

W. VAN DEN BROEK, A. PARVIZI, H. KIRMSE,
AND C.T. KOCH

SPATIALLY-RESOLVED ELEMENTAL ANALYSIS IN THE SCANNING ELECTRON MICROSCOPE

AG STRUKTURFORSCHUNG / ELEKTRONENMIKROSKOPIE,
HUMBOLDT UNIVERSITÄT ZU BERLIN, BERLIN

Copyright © 2017 W. Van den Broek, A.
Parvizi, H. Kirmse, and C.T. Koch

Published by AG Strukturforschung / Elek-
tronenmikroskopie, Humboldt Universität zu
Berlin, Berlin, in January 2017

www.physik.hu-berlin.de/en/sem/

This work is distributed in the hope that it
will be useful, but WITHOUT ANY WAR-
RANTY; without even the implied warranty
of MERCHANTABILITY or FITNESS FOR A
PARTICULAR PURPOSE.

Preface

This is the manual for the F-Praktikum on the Carl Zeiss GeminiSEM 500, a high-end SEM (scanning electron microscope) installed in 2016 at the AG Strukturforschung / Elektronenmikroskopie at the Humboldt Universität zu Berlin in Berlin.

Prof. Christoph T. Koch is responsible for the experiment, and the supervisors are Dr. Amin Parvizi, Dr. Wouter Van den Broek and Dr. Holm Kirmse. If you have questions or remarks, please refer to our contact details in the margin, or look here: www.physik.hu-berlin.de/en/sem/teaching/FPraktikum_SEM Dr. Peter Schäfer is responsible for the microscope itself; if none of the people listed above can be reached, you can try to contact him.

The manual aims at giving an overview of the technique in general and the physics behind it, as well as providing a step-by-step guide through the experiment. Care has been taken to make it as unambiguous as possible, however, if something is still unclear, rather than giving it your best guess, consult one of the supervisors instead lest you risk damaging the microscope.

The manual is heavily illustrated to make it as comprehensible as possible. We paid special attention to the software buttons and, just like in this¹ example, have often reproduced them in the margin right next to where they are mentioned. Furthermore, additional information was often relegated to the margin too, to prevent cluttering the main text.

The figures in Chapter 2 and those elsewhere that pertain to the Carl Zeiss hardware are courtesy of the Carl Zeiss Microscopy GmbH².

Prof. C.T. Koch
NEW 15, Room: 3'210
Tel: +49 (0)30 2093 **7640**
christoph.koch@hu-berlin.de

Dr. A. Parvizi
NEW 15, Room: 3'304
Tel: +49 (0)30 2093 **7658**
aparvizi@physik.hu-berlin.de

Dr. W. Van den Broek
NEW 15, Room: 3'305
Tel: +49 (0)30 2093 **7937**
vandenbroek@physik.hu-berlin.de

Dr. H. Kirmse
NEW 15, Room: 3'308
Tel: +49 (0)30 2093 **7641**
holm.kirmse@physik.hu-berlin.de

Dr. P. Schäfer
NEW 15, Room: 2'519
Tel: +49 (0)30 2093 **7894**
peter.schaefer@physik.hu-berlin.de



² Mit freundlicher Genehmigung von Carl Zeiss Microscopy GmbH.

Contents

1	Practicum goals	7
1.1	The Si-Ge sample	7
1.2	Practical and scientific goals	7
2	Working principles of the SEM and its detectors	9
2.1	Electron waves	9
2.2	The generation of characteristic X-rays	10
2.3	The SEM	10
2.3.1	The electron-optical setup	10
2.3.2	Detectors	11
2.4	The Bruker EDX spectrometer	12
3	Start-up and shut-down	15
3.1	Start-up	15
3.2	Shut-down	15
3.3	Setting up the workspace	16
3.4	Beam alignment	18
4	Experiments	19
4.1	Imaging Si-Ge islands	19
4.2	Energy-dispersive X-ray spectroscopy—EDX	20
4.2.1	Setting up the EDX system	20
4.2.2	Individual EDX spectra	21
4.2.3	Elemental maps	22
4.3	Influence of the EHT	23
4.4	Export data	23
5	Bibliography	25

1

Practicum goals

In this practicum you are expected to make yourself familiar with the operation of a scanning electron microscope (SEM). This means you need to understand the experimental setup of a SEM; the working principles of electron lenses; and how the various detectors work.

From the physics side of things, you need to learn about the elastic and inelastic scattering processes the electrons undergo in the sample and what type of signals they generate.

1.1 The Si-Ge sample

The sample used for the experiment¹ was grown by liquid phase epitaxy. Single-crystalline islands consisting of a mix of Si and Ge form in a self-organized manner on a [001]-oriented Si substrate. The sample is sketched in cross-sectional view in Figure 1.1a. The top-view presented in Figure 1.1b is what we expect to see in the SEM. The nominal Si-to-Ge ratio of an average island is 0.6 (Si) to 0.4 (Ge). The facets are {111} planes with an inclination angle of 55° . From transmission electron microscopy the height of an average island was measured to be 60 nm. Note the top facet cutting the tip of the island.

