Quantification of the In concentration of InGaAs quantum wells by transmission measurements in a scanning electron microscope

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Scanning transmission electron microscopy (STEM) with a high-angle annular dark-field (HAADF) detector is well suited to obtain composition-sensitive (Z-)contrast but only few examples are found in the literature for the quantification of composition by HAADF STEM [1] which are confined to transmission electron microscopes (TEM) operating between 100 and 300 keV. However, composition determination of materials which are sensitive towards knock-on damage require measurements at low energies. In this work a standard scanning electron microscope (SEM) equipped with a semiconductor STEM detector is used to evaluate the composition of InGaAs/GaAs heterostructures.

The STEM detector in the FEI Strata400S microscope is positioned below and close to the sample holder. It is divided in several ring-like segments with the possibility to select bright-field (BF) and (HA)ADF sectors. HAADF images originating from electrons transmitted in a hollow cone between 0.2-0.7 rad show Z-contrast. The contrast is insensitive to dynamical scattering effects because the mean free path of the electrons with energies below 30 keV are much smaller compared to those at high energy electrons in a TEM [2,3]. Stochastical and plural scattering processes as well as a large number of material parameters (density, thickness, atomic number) require the comparison of the measurements with simulations. The quantification of measured intensities is performed by Monte Carlo (MC) simulations. The NISTMonte package is used, applying elastic Mott scattering cross sections and the continuous energy loss approximation [4].

To verify the validity of the simulations a well defined wedge-shaped sample was prepared by means of the focused ion beam (FIB) technique. The wedge angle and hence the local thickness are determined by top-view imaging of the sample with secondary electrons (SE). The investigated sample is a semiconductor layer system with four $In_xGa_{1-x}As$ layers of 25 nm thickness between GaAs layers with a thickness of 35 nm. The In concentrations x = 10 %, 20 %, 30 % and 40 % are well known from careful calibrations during molecular beam epitaxy growth and are confirmed by composition evaluation by lattice fringe analysis (CELFA) [5] and EDX measurements.

Intensity line scans perpendicular to the edge of the sample in the HAADF STEM image (Fig.1a) show a characteristic shape and maximum. Considering appropriate scaling factors and a thickness offset of the wedge, the measured intensity in the $In_{0.4}Ga_{0.6}As$ can be well fitted by MC simulations as shown in Fig.1b. Therefore, simulations can be used for precise thickness measurements, avoiding perturbing edge effects in SE images. Convolution with detector characteristics must be taken into account while comparing with simulations.

The intensity of the InGaAs layers is always normalized with respect to the intensity of the adjacent GaAs layer. The transmission reveals the optimal thickness at which intensity

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ratios should be analyzed to yield a good signal-to-noise ratio and to minimize the influence of local thickness variations.

Measured and simulated normalized intensities for layers of different In concentrations are shown in Fig.2. Although the signal variation is quite low the two curves show good agreement and reproduce the relative composition of the layers.

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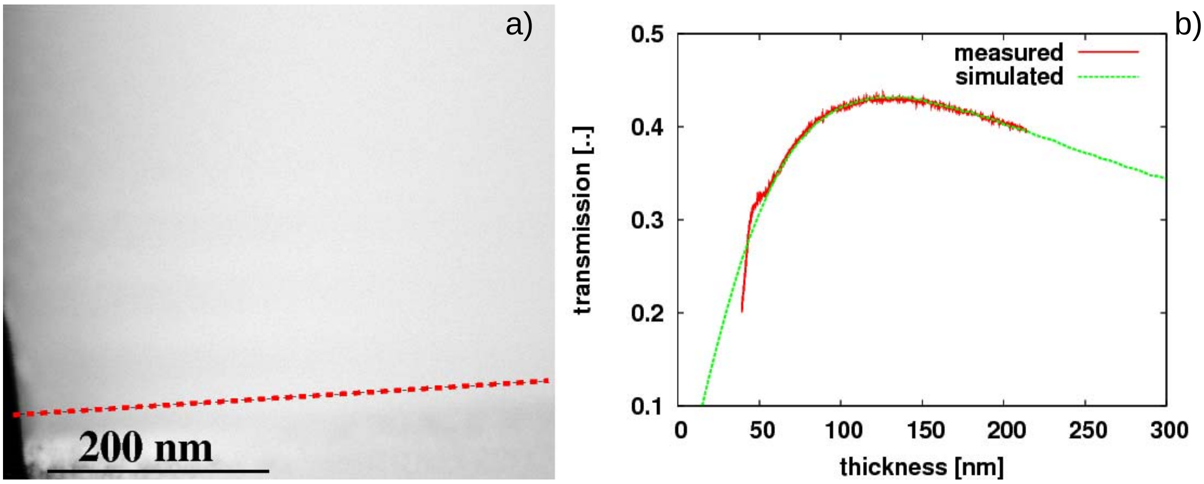

Figure 1. (a) HAADF STEM image of a 30° wedge at 25 keV with line scan (dashed line) perpendicular to the edge along the $In_{0.4}Ga_{0.6}As$ layer. (b) Comparison of the measured normalized intensity with MC simulations as a function of the sample thickness.

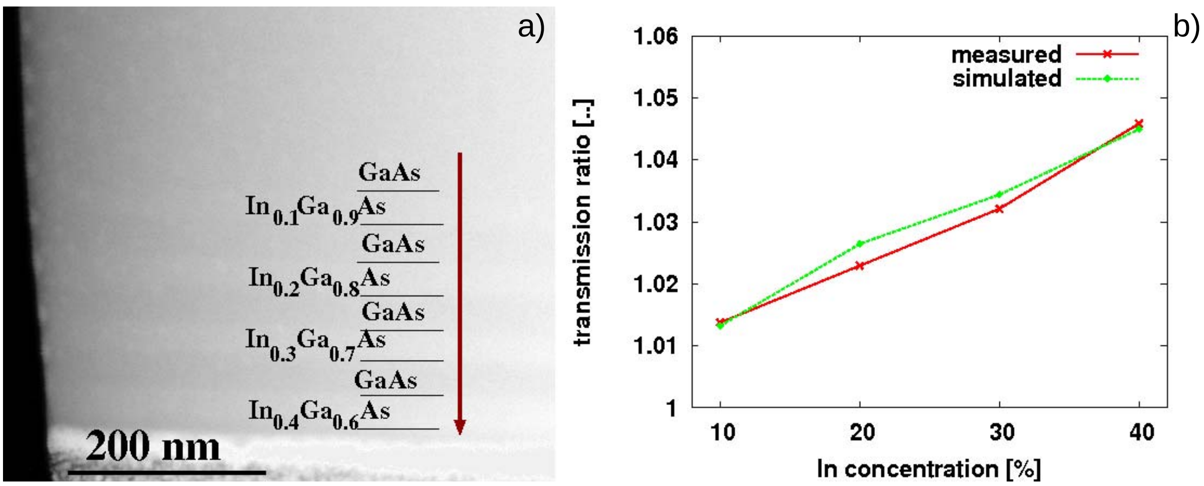

Figure 2. (a) HAADF STEM image of a 10° wedge sample at 25 keV with linescan perpendicular to the $In_xGa_{1-x}As$ layers. (b) Comparison of measured normalized intensities with MC simulations as a function of the In concentration.

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