



Composition quantification of electron-transparent samples by backscattered electron imaging in scanning electron microscopy



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ABSTRACT

The contrast of backscattered electron (BSE) images in scanning electron microscopy (SEM) depends on material parameters which can be exploited for composition quantification if some information on the material system is available. As an example, the In-concentration in thin $\text{In}_x\text{Ga}_{1-x}\text{As}$ layers embedded in a GaAs matrix is analyzed in this work. The spatial resolution of the technique is improved by using thin electron-transparent specimens instead of bulk samples. Although the BSEs are detected in a comparably small angular range by an annular semiconductor detector, the image intensity can be evaluated to determine the composition and local thickness of the specimen. The measured intensities are calibrated within one single image to eliminate the influence of the detection and amplification system. Quantification is performed by comparison of experimental and calculated data. Instead of using time-consuming Monte-Carlo simulations, an analytical model is applied for BSE-intensity calculations which considers single electron scattering and electron diffusion.

1. Introduction

Quantification of chemical composition with high spatial resolution is often required in solid state physics and materials science. Electron microscopic techniques are well suited for this task because chemical analyses can be performed with a spatial resolution up to the atomic scale. A distinction can be made between analytical techniques for composition analysis, like energy-dispersive X-ray spectroscopy and electron energy loss spectroscopy, and techniques which rely on the exploitation of the image intensity itself. The latter necessitates a thorough understanding of the image formation process and does not need additional instrumental attachments to the electron microscope. However, the analysis of the image intensity requires pre-information on the materials system to be studied. Ternary and quaternary semiconductor systems are prototype materials in this regard. They are also interesting objects because the properties of semiconductor nanostructures are strongly dependent on the composition at the nanoscale and a thorough understanding of their properties can be only achieved if the composition on the nanoscale is known. Several techniques for quantitative composition analysis have been developed that exploit images obtained by (scanning) transmission electron microscopy (S)TEM [1–6] in a transmission electron microscope. However, little effort has been devoted up to now regarding composition determination on the basis of scanning electron microscopy (SEM) images. SEM-based techniques are interesting because scanning elec-

tron microscopes are widely available, and knock-on damage can be largely excluded at SEM-typical primary electron energies $E_0 \leq 30$ keV. Moreover, instrumental parameters, like E_0 or the detection angle range, can be easily changed and adapted to optimize the chemical sensitivity. This flexibility also enhances the reliability of the technique because measurements can be performed at different E_0 -values as a consistency check. SEM-based techniques may have been neglected up to now because the achievable spatial resolution is not yet competitive with (S)TEM-based approaches at higher electron energy. However, the resolution of scanning electron microscopes has improved substantially in the recent past and atomic-scale resolution was already achieved for thin samples in the bright-field STEM mode [7]. Along this line, the chemical sensitivity of annular dark-field STEM in a scanning electron microscope has already been demonstrated to be a sensitive technique for composition quantification of semiconductor heterostructures [8,9]. Alternatively, backscattered electron (BSE) SEM images have been exploited in the past to determine the average atomic number of materials by comparison with the BSE intensity of reference materials with known properties (e.g. [10–12]). The quantification is based on the composition sensitivity of the backscattering coefficient η , which is defined as the number of backscattered electrons per primary electron. For BSE SEM images, the spatial resolution is limited by the size of the interaction volume of the primary electrons which prevents the analysis of nanoscaled structures.

This limitation is overcome in our present work where BSE images

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of thin specimens are analyzed. As an example we determine the In-concentration x in an $\text{In}_x\text{Ga}_{1-x}\text{As}/\text{GaAs}$ -heterostructure which contains $\text{In}_x\text{Ga}_{1-x}\text{As}$ -layers with only 25 nm thickness. The analysis of thin specimens results in a thickness-dependent backscattering coefficient $\eta(t)$ which was measured by analysing the BSE intensity in a region with known composition (here GaAs) from a wedge-shaped sample with a well-defined thickness profile. Our technique does not rely on measurements from reference samples. Instead, we use pre-information on the material system to evaluate the composition. The dependence of the image intensity on instrumental parameters is significantly reduced by normalizing the BSE image intensities of InGaAs with respect to the GaAs BSE intensity obtained within one single image. The In-concentration is determined by comparing experimental and simulated BSE intensity ratios. We use a diffusion model for BSE intensity calculations which substantially reduces computation times for the calculation of $\eta(t)$ compared to Monte-Carlo (MC) simulations. Measurements are performed for different E_0 to verify the results.

2. Experimental

An $\text{In}_x\text{Ga}_{1-x}\text{As}/\text{GaAs}$ -heterostructure grown by molecular beam epitaxy on a GaAs-substrate was studied in this work. The sample contains four $\text{In}_x\text{Ga}_{1-x}\text{As}$ layers of 25 nm thickness with In-concentrations of $x=0.1, 0.2, 0.3$ and 0.4 which are embedded in GaAs barrier layers with 35 nm thickness. Details on the growth and verification of the composition of the analyzed sample by alternative techniques are outlined by Volkenandt et al. [9]. A cross-section specimen perpendicular to the InGaAs/GaAs layer system was prepared by focused-ion-beam (FIB) milling. A Pt-protection layer was deposited before FIB-milling to avoid damage to the sample. The final polishing was performed with a reduced Ga^+ -ion energy of 5 keV to minimize Ga-implantation. It is expected that Ga^+ -ion implantation cannot be completely avoided but a noticeable increase of the Ga-concentration is only expected for the thinnest specimen regions. A wedge-shaped thickness profile with a wedge angle of about 30° was milled to obtain a specimen with well-defined thickness profile. This specimen geometry is well suited to study the thickness dependence of the BSE intensity.

