Practical course in scanning electron microscopy

Fortgeschrittenen Praktikum
an der Technischen Universität München

Sommersemester 2019

Version with figures on request to “Versuchsbetreuer(Innen)”

Contents

1. Introduction

Microscopes are used in many fields of science to probe beyond the limits of sight. In most individuals, the human eye is able to distinguish two points as separate when the distance between them is roughly 100 µm. Resolving power can be improved by magnification with a system of lenses, i.e. optical microscopes. Unfortunately, there exists a resolution limit for optical microscopes based on the wavelength of light. The smallest distance d between two points which can be resolved is given by the Rayleigh criterion:

$$d = \frac{0.61\,\lambda}{n \sin\alpha}$$

With
λ: the wavelength of the light
α: the semi-angle of the lens aperture
n: the refractive index of the viewing medium

The wavelength of visible light is about 400 nm for blue light and 700 nm for red light. The resolution which can be achieved with an optical microscope is therefore no better than 200 nm. Optical magnification is thus limited to about 1000x. To improve resolution further, smaller wavelengths are needed. Optical microscopy with ultraviolet light can be used to marginally improve resolution. However, a significant improvement of resolution requires the use of electron beams.

In this practical course, we investigate the techniques of scanning electron microscopy (SEM, De: Rasterelektronenmikroskop/REM). Due to the wave-particle duality, the wavelength λ of an electron can be calculated with the de-Broglie equation:

$$\lambda = \frac{h}{p} \quad .$$

With:
p: the electron momentum
h: the Planck constant

The wavelength depends on the electron momentum after acceleration in an electrostatic field.
The momentum is:

$$p = \sqrt{2m_e eU} \qquad \text{since} \qquad E_{kin} = eU = \frac{p^2}{2m_e}$$

With:
m_e: the elementary charge

e: the elementary charge
U: the accelerating voltage

This gives for the wavelength:

$$\lambda = \frac{h}{\sqrt{2m_e eU}} \quad .$$

Typical voltages used in scanning electron microscopes are between 1 kV and 30 kV, yielding wavelengths of about 10-40 pm. This results in a theoretical diffraction-limited resolution of less than 0.5 nm. In practice, however, the resolution is limited to larger values by other factors, such as cathode size and aberrations but also influence from outside like mechanical vibration or electro-magnetic fields. At high electron velocities (acceleration voltages greater than ~80 kV) a relativistic correction is needed. While this is unnecessary for scanning electron microscopes, it must be applied for other electron-based microscopes such as the transmission electron microscope.

The aim of this course is to acquaint students with scanning electron microscopy. This document provides an introduction to the fundamental principles of this class of microscope. In the practical session, students will learn to operate a scanning electron microscope and to interpret the images obtained with it.

2. Formation of an electron beam

To form an electron beam, an electron source (cathode) and an electrostatic potential for accelerating the electrons are required. The cathode produces electrons either by thermionic emission or field emission. The electrons are subsequently accelerated by the electric field between the cathode and an anode. Initial focusing of the electron beam is performed by a so-called Wehnelt cylinder (see Fig. 1). The Wehnelt cylinder is set to a negative electrical potential and repels electrons emitted by the cathode to an initial focal point known as the crossover, which forms a virtual electron source for the subsequent lenses. Since collisions with residual gas atoms scatter electrons, broadening the beam and reducing image quality, a high-quality vacuum must be maintained near the cathode.

In order to image a sample in fine detail, the electron beam must be further shaped and focused. As the electrons are electrically charged, this is performed by a system of electrostatic and/or electromagnetic lenses. Image quality is reduced by lens aberration. In the following, the three most relevant lens aberrations are described.