¹ C.L. Zheng, K. Scheerschmidt, H. Kirmse, I. Häusler, and W. Neumann. Imaging of three-dimensional (Si,Ge) nanostructures by off-axis electron holography. *Ultramicroscopy*, 124:108–116, 2013

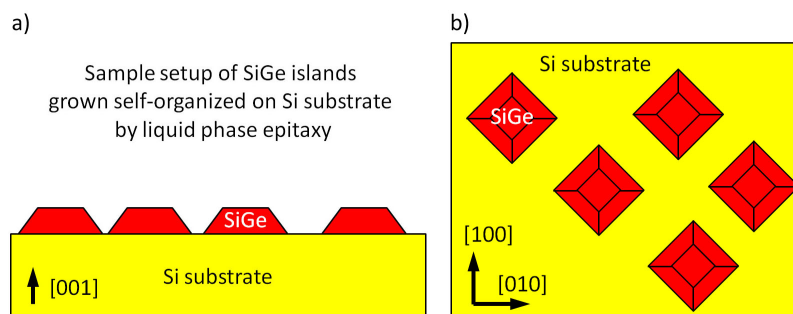


Figure 1.1: A cross-sectional view (a.) and a top-view (b.) of the Si-Ge sample.

1.2 Practical and scientific goals

From a more practical point of view, you need to

- learn how to operate and align the SEM;
- acquire a high-quality gray-scale image;
- map the chemical composition of a sample;
- investigate the effect of the electron-beam energy; and
- document and interpret your observations.

In your report please consider the following questions:

- Do you observe any difference in the detection efficiency of the two SE detectors (Inlens and SE2)? Explain.
- Do you observe any difference in the detection efficiency when changing the beam energy?
- Which characteristic X-ray energies have you used to quantify the Ge and Si concentrations?
- Which electronic transitions do these correspond to?
- Explain the dependence of the measured Ge:Si ratio in the EDX maps on the accelerating voltage.
- Calculate the height of the Si-Ge islands. Does it correspond to the results in²?

² C.L. Zheng, K. Scheerschmidt, H. Kirmse, I. Häusler, and W. Neumann. Imaging of three-dimensional (Si,Ge) nanostructures by off-axis electron holography. *Ultramicroscopy*, 124:108–116, 2013

2

Working principles of the SEM and its detectors

2.1 Electron waves

Louis de Broglie in his 1924 doctoral thesis ¹ proposed the wave nature of mass-carrying particles, giving them the wavelength

$$\begin{aligned}\lambda &= \frac{h}{p} = \frac{h}{\sqrt{\frac{E_{total}^2 - m_0^2 c^4}{c^2}}} \\ &= \frac{hc}{\sqrt{(E_{kin} + m_0 c^2)^2 - m_0^2 c^4}} = \frac{hc}{\sqrt{eV_0(2m_0 c^2 + eV_0)}}\end{aligned}\quad (2.1)$$

(h being Planck's constant, m_0 the electron rest mass, and $E_{kin} = |eV_0|$ the kinetic energy of the electron, with e being its charge and V_0 the accelerating voltage) is very short, almost 2 orders of magnitude shorter than typical X-ray wavelengths. For high electron beam energies, i.e. $E \geq 20$ kV, the wavelength can be approximated by the following expression:

$$\lambda \approx -0.0040\text{\AA} + \frac{0.408\text{\AA}}{\sqrt{E/\text{keV}}}\quad (2.2)$$

but as the beam energy approaches the rest energy of the electron the more precise expression (2.1) should be used.

beam energy (keV)	1	2	5	10	20	30
wavelength (pm)	38.76	27.40	17.3	12.20	8.59	6.98

The electric charge of the electrons not only allows them to be accelerated by electrostatic fields, but also deflected and focused by both, magnetic as well as electric fields according to the Lorentz force

$$\vec{F}_{\text{Lorentz}} = -e \left(\vec{E} + \vec{v} \times \vec{B} \right)\quad (2.3)$$

In 1927 Hans Busch suggested that the magnetic field of a short coil could be used to focus beams of electrons analogous to the way light is focused by convex optical lenses ². In 1931 Ernst Ruska and his doctorate adviser Max Knoll made use of this discovery in their development of the transmission electron microscope (TEM) ³, and soon after, in 1937, Manfred von Ardenne developed the scanning electron microscope in an attempt to overcome limitations due to the chromatic aberration in the TEM ⁴.

¹ Louis de Broglie. *Recherches sur la theorie des quanta*. PhD thesis, Sorbonne, 1924

Table 2.1: Electron wavelengths at different accelerating voltages

² Hans Busch. Über die Wirkungsweise der Konzentrierungsspule bei der Braunschen Röhre. *Arch. Elektrotech.*, 18:583, 1927

³ M. Knoll and E. Ruska. Das Elektronenmikroskop. *Z. Phys. A*, 78:318–339, 1932

⁴ Manfred von Ardenne. Improvements in electron microscopes., February 18 1937. GB 511204 (Germany)

2.2 The generation of characteristic X-rays

As shown in Figure 2.1, a fast electron impinging on an atom may transfer some of its kinetic energy to one of the electrons bound to the atom. This atom may then escape and be potentially detected as a secondary electron. In case the orbital from which the bound electron was removed is one of the inner shell orbitals, the vacancy it left behind will quickly be filled by an electron from an orbital with a higher (less negative) potential energy. The difference in the binding energy of these two orbitals is then emitted as a photon. The energies of these X-ray photons is characteristic for the species of atom that was hit by the electron beam. A list of such characteristic X-ray energies can be found at this link: www.bruker.com/fileadmin/user_upload/8-PDF-Docs/X-rayDiffraction_ElementalAnalysis/HH-XRF/Misc/Periodic_Table_and-X-ray_Energies.pdf

