Quantitative Transmission Measurements in a Scanning Electron Microscope

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Scanning transmission electron microscopy (STEM) is known to show material-sensitive (Z-) contrast which increases if the electron energy is lowered. We will show in this contribution that low-energy STEM is a promising technique for quantitative analysis of the sample thickness and even composition if a reference region with known composition is available. Since knock-on damage is reduced at low beam energies, examination of radiation-sensitive samples like group-III nitride semiconductors or especially biological materials is facilitated. Standard scanning electron microscopes equipped with a STEM detector can be used without the need for an elaborate transmission electron microscope. STEM in a scanning electron microscope allows to change easily instrumental parameters like electron energy, collection angle or alignments to optimize the imaging parameters. For thin samples, a high lateral resolution of about 1-2 nm can be achieved with an adequate signal-to-noise ratio. Despite these advantages of low kV STEM only few reports are found in literature, that are concerned with quantitative low-energy STEM [1].

The quantification method [2] presented in the following is based on the comparison of measured intensities of high-angle annular dark-field (HAADF) images with Monte-Carlo (MC) simulations. The electron transmission is measured with a segmented annular semiconductor detector in a combined focused-ion-beam(FIB)/SEM FEI Strata400S microscope. Only electrons transmitted in a hollow cone between 0.2 and 0.7 mrad are recorded. Our test sample for the verification of the technique consists of four $In_xGa_{1-x}As$ quantum wells (QWs) separated by GaAs layers which was grown by molecular beam epitaxy (MBE) on a GaAs(001) substrate. The In-concentrations x are 10, 20, 30 and 40 %. Samples with defined geometry were prepared by FIB-based techniques. Lamellas suitable for STEM were produced with homogeneous and wedge-shaped thickness profiles. The latter allows the examination of a range of sample thicknesses within one sample. The NISTMonte [3] program package is used for the MC simulations. Simulated data has to be carefully post-processed to account for influences induced by the detector. Electron transmission or transmission ratios on the basis of MC simulations can be extracted and directly compared to measured image intensities.

Fig. 1a shows a HAADF STEM cross-section image of a wedge-shaped sample taken at 25 keV.The four $In_xG_{1-x}As$ QWs show brighter contrast under these imaging conditions. The intensity along the linescan along one of the GaAs layers (indicated by an arrow labeled 1 in Fig.1a) shows a characteristic maximum and shape. The measured intensity can be well fitted by the simulated electron transmission (Fig. 1b) thus revealing precise values for the wedge angle (here 19.6 degrees) and the minimum thickness (here 51 nm) at the edge of the wedge sample. Fig. 2a shows the intensity along the linescan 2 in Fig.1a perpendicular to the QW structure at a constant sample thickness of 80 nm. The intensity increases with the In-concentration. Note that the difference is only 4 to 10 gray levels which is sufficient for quantification of the In-concentration. Intensity ratios can be calculated from the averaged intensity in the In-containing layers with respect to the adjacent GaAs layer. These values can be directly compared with transmission ratios derived from the MC simulations. Fig. 2b shows results from the above mentioned sample together with those from samples with higher In-concentrations. The curves show good agreement and reproduce the composition of the layers [4].

