Practical course for the lecture
Electron Microscopy I

Scanning Transmission Electron Microscopy
(winter term 2023/2024)

Supervisors

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Introduction

In this experiment it is demonstrated that in the scanning mode of an electron microscope not only information about the surface properties (topography contrast) and chemical composition (material contrast) of solid samples can be obtained, but also the internal properties of extremely thin samples (typically some 10 nm in thickness) can be characterized. In this case, we speak of scanning transmission electron microscopy (STEM), which can be performed in conventional scanning electron microscopes at relatively low electron energies (about 10 - 30 keV) using suitable sample holders, but is more commonly done in combined TEM/STEM instruments at accelerating voltages of typically 80 - 300 kV. Important basic requirements for this are illumination optics that allow the generation of a fine electron probe (1 nm diameter and smaller), and the presence of appropriate scanning coils to scan the probe across the electron-transparent TEM sample. In addition, suitable electron detectors (usually in the form of scintillators) such as a bright-field and dark-field detectors must be available below the sample.

In the course of the experiment, the adjustment of a transmission electron microscope for STEM operation and its basic properties will be taught, and typical application possibilities will be demonstrated. Since the STEM beam path corresponds to that of the convergent beam electron diffraction (CBED), it will be demonstrated in this context that CBED analyses of the structural properties of materials can also be carried out using STEM. The content of the experimental work will focus on the application of the different imaging modes, i.e. bright-field and dark-field imaging, to characterize different materials. In particular, it will be studied which instrumental parameters (especially detection angle range) have to be chosen in the case of STEM dark-field imaging with the ring detector for electrons scattered in wide angles (high-angle annular dark field - HAADF) in order to obtain atomic-number (Z) dependent image contrast. Similarly, we will investigate how diffraction-related contrast can be enhanced in STEM bright-field imaging.

Two different electron-transparent samples are used for the experiment. The first sample consists of a mixture of silica (SiO_2) and gold nanoparticles deposited on a thin carbon support film. The aim is to investigate the properties of nanoparticles, which are of great interest for different applications. Due to their special features, such as extremely large surface-to-volume ratio, nanoscale particles are widely used - for example, a specific and long-established field of application is catalysis. In order to optimize the catalytic efficiency of metallic nanoparticles, their size, shape, structural nature, and chemical composition must be known. In general, electron-microscopic investigations are indispensable for this purpose. The characterization in STEM mode has some advantages compared to conventional TEM mode, which will be demonstrated practically during the experiment.

The second sample consists of a semiconductor heterostructure consisting of an about 2 μm GaAs layer epitaxially grown on a Si(001) substrate. In general, semiconductor heterostructures have been of great application relevance since the beginning of microelectronics. Significant insights into the (opto)electronic properties of semiconductor devices are based on electron-microscopic studies of defects and the microchemistry of heterolayer systems contained therein. Specifically, by using TEM and STEM in combination with analytical techniques (e.g. energy-dispersive X-ray spectroscopy - EDXS), information is available on the structural/chemical nature of the respective layer and its interface with neighboring sample regions such as the substrate. This will be presented by the example of the investigation of a GaAs/Si(001) heterostructure prepared by molecular beam epitaxy (MBE: evaporation in ultra-high vacuum). The mismatch of the lattice constants of GaAs and Si (a_{Si} = 0.5431 nm, a_{GaAs} = 0.56535 nm) is approximately 4%. The TEM sample was prepared so that the approximately 2 µm thick GaAs layer grown on the Si wafer is visible in cross section. The layers should be transmitted along the [110] direction of the Si crystal lattice. Due to the lattice mismatch and the thickness of the GaAs layer, there are relatively many crystal structure defects (dislocations, stacking faults) at the interface between GaAs and Si substrate as well as inside the GaAs layer. Within the course of the experiment, it should be investigated under which experimental conditions such crystal defects can be visualized in good quality.

Samples

- Silica (SiO_2) and gold nanoparticles of different sizes on a carbon support film (3 mm copper mesh),
- MBE-grown GaAs layer (ca. 2 µm thickness) on Si(001) substrate; from this layer system a TEM cross-section sample was prepared in a conventional way (the steps of preparing a cross-section sample are briefly explained by the supervisor).