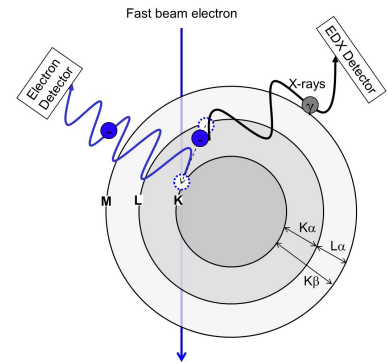


Figure 2.1: Illustration of an inelastic interaction of a fast electron with an atom, generating characteristic X-rays and secondary electrons.

2.3 The SEM

In this practicum you will operate a Carl Zeiss GeminiSEM 500, a high-end SEM installed at Humboldt University in 2016. This SEM is equipped with a range of detectors, but in this practical you will only use the SE detector and the EDX spectrometer.

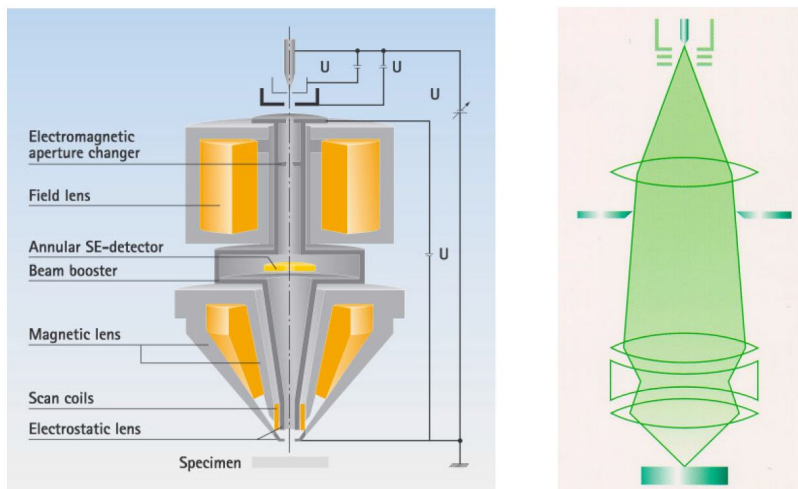


Figure 2.2: Diagram showing the electron optical setup of the GeminiSEM 500.

2.3.1 The electron-optical setup

Fig. 2.2 shows the electron optical setup of the SEM you will be using in this practical. The objective lens is equipped with an electrostatic lens at its end. This has the advantage that the specimen itself is not immersed in a magnetic field, while still enabling a spatial resolution of 0.6 nm (and better if the transmitted electrons are detected and the specimen is sufficiently thin). This is particularly useful for investigating magnetic specimen. Most other

high-resolution SEMs are equipped with immersion lenses in which the specimen is exposed to a strong magnetic field.

Another advantage of the electrostatic lens is that slow electrons which escape the surface of the specimen are collected very efficiently and are accelerated into the electron optical column and can be detected very efficiently by the inlens detectors inside the microscope column.

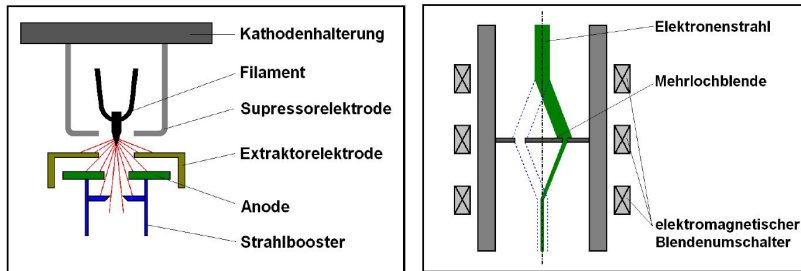


Figure 2.3: Left: Working principle of the field emission gun in the GeminiSEM 500. Right: The beam current and size of the focused spot on the sample may be adjusted by deflecting the beam through differently sized aperture holes. The smallest aperture provides the highest lateral resolution and is located in the center of this aperture. For EDX spectroscopy a larger current is required, so a larger aperture should be used.

2.3.2 Detectors

When the electron beam hits the specimen, it will be scattered by the atoms they encounter on their path with some probability. Depending on their kinetic energy they will be stopped more or less quickly. Their penetration depth also depends on how heavy the atoms are, they are scattered by. The interaction volume is shown on the left hand side of Fig. 2.4 (taken from⁵). The different depth ranges of where different signals may be generated are due to the fact that electrons with low energy cannot escape from deep within the specimen. High-energy back-scattered electrons, for example have energies close to the primary beam energy and may thus escape from deep within the specimen. Secondary electrons generated deep within the sample will be absorbed before they can make it to the surface. Electrons that are absorbed deep within the sample may generate X-rays that may then easily escape, because the scattering cross section of X-rays with matter is more than 5 orders of magnitude smaller than that of electrons, and even more at electron energies available in an SEM.

