detector
FEG	Field Emission Gun
GSED	Gaseous Secondary Electron Detector
LEED	Low Electron Energy Diffraction
LFD	Large Field Detector
LVSEM	Low Vacuum Scanning Electron Microscope
PLA	Pressure Limiting Aperture
RHEED	Reflection High Energy Electron Diffraction
SE	Secondary Electrons
SEM	Scanning Electron Microscope
TEM	Transmission Electron Microscope
WD	Working Distance
WDXS	Wavelength Dispersive X-ray Spectrometry
YAG	Yttrium-Aluminium-Garnet

11.5 Source Codes

11.5.1 MOTT

The following program can be used to visualise the Mott database by D.C. Joy (Joy et al., 2001) based on the publication by Z. Czyzewski (Czyzewski et al., 1990). This Matlab source code can be used to calculate possible contrast inversions based on Mott differential cross-sections. This data set is available for the chemical elements between Hydrogen ($Z=1$) and Plutonium ($Z=94$). There are 26 electron energies available between 20 eV and 30 keV, differential cross-sections for 96 angles between 0.1 degree and 180 degree and the total elastic cross-section.

This Matlab program compares the differential cross-sections of two elements at 180 degree scattering as a function of electron energy (plot 1), it displays both differential cross-sections separately and together for chosen electron energy (plot 2-4) and both differential cross-section in a 3D plot (logarithms of the differential cross-section as a function of electron energy and scattering angle, plot 5, plot 6).

```
%function Mott
% Programm to compare the Mott differential cross-sections of two elements
% source of database: http://web.utk.edu/~srcutk/Mott/mott.htm
% [Czyzewski 1990]

% 26 energies : 20, 50, 75, 100, 200, 300, 400, 500, 600, 700, 800,900 eV. 1, 2, 3, 4, 5, 6, 7, 8, 9, 10, 15, 20, 25
and 30
% and 96 angles : 0.1 degree, 1, 2, 3, 4, 5, 6, 7, 8, 9, and 10 degrees, then by 2 degree steps to 180 degrees
% the 97th value in the table is the total Mott cross-section for
% that energy value

clear all;
close all;

File1 = input('Element 1 (i.e. 1 for hydrogen): ','s');
File2 = input('Element 2 (i.e. 1 for hydrogen): ','s');

% opening the Mott data-file
fid = fopen(File1);
[bss1] = fscanf(fid,'%f',inf);

fid = fopen(File2);
[bss2] = fscanf(fid,'%f',inf);

%defining the matrix for element 1 and 2
k=0;
for j=1:1:26
    for i=1:1:98
        k=k+1;
        daten1(i,j)=bss1(k);
    end
end
```

```

end

k=0;
for j=1:1:26
    for i=1:1:98
        k=k+1;
        daten2(i,j)=bss2(k);
    end
end

grad=[0.1,1,2,3,4,5,6,7,8,9,10];
grad(12:96)=12:2:180;

%defining the energy scale
Bereich = input('Which energy scale should be displayed? (1=200V-30kV; 2=100V-1kV; 3=20V-1kV) : ');
if Bereich==1

energy=[200,300,400,500,600,700,800,900,1000,2000,3000,4000,5000,6000,7000,8000,9000,10000,15000,20000,25000,30000]; n=26;
end
if Bereich==2
    energy=[100,200,300,400,500,600,700,800,900,1000]; n=13;
end

if Bereich==3
    energy=[20,50,75,100,200,300,400,500,600,700,800,900,1000]; n=13;
end

Winkel_Energie = input('For which energy, should the angle dependancy be displayed? (in electron volts): ');

k=0;

% determining the differential cross-section for 180° scattering for the
% chosen energy range
if Bereich==2
    for i=4:1:n
        k=k+1;
        auswert1(k)=daten1(96,i);
    end

    k=0;
    for i=4:1:n
        k=k+1;
        auswert2(k)=daten2(96,i);
    end
end

if Bereich==1
    for i=5:1:n
        k=k+1;
        auswert1(k)=daten1(96,i);
    end

    k=0;
    for i=5:1:n
        k=k+1;
        auswert2(k)=daten2(96,i);
    end
end