Equipment

- TEM/STEM: 200 kV transmission electron microscope FEI Tecnai Osiris (building 30.25 [CFN], room no. -108)

Experiment preparation

Using the lecture notes and handed out literature, please read up on the following topics:

- Components, set-up, and operation of a scanning transmission electron microscope.
- What are the detectors and different imaging techniques in STEM operation?
- What is meant by the reciprocity principle and in what way diffraction-related image contrast can be particularly obtained in STEM bright-field imaging?
- On which sample properties do the image contrasts in STEM bright-field and STEM dark-field imaging (especially HAADF-STEM imaging) depend?
- How do imaging parameters (especially detection-angle range of bright-field and ADF detector) affect the observed image contrast?
- Further questions - see experimental procedure.

Experimental procedure

Important: Document the performed STEM experiments with selected images, so that you can show all experiments well in the protocol.

1. ***Adjustment of the 200 kV transmission electron microscope in STEM mode*** (this will be demonstrated and explained by the supervisor; please describe the main adjustment steps in the protocol).

2. ***STEM examination of the samples.***

Perform the following investigations and please answer the associated questions:

A) Silica/gold nanoparticles.

1. Take STEM images of a sample area with both types of particles using BF(bright-field) and HAADF detector and describe the image contrast to be observed. How do the HAADF images differ for silica and gold particles? Based on your investigations/images, compare in a few words the parameters on which the HAADF signal intensity depends?

2. Describe the BF-STEM contrast of the two types of particles obtained from the previously selected image area. In what way and why do the BF images of the silica and gold nanoparticles differ from the corresponding HAADF images? To what extent does the structure, i.e. amorphous (silica) or crystalline (gold), influence the image contrast?

3. Determine exemplarily for the BF-STEM - as well as for the corresponding HAADF-image for a selected SiO_2 or Au particle the signal course (intensity line profile) across the center of the selected particles and discuss the obtained profile courses.

4. Take an X-ray element map of a sample area with both types of particles to complement the information obtained by BF/HAADF imaging. What is the advantage of this analytical imaging technique?

B) GaAs/Si(001) heterostructure

1. Get an overview of the structural nature of the GaAs/Si(001) heterostructure by taking STEM BF and HAADF images at medium microscope magnification. How does the image contrast of the GaAs layer differ from that of the Si substrate and what are the reasons for the observed differences?

2. Tilt the sample in the silicon region to a <110> zone axis orientation (Laue condition) and take a CBED image with a C2 aperture of 150 µm. The camera length should be chosen such that as many diffraction disks as possible can be seen. Repeat this experiment with a C2 aperture of 50 µm. Describe qualitatively the differences between the two CBED images. Index the diffraction patterns and determine the electron-probe convergence angle for both C2 aperture diameters.

3. Acquire STEM bright-field images of the GaAs layer at medium and high magnification and two very different camera lengths (e.g., 43 mm and 550 mm) under two-beam conditions, e.g., with strongly excited 2-20 reflection. Describe the image contrast visible in the GaAs/Si transition

region and within the GaAs layer as a function of the camera length. Under which experimental conditions are diffraction-induced contrast differences more pronounced, especially in the region of crystal structure defects, and what is the reason?

Protocol

Prepare a protocol in which you answer the questions in “2. ***STEM examination of the samples***” by using the recorded images. Give the answers or explanations as briefly and concisely as possible. You may use graphs from lecture or literature (with references) to explain questions.

Literatur

J. Rodenburg, Optimising the resolution of TEM/STEM with the electron Ronchigram, Microscopy and Analysis, July 2002

P. D. Nellist, The Principles of STEM Imaging, In: Scanning Transmission Electron Microscopy – Imaging and Analysis, Eds. S.J. Pennycook, P.D. Nellist, Springer Verlag, 2011, pp. 91-115

J. Liu, Scanning transmission electron microscopy and its application to the study of nanoparticles and nanoparticle systems, Journal of Electron Microscopy, **54** (2005), 251-278