Fig.1: Schematic view of a thermionic electron gun. A high voltage is applied between the cathode and the anode. The electrical potential of the Wehnelt cylinder shapes the electron beam. The electrical field from the Wehnelt cylinder focuses the electrons into a crossover point, which is the virtual electron source for the lenses in the electron microscope illumination system. [4]

3. Lens aberrations

As in optical microscopes, the resolution of an image formed by a scanning electron microscope is limited by aberrations. Achieving the diffraction-limited sub-nanometer resolution is difficult in practice. Certain shortcomings of the lenses can be corrected, and others can be at least minimized. A summary of the three most important defects for scanning electron microscopes is given below:

3.1. Chromatic aberration

The refractive index of a lens always has a small wavelength dependence; rays with different wavelengths are therefore focused to different points (Fig. 2). This effect is called chromatic aberration. The wavelength of electrons in the incident beam varies due to thermal variations in electron energy (following a Boltzmann distribution). However, the electron beams produced by typical SEM cathodes are highly mono-energetic, and chromatic aberration is rarely a limiting factor of image quality in a scanning electron microscope.

Fig.2: Lens with chromatic aberration: Different colors, i.e. different wavelengths have different foci. [5]

3.2. Astigmatism

Astigmatism occurs if the focal length is different for perpendicular planes of the incident beam (Fig. 3). This defect is caused by manufacturing imperfections, e.g. lens elements that deviate from perfect cylindrical symmetry and off-center beam aperture stops. It can also originate from external electric fields and impurities in the beam column. Fortunately, astigmatism can be easily corrected using “stigmators”. These elements create a compensating electromagnetic field and must be adjusted to correct the image for astigmatism before taking images with an electron microscope.

Fig.3: Lens with astigmatism. [6]

3.3. Spherical aberration

Spherical aberration is a lens defect whereby off-axis rays are refracted more strongly than on-axis rays (Fig. 4). This aberration cannot easily be corrected. It can only be minimized by careful construction and by limiting off-axis rays with aperture stops.

Fig.4: A lens of nominal focal length F with spherical aberration. The focal length of a ray depends on its distance from the system’s axis. [5]

4. Interaction of electrons with matter

An electron beam incident on matter interacts in several ways. Electrons can be scattered, elastically or inelastically, or absorbed. Elastic collisions result in no energy loss, or the energy loss is negligible compared to the incident energy. In this case, only the direction of the electron is changed. Elastic collisions occur primarily between incident electrons and atomic nuclei within the sample; energy losses are typically less than 1 eV for a beam of several keV energy.

Fig 5: An overview of the different signals used in modern electron microscopes. The direction shown for each signal does not always represent the physical direction. [7]

Fig.6: A depiction of the electron interaction volume and relative information depth for various signals. [5]

During inelastic scattering events, incident electrons transfer a larger portion of their energy to other particles within the sample. This is typical of interactions with the electron shells of atoms in the sample. Inelastic collisions can induce a large variety of secondary effects, such as the emission of secondary electrons and auger electrons, cathodoluminescence, and emission of X-ray photons.

Fig.8: Overview of a scanning electron microscope. [9]

5. Function of a scanning electron microscope

5.1. General

In an SEM, a focused electron beam is deflected by electric fields to scan the surface of a sample point-by-point. The beam is shaped by electromagnetic lenses. The observed image results from the interaction between the incident electron beam and the sample surface. When electrons in the beam hit the sample, secondary electrons (SE), backscattered electrons (BSE), and X-ray photons are produced. The brightness of a given pixel in an SE or BSE image encodes the number of electrons counted in the selected detector while the electron beam scans over the corresponding point. Different magnifications are obtained by choosing a smaller or larger area to be scanned. Scanning a smaller area with the same number of pixels makes the effective size of each pixel smaller, resulting in a larger magnification. Although the resolution of an image improves at higher magnifications, the resolving ability of the microscope itself is limited by the electron wavelength and the design and construction of the microscope, and cannot be improved by increasing magnification.