```



```

end

if Bereich==3
    for i=1:1:n
        k=k+1;
        auswert1(k)=daten1(96,i);
    end

    k=0;
    for i=1:1:n
        k=k+1;
        auswert2(k)=daten2(96,i);
    end
end

% calculating maximum contrast at chosen energy range
z=max(abs(auswert1-auswert2));
a=z==(abs(auswert1-auswert2));
for i=1:1:length(a)
    if a(i)>=1
        imax=i;
    end
end

% Plot 1
% plotting the differential cross-section (at 180 degree) as a function of electron energy
% for element 1 and 2
b=num2str(energy(imax));
hold on;
plot(energy,auswert1,'-r');
plot(energy,auswert2,'b');
text(energy(imax),z,b);
legend(File1,File2);
xlabel('Energy [eV]');
ylabel('Cross-section/Atom [A²]');
hold off;

a=Winkel_Energie==(energy);
for i=1:1:length(a)
    if a(i)>=1
        ien=i+4;
    end
end

for i=1:1:96
    winkel1(i)=daten1(i,ien);
end

%total cross-section of element 1
total1=daten1(97,ien);

for i=1:1:96
    winkel2(i)=daten2(i,ien);
end

%total cross-section of element 2
total2=daten2(97,ien);

```

```

% Plot 2
%plotting differential cross-section of element 1
figure;
semilogy(grad,winkel1,'--r')
legend(File1);
xlabel('Scattering angle');
ylabel('Cross-section/Atom [A²]');

% Plot 3
%plotting differential cross-section of element 2
figure
semilogy(grad,winkel2,'b')
legend(File2);
xlabel('Scattering angle');
ylabel('Cross-section/Atom [A²]');

% Plot 4
%plotting differential cross-section of element 1 and 2
figure
semilogy(grad,winkel1,'--r',grad,winkel2,'b');
legend(File1);
legend(File2);
xlabel('Scattering angle');
ylabel('Cross-section/Atom [A²]');

for q=1:1:length(grad)
    rad(q)=(grad(q)*2*pi)/360;
    q=q+1;
end

hj=daten1(98,ien)
total1
total2

zahlen=0;
for i=91:1:96
    zahlen=zahlen+1;
    dss(zahlen)=winkel1(i)*sin(rad(i));
end

unten=91;
oben=96;

%3D Plot daten1
x=zeros(1,1);
a=1;
for i=1:1:length(energy)
    for j=1:1:96
        x(i,j)=energy(a);
    end
    a=a+1;
end

y=zeros(1,1);
a=1;
for i=1:1:length(energy)
    y(i,1:1:11)=[0.1,1,2,3,4,5,6,7,8,9,10];
    y(i,12:1:96)=12:2:180;
end

```

```

end

% 3D Plot daten1
% determining the z values for the 3D plot element 1
z1=zeros(1,1);
for j=1:1:96
    a=1;

    if Bereich==1
        for i=5:1:26
            z1(a,j)=log(daten1(j,i));
            a=a+1;
        end
    end

    if Bereich==2
        for i=4:1:13
            z1(a,j)=log(daten1(j,i));
            a=a+1;
        end
    end

    if Bereich==3
        for i=1:1:13
            z1(a,j)=log(daten1(j,i));
            a=a+1;
        end
    end

end

% Plot 5
% 3D plot element 1
figure
surf(x,y,z1);
xlabel('Energy [eV]');
ylabel('Scattering angle');
zlabel('log (Cross-section/Atom [A²])');
view([-240,45]);

%3D Plot daten2
%determining the z values for the 3D plot element 1
z2=zeros(1,1);
for j=1:1:96
    a=1;

    if Bereich==1
        for i=5:1:26
            z2(a,j)=log(daten2(j,i));

            a=a+1;
        end
    end

    if Bereich==2
        for i=4:1:13
            z2(a,j)=log(daten2(j,i));

```

```

        a=a+1;
    end
end

if Bereich==3
    for i=1:1:13
        z2(a,j)=log(daten2(j,i));
        a=a+1;
    end
end

end

% Plot 6
% 3D plot element 2
figure
surf(x,y,z2);
xlabel('Energy [eV]');
ylabel('Scattering angle');
zlabel('log (Cross-section/Atom [A²])');
view([-240,45]);

```

11.5.2 MOTT (inversion)

The following program can be used to visualise the Mott database by D.C. Joy (Joy et al., 2001) based on the publication by Z. Czyzewski (Czyzewski et al., 1990). For detailed information see chapter 0.