⁵ J. Ackermann. Carl Zeiss SMT Ltd, Oberkochen, 2004

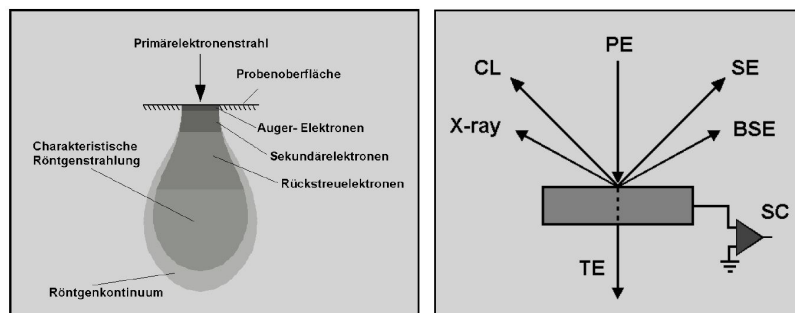


Figure 2.4: Left: the scattering volume contributing to different detectable signals in the SEM. Right: Different signals that can be detected when a sample is hit by high-energy primary electrons (PE): transmitted electrons (TE), secondary electrons (SE), back-scattered electron (BSE), Cathodoluminescence (CL), X-rays, and an electron beam induced specimen current (SC/EBIC).

As shown on the right hand side of Fig. 2.4, a multitude of sig-

nals is produced when the electron beam impinges on the surface of the sample. The SEM used in this practicum has detectors for some of these, namely: transmitted electrons (TE), secondary electrons (SE), back-scattered electron (BSE), and X-rays.

Fig. 2.5 (taken from⁶) shows more clearly the different possibilities for secondary electrons to be generated, distinguishing between:

- SE1 electrons that are generated at the position where the focused electron beam impinges on the specimen.
- SE2 electrons are generated in the course of multiple scattering events which reach the sample surface at some distance from the point where the primary beam hits the surface.
- SE3 electrons that are generated by back scattered electrons somewhere else in the column, or on the specimen. These will hardly be detected.

Fig. 2.6 shows the SE and BSE detectors available in the GeminiSEM 500. The in-lens SE detector is efficient in collecting the SE1 and SE2 electrons that are emitted very close to the position of the probe, especially at low beam energies, because the electrostatic lens accelerates the emitted electrons and thus sucks also the very slow electrons into the column. The Everhart- Thornley- Detector (SE2) detector detects both secondary and back-scattered electrons.

⁶ J. Ackermann. Carl Zeiss SMT Ltd, Oberkochen, 2004

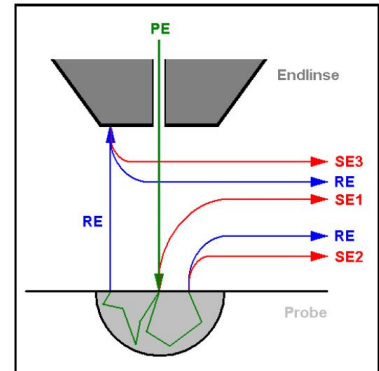


Figure 2.5: Diagram illustrating the origin of back-scattered electrons (BSE) and different types of secondary electrons (SE, SE2, SE3).

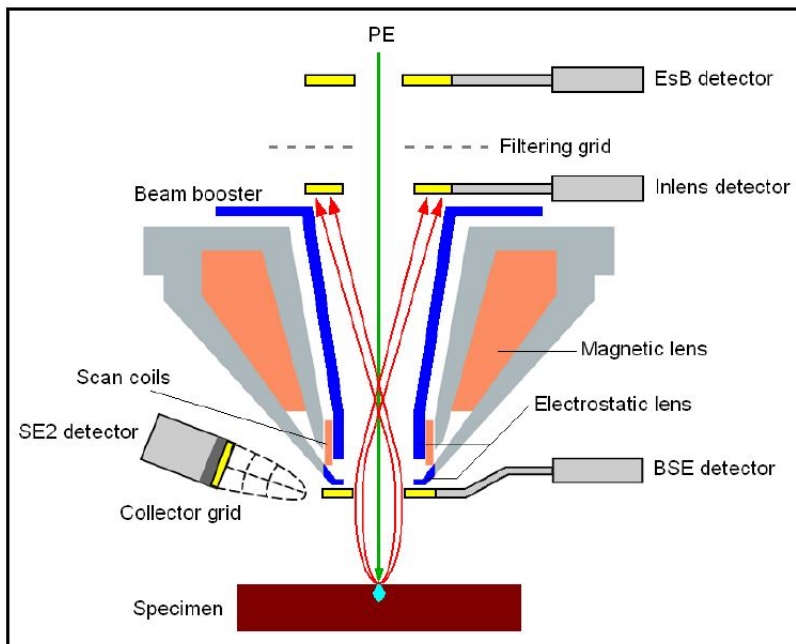


Figure 2.6: Different detectors available within the microscope: energy-selective back-scatter detector (EsB), inlens SE detector, in-chamber SE2-detector (also called Everhart- Thornley- Detector), and a segmented back scatter detector which can be inserted, positioning itself immediately under the lens. The EDX detector and transmission (STEM) detector are not shown in this diagram.

2.4 The Bruker EDX spectrometer

The EDX spectrometer installed on this SEM is a Bruker Quantax 400 - Z30 system using a 30 mm² Xflash 6|30 and contains a Peltier-

cooled solid state detector (a silicon drift detector made of high purity silicon with a very low leakage current). Please familiarize yourself with the technology that allows energy-dispersive detection of X-ray photons. An illustrative animation can be found here: www.ketek.net/products/sdd-technology/working-principle/

3

Start-up and shut-down

In this chapter we detail start-up and shut-down of the microscope, loading the sample, and making a first, rough, beam alignment.