5.2. Secondary electrons

Secondary electrons are electrons originally bound to atoms in the sample that have been ejected by incident or backscattered electrons. The production of secondary electrons is an inelastic scattering event, i.e. the incident electrons lose energy and change their direction. The cross-section for these events increases with decreasing energy of the incident electrons and does not depend strongly on Z. Ejected secondary electrons have low energies. Because of their low energy, only secondary electrons produced close to the surface at depths less than 50 nm can escape the sample and be detected. As the secondary electron signal originates only from depths up to 50 nm, its intensity mostly depends on the topography of the sample surface and gives a high quality image of the surface structure. When a surface is tilted relative to the incident electron beam, the volume from which secondary electrons can exit the sample is large and a stronger signal is produced (see Fig. 10). Tilted surfaces thus appear brighter. This is also the case at edges and small pointed structures, which become highlighted in SE images.

The apparent “light source” in SE images is the SE detector. This detector is mostly positioned at an angle of about 45° in respect to the electron beam and at that side of the stage that the image appers to be illuminated from top, i.e., between 10 to 14 o’clock. Correct interpretation of images requires knowledge of the SE detector’s position relative to the sample.

Fig. 9: SE production as a function of the incident angle of the electron beam. [10]

5.3. Back-scattered electrons

Backscattered electrons are electrons which have been elastically scattered by atomic nuclei in the sample. The probability of an elastic scattering event is given by its cross-section σ_{el}. Its value depends on the incident electron energy: $\sigma_{el} \sim \frac{1}{E_0^2}$. Since the cross-section decreases with increasing electron energy, elastic scattering occurs less frequently at higher energies and incident electrons penetrate deeper into the sample. The elastic cross-section also depends on the atomic number of the analyzed sample: $\sigma_{el} \sim Z^2$. With increasing Z, the incident electrons are more frequently scattered. Atoms with larger Z also result in higher scattering angles, producing a shallower, wider interaction volume (Fig. 9). The Z-dependence of elastic scattering provides information about the composition of the sample. If a sample consists of two material phases with different Z, then the high-Z material will appear brighter in a BSE image.

Fig. 12: The dependence between the interaction volume of the electron beam (EB) and the acceleration voltage and the material atomic number Z. [10]

It is important to remember that incident electrons can undergo several elastic and inelastic collisions before reaching the surface and being detected. Therefore, electrons will not have discrete energies but rather an energy distribution as shown in Fig. 11. Electrons with energies close to the primary incident energy E_0 experienced only a few interaction events before leaving the sample. In practice, all electrons with energies below 50 eV are generally denoted as secondary electrons. Electrons with energies between 50 eV and the primary energy ($50\ eV < E < E_0$) undergo numerous inelastic scattering events before leaving the sample.

Fig. 11: Energy distribution of the BSE and SE leaving the sample surface. [10]

5.4. X-rays

In addition to forming images with secondary and backscattered electrons, an SEM can characterize the chemical composition of a sample by analyzing its X-ray emission. This is performed by using X-ray spectroscopy to sort detected X-ray photons based on energy. Elements in the sample can then be identified from spectral peaks corresponding to their characteristic emission lines (Fig. 12). These characteristic emission lines depend on the electron shell configuration of the atom. After an electron is ejected from lower shells by an incident electron, a photon is emitted as an outer-shell electron falls to a lower energy state to fill the resulting vacancy. The energy of the emitted photon depends on the location of the vacancy and the number of shells by which the replacement electron falls to reach the vacancy. Emission lines (e.g. Cu-Kα) are thus characterized by 1) the chemical element (Cu),

2) the vacancy location (K), and 3) the transition distance (α). In X-ray spectroscopy, the spectroscopic lines from the K, L and M shells are most often used.

An X-ray spectrum contains not only characteristic X-ray radiation but also X-ray radiation resulting from the deceleration of the electrons in the electrostatic potential of atoms in the sample. This is called Bremsstrahlung and is visible as background in the X-ray spectrum.

Fig.12: Schematic view of the origin of characteristic X-ray radiation [11] (left). Example spectrum (right) [12].