This Matlab program visualise the maximum contrast between the 94 elements (plot 1), the electron energy at maximum contrast (plot 2), the maximum contrast inversion (plot 3) and the electron energy at maximum contrast inversion (plot 4).

```

%function Mott
%source of database: http://web.utk.edu/~srcutk/Mott/mott.htm
%[Czyzewski 1990]

% 26 energies : 20, 50, 75, 100, 200, 300, 400, 500, 600, 700, 800,900 eV. % 1, 2, 3, 4, 5, 6, 7, 8, 9, 10, 15, 20,
25 and 30
% and 96 angles : 0.1 degree, 1, 2, 3, 4, 5, 6, 7, 8, 9, and 10 degrees,
% then by 2 degree steps to 180 degrees
% the 97th value in the table is the total Mott cross-section for

clear all;
close all;

% setting the parameters
sammel=zeros(94,94);
sammel2=zeros(94,94);
grad=[0.1,1,2,3,4,5,6,7,8,9,10];
grad(12:96)=12:2:180;
energy=[400,500,600,700,800,900,1000,2000,3000,4000,5000,6000,7000,8000,9000,10000,15000,20000,25000,
30000]; n=26;

%comparison => iteration with each element
for w=1:1:94
    for l=94:-1:1

```

```

invert1=0;
invert2=0;
invert3=0;
invert4=0;
invert5=0;

%loading of the Mott database:
%filename = atomic number
File1 = num2str(w);
fid = fopen(File1);
[bss1] = fscanf(fid,'%f',inf);

File2 = num2str(l);
fid = fopen(File2);
[bss2] = fscanf(fid,'%f',inf);
k=0;

%defining the matrix for element 1
for j=1:1:26
    for i=1:1:98
        k=k+1;
        daten1(i,j)=bss1(k);
    end
end

%defining the matrix for element 2
k=0;
for j=1:1:26
    for i=1:1:98
        k=k+1;
        daten2(i,j)=bss2(k);
    end
end

winkel=90:2:180;
winkel=winkel*pi/180;

% calculating the scattering angle for integration
for i=1:1:length(winkel)
    winkel2=2*pi*sin(winkel(i));
end

% integration element 1
k=0;
for i=7:1:n
    k=k+1;
    auswert1(k)=trapz(winkel,(daten1(51:1:96,i)*winkel2));
end

% integration element 2
k=0;
for i=7:1:n
    k=k+1;
    auswert2(k)=trapz(winkel,(daten2(51:1:96,i)*winkel2));
end

z=0;
a=0;
a2=0;

```

```

z2=0;

hss=auswert2-auswert1;

% finding maximum contrast + energy of maximum contrast
if w<1
    lss=min(hss);

    z2=min(hss);
    a2=z2==hss;
    for i=1:1:length(a2)
        if a2(i)>=1
            imax2=i;
        end
    end
end

% finding maximum contrast + energy of maximum contrast
if w>1
    lss=(-1)*max(hss);
    z2=max(hss);
    a2=z2==hss;
    for i=1:1:length(a2)
        if a2(i)>=1
            imax2=i;
        end
    end

end

z=max(abs(auswert1-auswert2));
a=z==(abs(auswert1-auswert2));
for i=1:1:length(a)
    if a(i)>=1
        imax=i;
    end
end

sammel4(w,l)=energy(imax2);
sammel3(w,l)=lss;
sammel2(w,l)=energy(imax);
sammel(w,l)=z;
st = fclose('all');
end
end

for i=1:1:94
    sammel2(i,i)=0;
end

%3D plot
x=zeros(1,1);
y=zeros(1,1);
for i=1:1:94
    for j=1:1:94
        y(i,j)=i;
        x(i,j)=j;
    end
end
end

```

```

% Plot 1
%ploting maximum contrast
figure
surface(x,y,sammel);

% Plot 2
%ploting electron energy at maximum contrast
figure
surface(x,y,sammel2);

% Plot 3
%maximum contrast inversion
figure
surface(x,y,sammel3);

% Plot 4
%ploting electron energy at maximum contrast inversion
figure
surface(x,y,sammel4);

```

11.5.3 TSCS of Monatomic Gases

This Matlab program is based on Danilatos (1988) theory to calculate the elastic and inelastic differential cross-section and the total scattering cross-section of argon and neon. The results are compared with the analytical expression by Jost and Kessler (1963) (see formula 6.7).

```

clear all;
close all;

% ah (Bohr Radius)
ah=5.29177E-11;

% Argon is parameter 1 and Neon parameter 2
% J (ionisation energy), R (effective atomic radius)
Bereich = input('Argon=1; Neon=2; ');

if Bereich==1
    J=15.75;
    R=sqrt(((4.71E-10)*ah)/(2*18));
end

if Bereich==2
    J=21.56;
    R=sqrt(((1.66E-10)*ah)/(2*10));
end

% Definition of step sizes
q=0;
intervallklein=0.00001;
intervallgross=0.001;
intervallsehrklein=0.0000005;
intervall=100;
integrationslimit=0.0001;
obereseVlimit=30000;