3.1 Start-up

Warning: You are not permitted to execute the steps in this section and the next (Sections 3.1 and 3.2) without one of the assistants being present! This is to avoid damage to the microscope.

As illustrated in Figure 3.1, the sample is attached to a stub set in a multi-stub holder. In order to insert it into the SEM, the following steps must be followed.

- Vent the chamber
 - Press Vent¹ in the Vac menu² in the lower right corner of the left-most screen
 - Wait for one minute
- Slide open the door
- Insert the holder
 - Wear gloves to avoid contamination and vacuum problems
- Close the door
- Pump the chamber
 - Press Pump in the Vac menu³
 - Wait for approx. 5 minutes until the vacuum is ready, indicated by a green tick next to the Vac button
- Start up the gun (only if it was off, see Sec. 3.2)

3.2 Shut-down

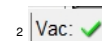
To shut-down and unload the sample, follow these steps.

- If you have done EDX
 - Retract the EDX detector by clicking this button.⁴

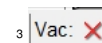


Figure 3.1: A multi-stub holder for seven stubs, with one stub inserted; the latter supports the specimen.

¹ Hear plops and hisses



²

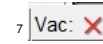
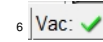
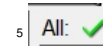


³



⁴

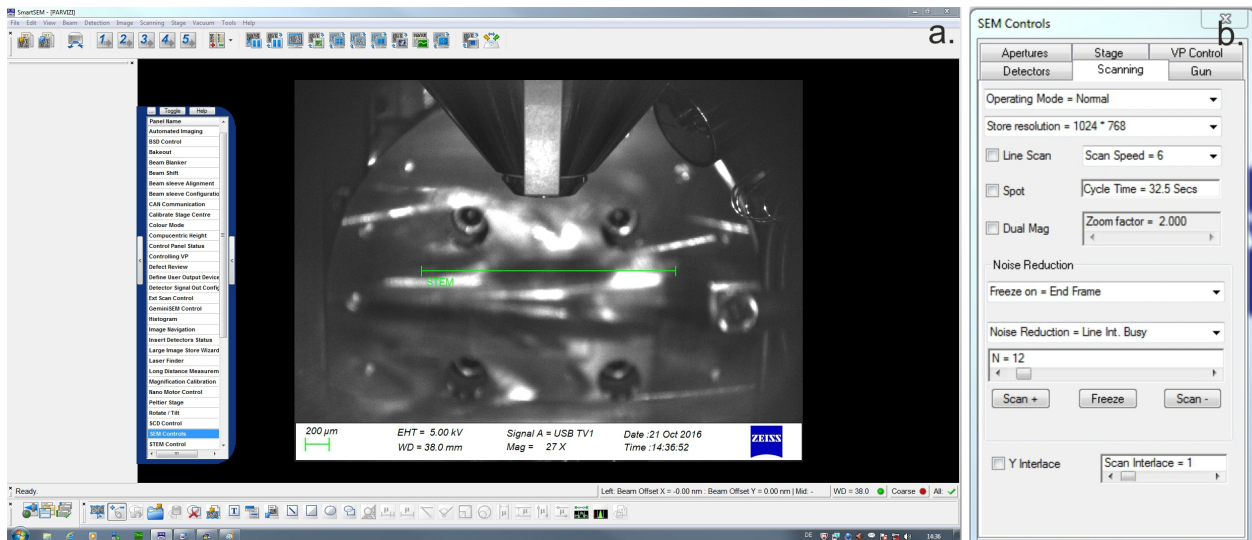
- Close Esprit
- Turn off EHT from the menu in the All button⁵
- Press Vent in the Vac menu⁶
 - The Stage is lowered automatically
- Remove the Specimen holder
- Close the door
- Press Pump in the Vac menu⁷
 - Wait for approx. 5 minutes until the vacuum is ready, indicated by a green tick next to the Vac button
- Close the SEM program
- Turn the computer off
- Press the Standby button⁸ located below the SEM chamber



⁸ **Caution:** Do not press the Off button, this causes vacuum problems.

3.3 Setting up the workspace

On the left-most computer screen, you see a live stream of the inside of the SEM, recorded with an infrared (IR) camera, see Figure 3.2.a. The sample holder is located at the bottom and the objective lens is the large up-side-down cone in the upper part. Open the SEM Controls from the side bar to open the window in Figure 3.2.b that will be used for adjusting most settings in the following sections.



The joystick on the far-left lets you control the sample height; try it out gently while keeping an eye on the IR camera to avoid driving the stage into the objective lens. The joystick to the right of that lets you move the sample in the horizontal directions. See Figure 3.3. The Stage Navigation menu on the right screen shows

Figure 3.2: (a.): A view of the SEM's inside with an infrared camera. Click on SEM Controls in the slide-out menu in the side bar to open the SEM Controls menu in Fig. 3.2.b. (b.): The SEM Controls menu that controls most of the SEM's settings.

you how your sample is oriented and lets you ride to one of the pre-defined stub-locations simply by clicking on the desired spot; see Figure 3.3.