References

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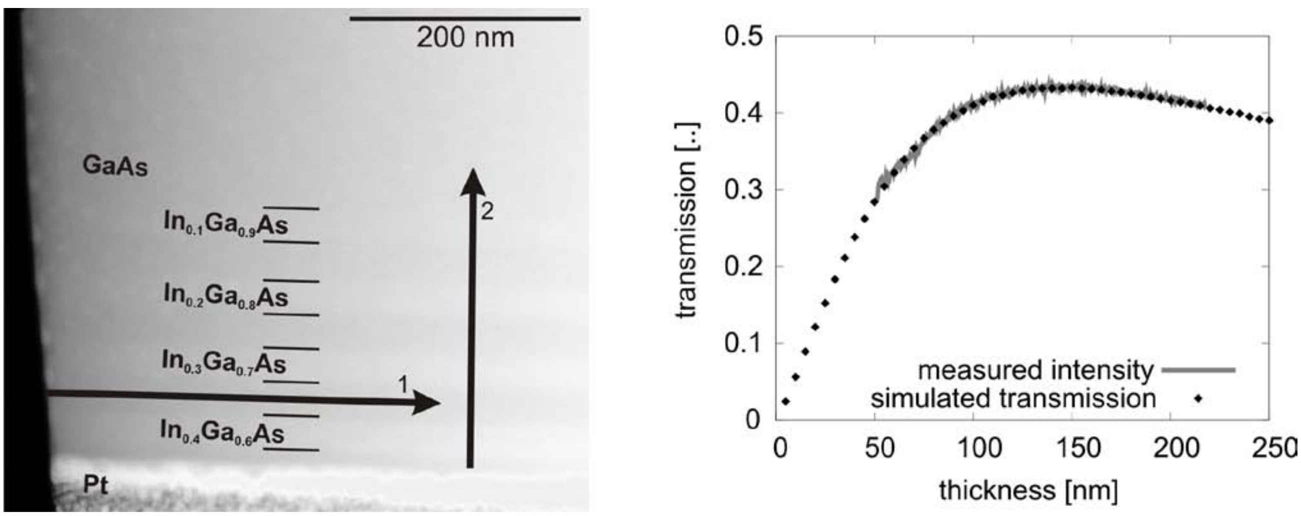

Fig. 1. a) HAADF STEM cross-section image of wedge-shaped sample at 25 keV. b) Comparison of the measured intensity along the GaAs layer with simulated electron transmission as a function the sample thickness.

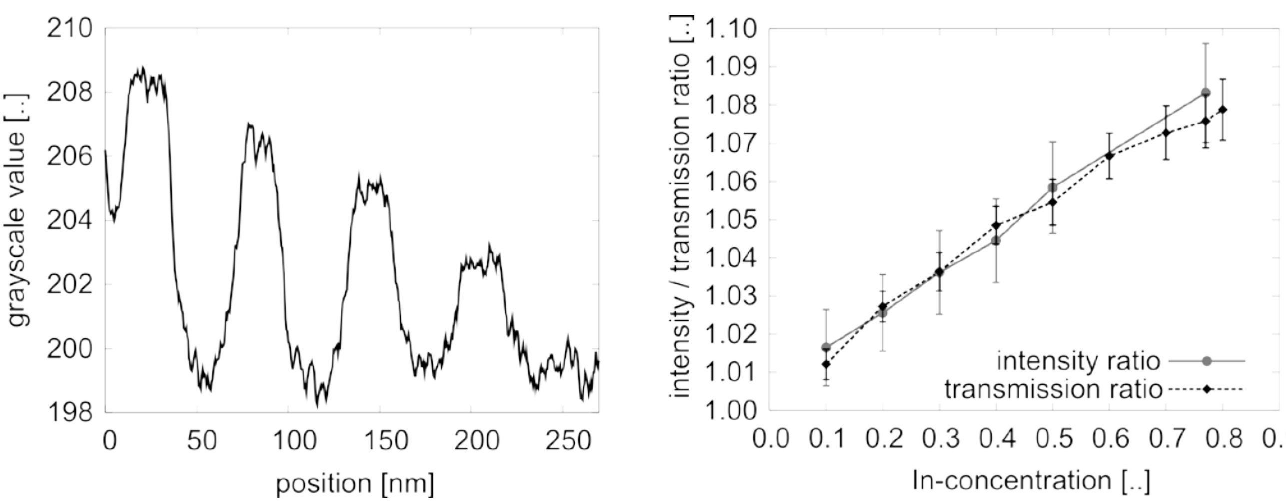

Fig. 2. a) Intensity linescan across the $In_xG_{1-x}As$/GaAs QW structure. b) Comparison of measured intensity ratios with simulated transmission ratios as a function of the In- concentration.