```

```

% epe (electron energy), le (electron wavelength)
% iteration
for epe=5000:intervall:obereseVlimit

    q=q+1;
    le=1.226E-9*(epe*(1+0.9778E-6*epe))^(0.5);

    if Bereich==1
        A=(le^4*18*(1+epe/(511000))^2)/(4*pi^4*ah^2);
    end

    if Bereich==2
        A=(le^4*10*(1+epe/(511000))^2)/(4*pi^4*ah^2);
    end

    detao=le/(2*pi*R);
    detae=J/(4*epe);
    w=0;

%interval 1
for deta=0.000001:intervallsehrklein:0.01
    w=w+1;

    sigmai(w)=((A*(deta^2+detae^2+2*detao^2))/((deta^2+detae^2)*(deta^2+detae^2+detao^2)^2))*sin(de
ta);

    if Bereich==1
        sigmae(w)=((A*18)/(16*(sin(deta/2)*sin(deta/2)+sin(detao/2)*sin(detao/2))^2))*sin(deta);
    end
    if Bereich==2
        sigmae(w)=((A*10)/(16*(sin(deta/2)*sin(deta/2)+sin(detao/2)*sin(detao/2))^2))*sin(deta);
    end
    winkel(w)=deta;

%without sin
    sigmai2(w)=((A*(deta^2+detae^2+2*detao^2))/((deta^2+detae^2)*(deta^2+detae^2+detao^2)^2));
    if Bereich==1
        sigmae2(w)=((A*18)/(16*(sin(deta/2)*sin(deta/2)+sin(detao/2)*sin(detao/2))^2));
    end
    if Bereich==2
        sigmae2(w)=((A*10)/(16*(sin(deta/2)*sin(deta/2)+sin(detao/2)*sin(detao/2))^2));
    end
end

%interval 2
for deta=0.01:intervallklein:intervallklein:0.17
    w=w+1;

    sigmai(w)=((A*(deta^2+detae^2+2*detao^2))/((deta^2+detae^2)*(deta^2+detae^2+detao^2)^2))*sin(de
ta);
    if Bereich==1
        sigmae(w)=((A*18)/(16*(sin(deta/2)*sin(deta/2)+sin(detao/2)*sin(detao/2))^2))*sin(deta);
    end
    if Bereich==2
        sigmae(w)=((A*10)/(16*(sin(deta/2)*sin(deta/2)+sin(detao/2)*sin(detao/2))^2))*sin(deta);
    end
    winkel(w)=deta;

%without sin

```



```

        sigmai2(w)=((A*(deta^2+detae^2+2*detao^2))/((deta^2+detae^2)*(deta^2+detae^2+detao^2)^2));
    if Bereich==1
        sigmae2(w)=((A*18)/(16*(sin(deta/2)*sin(deta/2)+sin(detao/2)*sin(detao/2))^2));
    end
    if Bereich==2
        sigmae2(w)=((A*10)/(16*(sin(deta/2)*sin(deta/2)+sin(detao/2)*sin(detao/2))^2));
    end
end

%interval 3
t=w;
for deta=0.17+intervallgross:intervallgross:pi
    w=w+1;
    sigmai(w)=0;
    if Bereich==1
        sigmae(w)=((A*18)/(16*(sin(deta/2)*sin(deta/2)+sin(detao/2)*sin(detao/2))^2))*sin(deta);
    end
    if Bereich==2
        sigmae(w)=((A*10)/(16*(sin(deta/2)*sin(deta/2)+sin(detao/2)*sin(detao/2))^2))*sin(deta);
    end
    winkel(w)=deta;

    %without sin
    sigmai2(w)=0;
    if Bereich==1
        sigmae2(w)=((A*18)/(16*(sin(deta/2)*sin(deta/2)+sin(detao/2)*sin(detao/2))^2));
    end
    if Bereich==2
        sigmae2(w)=((A*10)/(16*(sin(deta/2)*sin(deta/2)+sin(detao/2)*sin(detao/2))^2));
    end
end

ts2=sigmai2+sigmae2;
ts=sigmai+sigmae;
totalsc(:,q)=(sigmai+sigmae);
inesc(:,q)=sigmai;
elasc(:,q)=sigmae;

% numerical integration to calculate the total scattering cross-section
bss(q)=trapz(winkel,ts)*2*pi;
energie(q)=epe;

% calculating the analytical expression by Jost and Kessler (1963)
if Bereich==1
    kessler(q)=(le^2*R^2*18*(1+epe/5.11E5)^2)/(pi*ah^2)*(18-1-4*log((pi*R*J)/(2*le*epe)));
end

if Bereich==2
    kessler(q)=(le^2*R^2*10*(1+epe/5.11E5)^2)/(pi*ah^2)*(10-1-4*log((pi*R*J)/(2*le*epe)));
end

end

% plot: inelastic and elastic differential cross-section
figure
loglog(winkel,sigmai2,'b:',winkel,sigmae2,'b--',winkel,ts2,'r','LineWidth',6);
set(gca,'FontSize',35)
legend('inelastic','elastic','total');
xlabel('Scattering angle [radian]');