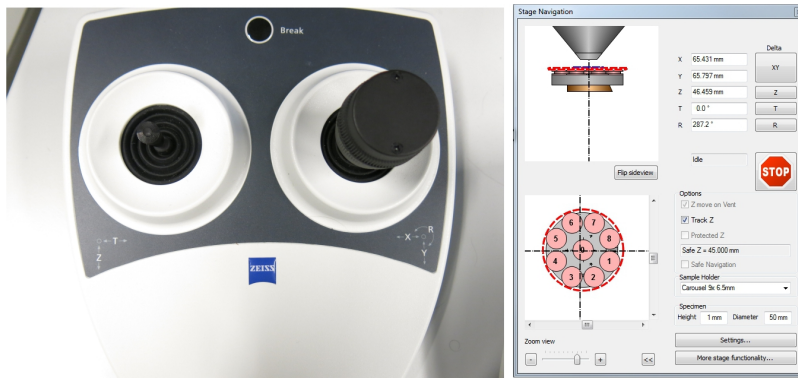


Figure 3.3: The joysticks and the Stage Navigation menu.

The Control Panel lets you control the most frequently needed settings directly without having to browse through all kinds of software menus. Have a look at Figure 3.4 and see if you can locate the following buttons: Stigmator X, Stigmator Y, Magnification, Reduced, Aperture X, Aperture Y, Wobble, Freeze, Camera, Brightness, Contrast, Focus, and +/-; these will be important later on, and we'll explain their use in due time.



Figure 3.4: The Control Panel.

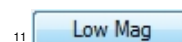
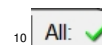
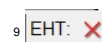
The image often looks saturated or under-illuminated. This means that the brightness and contrast are not well adjusted. To have the brightness and contrast set automatically, select the Detectors-tab in Sem Controls and set Signal Adjust to BC. If later on you're unhappy with the automatic setting, you can choose here Auto BC = Off instead, and use the Brightness and Contrast buttons on the Control Panel.

3.4 Beam alignment

Each session on the SEM starts with a rough beam alignment to produce a beam of sufficient quality to locate the specimen and to navigate over it.

- Set the EHT to the desired value of 15 kV
 - Press EHT On in the EHT menu⁹ in the lower right corner of the left-most screen
 - Wait for the green tick to appear next to the All button¹⁰
 - Open the Gun tab in the SEM Controls and set EHT Target to 15kV
- In the Apertures tab in SEM Controls, choose Aperture Size (1) 20.00 μm -Standard
- Drive the sample stage to the green line in the IR camera using the left-most joystick
- Choose the Low Mag button¹¹ in the Apertures tab in the SEM Controls
- Choose the SE2 detector in the Detectors tab of the SEM Controls
- Switch the live stream to the SE2 detector by pressing the Camera button on the Control Panel
- Bring the stage to the correct stub by clicking on the respective stub in the scheme in the Stage Navigation menu
- From now on do all the finer stage movements with the joystick to the right
- Iterate between the following until you have an acceptable image:
 - Make the image as sharp as possible with a combination of Focus¹², and Stigmation X and Stigmation Y¹³ on the Control Panel
 - Center the aperture on the optical axis using the Wobble function and the Aperture X and Aperture Y buttons on the Control Panel¹⁴

Now leave Low Mag and go the normal magnification regime by pressing the Resolution button¹⁵ in the SEM Controls. Bring a large feature in your field of view and keep it focused by adjusting Focus, Stigmation X and Stigmation Y as needed, while you gradually ramp up the magnification to about 10k by making use of the Magnification knob on the Control Panel. You can also use the Reduced knob on the Control Panel to work quicker on only a selection of the image, see Figure 3.5.



¹² Defocus makes the whole image appear blurry; make it as sharp as possible

¹³ Astigmatism makes the image look blurry in a certain direction only. Tune it until the image appears sharp.

¹⁴ Adjust the aperture's x and y-position until the wobble becomes *concentric*.

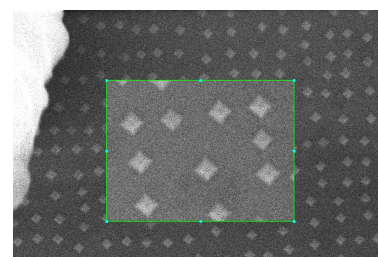
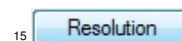


Figure 3.5: The inner region of this image is the so called reduced image, brought about with a press on the Reduced button.

4

Experiments

Now that the preparatory work has been done, it is time to start with the experiments themselves, namely imaging the Si-Ge islands, investigating the chemical composition of the sample and testing the influence of the electron-beam energy.

For the overall goals of the experiments and the scientific questions to answer, we refer you to Section 1; the current section strictly deals with the technical realization and practicalities. If you get delayed while performing the experiments, you should consider skipping Section 4.2.3 where elemental maps are recorded. Each elemental map takes about half an hour, and you shouldn't forget that you're expected to try out two different beam energies.

4.1 Imaging Si-Ge islands

First we image the Si-Ge islands as sharply as possible.