Fig. 1 shows a secondary-electron SEM image of a wedge sample. Deviations from the intended wedge angle can occur and the edge of the wedge is not infinitely sharp which leads to a thickness offset. Therefore, the local specimen thickness along the wedge must be determined by comparison of calculated and experimental BSE intensities as outlined in Section 3.

The material parameters used in the calculations in the work were obtained according to the additive rule for compound samples [13].

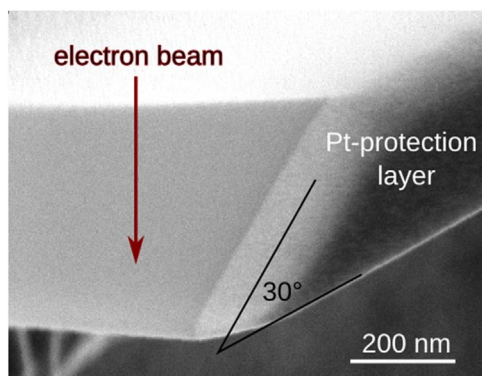


Fig. 1. 5 keV secondary-electron SEM image of a wedge-shaped specimen with a 30° wedge angle prepared by FIB-milling perpendicular to the InGaAs/GaAs layer system. The InGaAs-layers are not visible in the secondary-electron image. The red arrow indicates the incidence direction of the primary electron beam if a cross-section image of the InGaAs/GaAs-heterostructure is taken. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

The composition-dependent material density is determined by linear interpolation between $\rho=5.3 \text{ g/cm}^3$ for GaAs and $\rho=5.7 \text{ g/cm}^3$ for InAs. Further material parameters for the simulations comprise the relative atomic weight $A=69.72$ for Ga, $A=114.82$ for In and $A=74.92$ for As and the atomic numbers for Ga, In and As in the respective atom percentages.

SEM was performed in the non-immersion mode with a FEI Quanta 650 ESEM with a Schottky field emission gun. A commercial annular semiconductor BSE detector mounted below the objective lens was used. The detector geometry was measured by unmounting the detector from the pole piece. Backscattered electrons are collected in a hollow cone between 2.3 and 2.77 rad which corresponds to a working distance of 10 mm. In the following, the backscattering coefficient $\eta(t)$ will always be related to the limited collection angle range as opposed to literature where the BSE coefficient is related to electron backscattering in the full half space. Experiments show that electrons with energies below 2 keV do not lead to a measured BSE intensity in our BSE detector. This indicates that the threshold energy E_{thr} of our detector corresponds to 2 keV. Due to the high value of E_{thr} , the contribution of secondary electrons to the image intensity can be neglected.

Contrast and brightness were adjusted to make full use of the 16 bit grayscale resolution and to avoid over- and undersaturation of the image. The brightness offset can be determined from the vacuum region of the images and must be subtracted for the calculation of intensity ratios.

The energy-dependent efficiency of the detection system must be taken into account if calculated and experimental BSE intensities are compared. The response of a semiconductor detector in terms of the collection current I_{cc} as a function of the incident current I_0 is given according to Reimer [14] by

$$I_{cc} = I_0(1 - \eta_{Si}) \frac{E - E_{thr}}{E_{hp}} \varepsilon, \quad (1)$$

where E denotes the energy of the backscattered electrons, ε the charge collection efficiency, E_{thr} the threshold energy of the detector (2 keV in our case), η_{Si} the backscattering coefficient of the detector material and E_{hp} the generation energy for an electron-hole pair. Therefore, the calculated backscattering coefficients $\eta=n/n_0$ (number of backscattered electrons n , the number of primary electrons n_0) must be multiplied by a correction factor c_d given by Eq. (2)

$$c_d = \frac{E - E_{thr}}{E_0 - E_{thr}}. \quad (2)$$

The energy of backscattered electrons $E=E_0-E_{loss}$ is determined by the energy loss E_{loss} of the electrons in the sample. Details on the calculation of E_{loss} will be given in the following section.

Further properties of the detection system (like e.g. the amplifier characteristics) do not have to be considered. Moreover, measurements have shown that contrast transfer of the amplification system in the required intensity range is in a good approximation linear and cancels out by considering intensity ratios for the thickness and composition evaluation.

In crystalline materials dynamical electron diffraction can lead to channelling contrast and corresponding changes of the BSE intensity, which must be avoided for composition quantification. Channelling typically occurs if the incident electron beam is oriented parallel to a zone-axis orientation or if two-beam Bragg conditions prevail. Channelling can be recognized by tilting the sample and taking additional BSE images. The specimen orientation should be chosen at sample tilt positions where the BSE intensity does not change over a larger tilt angle range.

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