```

```

ylabel('Differential Cross-section [m²/steradian]');

% plot: total scattering cross-section as a function of electron energy
figure
hold on
plot(energie,bss,'LineWidth',6)
plot(energie,kessler,'LineWidth',6)
set(gca,'FontSize',35)
xlabel('Electron energy [eV]');
ylabel('Total Scattering Cross-section [m²]');
hold off

```

11.5.4 TSCS of Molecular Gases

This Matlab program is based on Danilatos (1988) theory to calculate the elastic, the inelastic differential cross-section and the total scattering cross-section of molecular gases. With the following source code the total scattering cross-section of water vapour can be calculated. By varying the ionisation energies, the distances between atoms and the effective atomic radii this program can be used to calculate for example nitrogen gas or oxygen gas.

```

% JH (ionisation energy of Hydrogen), RH (effective atomic radius of Hydrogen) and so on
% rOH distance between atom O and H and so on

% epe (electron energy), le (electron wavelength)
% ah (Bohr Radius)

clear all;
close all;

% Definition of step sizes
intervallklein=0.00001;
intervallgross=0.001;
intervallsehrklein=0.000005;
q=0;

% iteration
for epe=5000:500:30000

    q=q+1;

    le=1.226E-9*(epe*(1+0.9778E-6*epe))^(0.5); %Wellenlänge der Elektronen
    rOH=95.84E-12;
    rHH=151.508E-12;

    ah=5.29177E-11;
    AH=(le^4*1*(1+epe/(5.11E5))^2)/(4*pi^4*ah^2);
    AO=(le^4*8*(1+epe/(5.11E5))^2)/(4*pi^4*ah^2);

    RH=sqrt((0.529E-10*ah)/(2*1));
    RO=sqrt((2.01E-10*ah)/(2*8));
    detaoH=le/(2*pi*RH);
    detaoO=le/(2*pi*RO);

    JH=13.59;