5.5. Depth of field in SEM images

In addition to having excellent resolution, SEMs produce images with large depth of field. Depth of field describes the depth range over which the image remains in focus. Because of the large depth of field of an SEM, surfaces with deep roughness can be imaged without blurring. The depth of field (D) depends on the magnification (M), the resolution limit (d) of the eye, the angular aperture (α) of the objective lens, and the diameter of the beam (d_b):

$$D = \frac{\frac{d}{M} - d_b}{\alpha}$$

The high depth of field of SEM images results from the very small aperture angle α of the focused electron beam.

6. Detectors

6.1. Detection of secondary electrons

For the detection of secondary electrons, an scintillator-photomultiplier detector is used. Usually it is mounted at an angle of about 45° from the incident beam direction and has a collector mesh. Between the sample and the detector's collector mesh a potential (up to approx. 300 V) is applied which attracts the low-energy secondary electrons to the detector. This attractive potential significantly increases the effective solid angle for detection of secondary electrons, since electrons originating from the far side of the sample also reach the detector. After reaching the detector mesh, electrons are further accelerated by an inner potential towards the scintillator where they deposit their energy and produce light pulses. These light pulses are then conducted through an optical fiber to the photomultiplier. In the photomultiplier the light signal is amplified and subsequently converted to an electrical signal. This signal is used by the electronics to generate the image.

This type of detector also detects backscattered electrons. As these electrons have high energies, the additional potential between the sample and the mesh has little influence on their trajectories. Therefore, only backscattered electrons exiting the sample towards the detector produce a signal. The number of backscattered electrons is therefore negligible compared to the number of detected secondary electrons. In principle, a BSE image can be formed using the SE detector by reversing the collector mesh potential to repel low-energy secondary electrons. However, this produces a noisy, low-quality image. An SEM has often another dedicated detector to form BSE images.

6.2. Detection of backscattered electrons

Semiconductor detectors are typically used to produce high-quality BSE images. These devices consist of a reverse-biased diode junction. With a reverse bias applied to the junction, a depletion layer forms. When energetic backscattered electrons impinge on the depletion layer, they deposit their energy and generate a number of electron-hole pairs which create a current pulse. This current pulse is then amplified and measured.

BSE detectors are generally located directly above the sample and are made as large as feasible to ensure a large solid angle for detection. To enable small working distances, the BSE detector is located at the end of the SEM column. The semiconductor is formed mostly into a two-segment ring to allow the electron beam to pass through the center. This location is optimal to highlight material contrast due to the Z-dependent scattering of electrons, but suppresses the physical effects that provide high-contrast images of the surface topography. Signals from the detector's two segments can be viewed in a variety of ways (e.g. added, subtracted, viewed individually) to produce images with different features of the sample.

6.3. Detection of X-rays

There are two common techniques to sort X-ray photons to form a spectrum with an SEM: energy-dispersive spectrometry (EDX) and wavelength-dispersive spectrometry (WDX). EDX is used for most general-purpose analysis. WDX provides improved spectral resolution, but requires significantly more time to form a spectrum, but the different energies are measured sequentially. Often, WDX is used to refine selected regions of an existing EDX spectrum. In this course, only EDX will be used.

Most EDX detectors use silicon semiconductor crystals to detect and sort X-ray photons, and have a functional principle similar to the BSE detector discussed in the previous section. In order to minimize noise in the measured spectrum, only high-purity crystals are used and the detector is cooled (e.g. with liquid nitrogen or thermoelectric coolers) to minimize thermal background. When a photon reaches the reverse-polarized depletion region of the diode junction, it generates a number of electron-hole pairs which create a current pulse. As the energy needed to create an electron-hole pair in silicon is 3.8 eV, the number of created electron hole pairs by a photon is large. Integration of the electrical pulse yields a measure of the incident photon's energy. Electrical pulses are then sorted by energy in a multi-channel analyzer to create an X-ray energy spectrum similar to that shown in Fig. 12.