- Keep the EHT and the aperture to the values from the previous section (15 kV and 20 μm)
- Switch the live stream back to the IR camera¹
- Set the working distance WD to 3 mm with the left-most joystick²
- Select the InLens detector in the SEM Controls
- Bring the magnification up to 50k, while you keep the image focused with the Focus button
- Now go to 100k magnification and sharpen the image with a combination of Focus, Stigmation X, Stigmation Y, and Wobble, Aperture X and Aperture Y
- Bring the magnification back down to 50k; your image should now be as sharp as it gets; see Figure 4.1
- Record the image by pushing the Freeze button on the Control Panel
- Save by right-clicking and exporting as tiff

¹ With the Camera button on the Control Panel

² WD can be found in the figure legend on the left-most screen; be careful not to drive the stage in the objective lens.

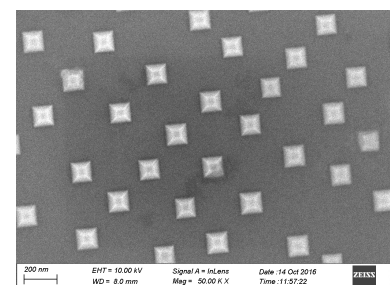


Figure 4.1: Si-Ge islands on a Si substrate, see Sec. 1 for an explanation.

If you're not satisfied with the noise properties of the image, experiment with a longer recording time³ and/or some of the noise reduction settings in the Scanning tab of the SEM Controls.

³ The +/- buttons on the Control Panel

4.2 Energy-dispersive X-ray spectroscopy—EDX

In this part of the experiment, we record EDX spectra at the center of a Ge-island and in the Si-matrix, and we do an elemental mapping of Si and Ge.

4.2.1 Setting up the EDX system

For recording of the spectra, we need to set the system to the following settings.

- The EHT is still kept at 15 kV
- Set the aperture to 60 μm ⁴
- Go down to a magnification of 10k
- Make the image sharp with a combination of Focus, Stigmation X, Stigmation Y, and Wobble, Aperture X and Aperture Y
- Set the working distance (WD) to 8 mm in several steps while keeping the image sharp with Focus⁵
- Now go up to a magnification of 100k
- Focus the image with a combination of Focus, Stigmation X, Stigmation Y, and Wobble, Aperture X and Aperture Y
- Go down to a magnification of 50k⁶

⁴ See Apertures tab in SEM Controls

⁵ Keep an eye on the WD value and toggle to the IR camera to double check that the stage is going *down* and not *up* into the lens.

⁶ The image is now as sharp as possible, but not as sharp as in the previous section; do you know why?

We now start the EDX software, Esprit. It will take over most of the control of the SEM.

- Open the Esprit software by clicking the hyperlink⁷ on the right-most computer screen
- Log in:
 - User: Praktikum
 - Password: *****



7

The EDX detector needs to be inserted and we need to make sure the InLens detector is still selected.

- Insert the EDX detector by clicking this⁸ button in the upper right of area of Esprit
- Monitor insertion on the IR Camera, see Figure 4.2
- Wait until the detector is inserted *completely*, as indicated by the progress bar and the button changing to⁹



8



9

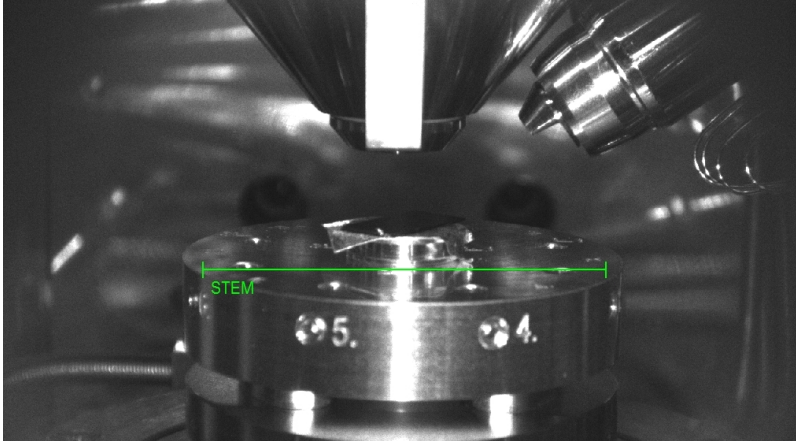


Figure 4.2: The EDX detector (conical object to the right of the objective lens) is inserted completely.

- Switch away from the IR Camera and check in SEM Controls to ensure the Inlens camera is selected

It now remains to ensure the detector settings are safe, so that no overexposure will occur.

- Check the kcps (kilocounts per second) indicator¹⁰ on the upper right
 - It should be about 65
 - If it's higher, *stop working* and call an assistant
- Right-click the Acquire button¹¹, select Counts and set it to 100k¹²
- While working, *always mind the intensity*: the indicator¹³ in the upper right corner must be *green*, when it is *red* it is too intense and the detector will get damaged

10 66 kcps

11 Acquire ▼

¹² This will cause the recording to last until 100k counts are reached.

13 

4.2.2 Individual EDX spectra

We are ready to record two EDX spectra, one on a Si-Ge island, and one on the bare Si-substrate.