```

```

JO=13.61;

detaoH=le/(2*pi*RH);
detaoO=le/(2*pi*RO);
detaeH=JH/(4*epe);
detaeO=JO/(4*epe);

w=0;

% interval 1
for deta=0.000001:intervallsehrklein:0.00005
    w=w+1;

    sigmai(w)=2*((AH*(deta^2+detaeH^2+2*detaoH^2))/((deta^2+detaeH^2)*(deta^2+detaeH^2+detaoH^2)^2))*sin(deta)+((AO*(deta^2+detaeO^2+2*detaoO^2))/((deta^2+detaeO^2)*(deta^2+detaeO^2+detaoO^2)^2))*sin(deta);

    sigmai2(w)=2*((AH*(deta^2+detaeH^2+2*detaoH^2))/((deta^2+detaeH^2)*(deta^2+detaeH^2+detaoH^2)^2))*sin(deta)+((AO*(deta^2+detaeO^2+2*detaoO^2))/((deta^2+detaeO^2)*(deta^2+detaeO^2+detaoO^2)^2));

    fH=(sqrt(abs((AH*1)/(16*(sin(deta/2)*sin(deta/2)+sin(detaoH/2)*sin(detaoH/2))^2)))));
    fO=(sqrt(abs((AO*8)/(16*(sin(deta/2)*sin(deta/2)+sin(detaoO/2)*sin(detaoO/2))^2)))));

    s=(4*pi*sin(deta/2))/le;

    sigmae(w)=((2*fH*fH*(sin(s*rHH)/(s*rHH)))+(4*fH*fO*(sin(s*rOH)/(s*rOH)))+2*fH*fH+fO*fO)*sin(deta);

    sigmae2(w)=((2*fH*fH*(sin(s*rHH)/(s*rHH)))+(4*fH*fO*(sin(s*rOH)/(s*rOH)))+2*fH*fH+fO*fO);

    winkel(w)=deta;
    si(w)=sin(deta);

end

% interval 2
for deta=0.00005:intervallklein:intervallklein:0.17
    w=w+1;

    sigmai(w)=2*((AH*(deta^2+detaeH^2+2*detaoH^2))/((deta^2+detaeH^2)*(deta^2+detaeH^2+detaoH^2)^2))*sin(deta)+((AO*(deta^2+detaeO^2+2*detaoO^2))/((deta^2+detaeO^2)*(deta^2+detaeO^2+detaoO^2)^2))*sin(deta);

    sigmai2(w)=2*((AH*(deta^2+detaeH^2+2*detaoH^2))/((deta^2+detaeH^2)*(deta^2+detaeH^2+detaoH^2)^2))*sin(deta)+((AO*(deta^2+detaeO^2+2*detaoO^2))/((deta^2+detaeO^2)*(deta^2+detaeO^2+detaoO^2)^2));

    fH=(sqrt(abs((AH*1)/(16*(sin(deta/2)*sin(deta/2)+sin(detaoH/2)*sin(detaoH/2))^2)))));
    fO=(sqrt(abs((AO*8)/(16*(sin(deta/2)*sin(deta/2)+sin(detaoO/2)*sin(detaoO/2))^2)))));

    s=(4*pi*sin(deta/2))/le;

    sigmae(w)=((2*fH*fH*(sin(s*rHH)/(s*rHH)))+(4*fH*fO*(sin(s*rOH)/(s*rOH)))+2*fH*fH+fO*fO)*sin(deta);

```

```

sigmae2(w)=((2*fH*fH*(sin(s*rHH)/(s*rHH)))+(4*fH*fO*(sin(s*rOH)/(s*rOH)))+2*fH*fH+fO*fO);

winkel(w)=deta;
si(w)=sin(deta);

end

t=w;

% interval 3
for deta=0.17+intervallgross:intervallgross:pi
    w=w+1;

    sigmai(w)=0;
    sigmae2(w)=0;

    fH=(sqrt(abs((AH*1)/(16*(sin(deta/2)*sin(deta/2)+sin(detaoH/2)*sin(detaoH/2))^2))));
    fO=(sqrt(abs((AO*8)/(16*(sin(deta/2)*sin(deta/2)+sin(detaoO/2)*sin(detaoO/2))^2))));

    s=(4*pi*sin(deta/2))/le;

    sigmae(w)=((2*fH*fH*(sin(s*rHH)/(s*rHH)))+(4*fH*fO*(sin(s*rOH)/(s*rOH)))+2*fH*fH+fO*fO)*si
    n(deta);

    sigmae2(w)=((2*fH*fH*(sin(s*rHH)/(s*rHH)))+(4*fH*fO*(sin(s*rOH)/(s*rOH)))+2*fH*fH+fO*fO);

    winkel(w)=deta;
    si(w)=sin(deta);
end

inesc(:,q)=sigmai;
elasc(:,q)=sigmae;

ts=(sigmae+sigmai);
ts2=(sigmae2+sigmai2);

% numerical integration to calculate the total scattering cross-section
bss(q)=(trapz(winkel,sigmae)+trapz(winkel,sigmai))*2*pi;
energie(q)=epe;

end

% plot: elastic, inelastic and total differential cross-section of water vapour
figure
loglog(winkel,sigmai2,'b-',winkel,sigmae2,'b--',winkel,ts2,'r','LineWidth',6);
set(gca,'FontSize',35)
legend('inelastic','elastic','total');
xlabel('Scattering angle [radian]');
ylabel('Differential Cross-section [m²/steradian]');

% plot: total scattering cross-section of water vapour as a function of electron energy
figure
hold on
plot(energie,bss,'LineWidth',6);
set(gca,'FontSize',35)
xlabel('Electron Energy [eV]');
ylabel('Total Scattering Cross-section [m²]');

```

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Talks:

- Rattenberger J. 01.09.2009. Nonlinear material contrast in low voltage backscatter electron images. Microscopy Conference 2009, Graz.
- Invited: Rattenberger J. 31.08.2010. Kontrastinversion in Niederenergie Rückstreuelekttronen - Abbildungen. Gemini User Meeting, Bochum.

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