7. Experiment

First, get acquainted with the microscope: look into the vacuum chamber, identify the different detectors and note their location relative to the sample stage. Always use gloves when handling samples.

Sample 1: Screw and coil filament

Exercise 1: Take images in SE mode and BSE mode

- Check the crossover: if it is not in the middle of the axis, move it there.
- Adjust brightness/contrast and bring the image roughly into focus.
- Perform fine adjustment of the crossover to maximize signal intensity.
- Focus your image in a high magnification. When the sample is in focus, click Z <-> FWD to set the working distance (Z).
- Check whether there is astigmatism; if so, correct it by adjusting the stigmator.
- Take images in the SE mode.
- Switch to the BSE mode. Readjust the crossover and contrast/brightness. Take some images.

Exercise 2: Record an EDX spectrum and identify the elements with the EDAX Genesis software

- Start the program EDAX Genesis and collect the spectrum by clicking button “Collect”. Ensure that the detector count rate is high enough to produce a good signal but the dead time is not too high.
- Identify peaks with Peak ID and consider whether the peak identification by the program is correct.
- If any peaks are missing after automatic identification, click the peak in the spectrum and identify it manually.

Sample 2: Sample with rough surface coating

Exercise 1: Take SE images of the sample and use measurement tools to add measurements to the image. Take BSE (A-B) images to demonstrate topography contrast with BSE.

Exercise 2: Rotate the image with scan rotation and consider how to identify peaks and valleys in rotated images.

Sample 3: Polished sample

Exercise 1: Take SE and BSE images.

Exercise 2: Take EDX spectra at various positions.

- Does the spectrum match your expectation? What does this tell you about the meaning of the BSE images.

Sample 4: Unknown sample

Exercise 1: Take images in SE and BSE (A+B), (A-B) modes.

- How do the BSE images compare to the SE image?

Exercise 2: Take an EDX spectrum.

- Manually identify the composition of the sample.

8. Evaluation

You are relatively free to format your evaluation as you see fit. You may write in either English or German. Please discuss the following:

- The motivation for using an SEM instead of an optical microscope. General capabilities and limitations of SEMs.
- The physical principles that produce the 3 most important signals, and how each signal can be used to analyze samples.
- The function of detectors for each signal and the characteristics of detectors that need to be considered when using an SEM.
- Each of the four samples. Discuss not only what the sample is and the analysis you performed, but also what the sample demonstrates about the use of SEMs in general. Pay specific attention to important factors that have to be considered when interpreting images and spectra produced by an SEM.

9. References

[1] Peter Fritz Schmidt et al.: „Praxis der Rasterelektronenmikroskopie und Mikrobereichsanalyse“, 1994, expert verlag, Renningen-Malmsheim

[2] Andrzej Barbacki: „Mikroskopia elektronowa“, 2007, Wydawnictwo Politechniki Poznanskiej, Poznan

[3] David B. Williams, C. Barry Carter “Transmission Electron Microscopy part 1 basics”, 1996, Springer Science+Business Media, New York

[4] https://lp.uni-goettingen.de/get/text/353

[5] wikipedia.de; wikipedia.org, wikipedia.pl

[6] https://www.onlinesehtests.de/astigmatismus/

[7] David B. Williams, C. Barry Carter “Transmission Electron Microscopy part 1 basics”, 1996, Springer Science+Business Media, New York

[8] http://wiki.polymerservice-erseburg.de/index.php/Transmissionselektronenmikroskopie

[9] http://www.technoorg.hu/news-and-events/articles/high-resolution-scanning-electron-microscopy-1/

[10] Peter Fritz Schmidt et al.: „Praxis der Rasterelektronenmikroskopie und Mikrobereichsanalyse“, 1994, expert verlag, Renningen-Malmsheim

[11] http://geo-consulting.warnsloh.de/rfaxrf-allgemein/

[12] Andrzej Barbacki: „Mikroskopia elektronowa“, 2007, Wydawnictwo Politechniki Poznanskiej, Poznan