- Click the Preview button
- Esprit takes over the microscope and produces an image like in Figure 4.3, use the overlay to manually adjust Brightness and Contrast
- When you are satisfied with the preview, record an auxiliary image with the Capture button¹⁴
- Then click the Record Spectra button¹⁵ and subsequently the Spectrum Location button¹⁶ and use it to indicate the spectra locations, like in Figure 4.3
- Now press the Acquire button¹⁷ to record the spectra

14 Capture ▼

15 

16 

17 Acquire ▼

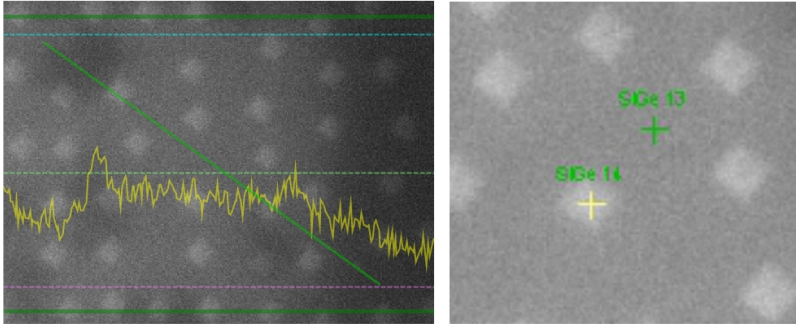


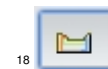
Figure 4.3: *Left*: Preview image, produced by Esprit. *Right*: The user can indicate the positions of the spectra on the sample.

Now that the spectra have been recorded, we'll apply quantification to find out which elements the peaks belong to.

- Press the Periodic Table button¹⁸
- The periodic table pops up; choose Si and Ge, and verify that the indicated peaks coincide with the measured ones

Export the data by first adding it to the current project with the aid of the I/O button¹⁹. Then export the spectra as text or as xlsx files.

Important! If you decide not to do the elemental maps, **remove the EDX detector** by clicking this button.²⁰



4.2.3 Elemental maps

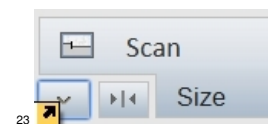
In this section we describe how to record a Si- and a Ge-map. If you are strapped for time you could skip this part of the exercise. When making your decision, don't forget that at the end you are expected to repeat the experiments so far at a different beam-energy.

The mapping time will be set to Manual, meaning that you have to stop the recording by hand when you deem it good enough. The mapping takes about half an hour and since the sample cannot be expected to remain still for this long, it will be necessary to apply drift correction.

- Switch Esprit to elemental mapping mode by clicking the Element Map button²¹
- Click the Preview button; the preview image now has a green rectangle surrounding the region of the elemental map
- Right-click the Acquire button and set
 - Map time to Manual
 - Map area to Variable
 - Map width and Map height to approx. $2 \mu\text{m}$ ²²
- Set the Scan configuration by clicking the Scan configuration button²³



²² Make sure that the map encompasses approx. 10 islands.



- Set Image resolution [pixel] to 512
- Set Image input to Ch 1
- Under Mapping set Dwell time [μ s] to 64
- Click the Drift correction button to set these drift correction values
 - * Image input to Current input
 - * Correction interval to 10 s
 - * Dwell time to Current value
- Activate the drift correction by clicking this button²⁴ in the Scan field
- Click the Periodic table button²⁵ to select Si and Ge
- Press the Capture button to verify the SEM scans the desired region
- Finally, press the Acquire button to record the elemental maps
- When you can clearly distinguish the islands in the maps, again press Acquire to stop acquisition

If you want, you can try out different colors for the maps. But while you are exploring the menu, *do not press this button*²⁶ as it seems to cause a software crash.

To save your results, use the I/O button²⁷ by right-clicking and exporting as raw. Do not forget to use this button to export the results to the current Project as well. *Be vigilant when saving*, check the size of the saved files²⁸; furthermore, save the elemental maps as images (tiffs) too, just to make sure.

Important! Remove the EDX detector by clicking this button.²⁹

4.3 Influence of the EHT

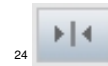
Repeat Sections 4.1 and 4.2 with an EHT of 5 kV.

Close Esprit so that it gives up its control of the SEM. **Important! Before closing Esprit, remove the EDX detector** by clicking this button.³⁰

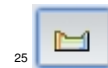
Refer to Section 3.4 for instruction on how to switch the EHT. The amount of counts to expect for EDX is about 5 kcps.

4.4 Export data

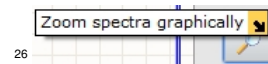
No USB sticks allowed!³¹ To export your data we recommend you use the physics department's cloud service at cloud.physik.hu-berlin.de/



24



25



26



27

²⁸ A small file could not contain the complete data cube.



29



30

³¹ Do you want a computer virus? Because that's how you get a computer virus.

5

Bibliography

- [1] J. Ackermann. Carl Zeiss SMT Ltd, Oberkochen, 2004.
- [2] Hans Busch. Über die Wirkungsweise der Konzentrierungsspule bei der Braunschen Röhre. *Arch. Elektrotech.*, 18:583, 1927.
- [3] Louis de Broglie. *Recherches sur la theorie des quanta*. PhD thesis, Sorbonne, 1924.
- [4] M. Knoll and E. Ruska. Das Elektronenmikroskop. *Z. Phys. A*, 78:318–339, 1932.
- [5] Manfred von Ardenne. Improvements in electron microscopes., February 18 1937. GB 511204 (Germany).
- [6] C.L. Zheng, K. Scheerschmidt, H. Kirmse, I. Häusler, and W. Neumann. Imaging of three-dimensional (Si,Ge) nanostructures by off-axis electron holography. *Ultramicroscopy*, 124:108–116, 2013.