Quantitative Composition Evaluation from HAADF-STEM in GeSi/Si Heterostructures

M Tewes[1], F F Krause[1,3], K Müller[1], P Potapov[2], M Schowalter[1], T Mehrtens[1] and A Rosenauer[1]

[1] Institut für Festkörperphysik, Universität Bremen, Otto-Hahn-Allee 1, 28359 Bremen, Germany
[2] GLOBALFOUNDRIES, Wilschdorfer Landstr. 101, 01109 Dresden, Germany

f.krause@ifp.uni-bremen.de

Abstract. High-angle annular dark field scanning transmission electron microscopy has been successfully used for composition evaluation in various material systems. In this work, the quantitative applicability of this method to GeSi/Si heterostructures was studied. Reference images were simulated by frozen lattice multislice simulations for different Ge concentrations accounting for static atomic displacements and biaxial strain due to pseudomorphic growth. Specimen thickness and composition are obtained by comparison of simulated and normalised experimental intensities. The measured thickness of a pure Si wedge specimen is compared to thickness determined from Pendellösung fringes in dark field micrographs. The deviation is below 10 nm coinciding with the accuracy of prior works. The composition of a GeSi-layer structure was measured in a calibration sample of known concentration and good agreement is found. Two-dimensional concentration maps of a GeSi/Si transistor structure were created. Measured concentrations agree with nominal values. However, strain fields in the Si lead to a variation of the image intensity causing an artificial fluctuation of the measured concentrations of ±4%.

1. Introduction

Germanium-Silicon (GeSi) heterostructures are of technological importance for modern semiconductor devices, e.g. metal-oxide semiconductor field effect transistors (MOSFETs) [1] that are widely used in modern electronics. Hence, the measurement of the Ge concentration is of high interest in those systems. The evaluation of the chemically sensitive high-angle annular dark field (HAADF) signal of scanning transmission electron microscopy (STEM) has been successfully used for accurate chemical composition measurement at Ångström resolution within many other material systems such as InGaN, SiGaAs, AlGaN or GaNAs [2-5].

In this work the applicability of quantitative HAADF-STEM for composition evaluation was studied in GeSi/Si heterostructures [6] by investigation of several specimens of this material system.

2. Simulation of reference images

To get reference data for evaluation of the measured HAADF-intensities, STEM-images were simulated by frozen lattice multislice simulations conducted with the STEMsim Software [7].

[3] corresponding author

A supercell laterally consisting of 7×7 orthorhombic unit cells in [110] zone axis was used with a numerical grid of 2100×1500 pixels.

The mean square thermal displacements used in these simulations to account for thermal diffuse scattering (TDS) were derived from density functional theory calculations as described in reference [8]. Static atomic displacements caused by the different covalent radii of Ge and Si, which have influence on the diffuse signal [9], were computed with an empirical potential suggested by Tersoff [10]. With this potential, the supercells for the simulations were relaxed by energy minimization with the LAMMPS software [11]. Furthermore, to account for the biaxial strain due to pseudomorphic growth of GeSi layers on bulk Si, the supercells were strained according to the results of elasticity theory calculations. Thus, various effects were taken into account whereas prior works assumed a Rutherford scattering model [6].

These simulations were executed for various specimen thicknesses and Ge concentrations, where for each concentration twenty individual supercell configurations with statistically distributed atoms and thermal displacements were used. The non-uniform sensitivity of the HAADF-detector, which was used in the experiments, was also considered. Thus, a dataset of HAADF-intensities was obtained as a function of specimen thickness and Ge concentration and was used as a reference in the evaluation of the experimental data.

3. Experimental procedure

The specimens used in this work were, except for the purchased calibration sample in section 4.2, prepared as lamellae with a focused ion beam (FIB) and treated with low energy Ar ion milling to remove amorphous surface layers [12]. The experimental investigation was performed with a FEI Titan 80/300 (S)TEM. To ensure comparability to the reference simulations, the measured HAADF-intensities are normalised with respect to the incident electron beam intensity, that were measured by scanning the focused beam directly over the detector as described in reference [13].

The specimen thickness is then determined from regions of known composition, in most cases of pure Si, by comparison with the reference. For regions with unknown concentration the thickness is polynomial inter- or extrapolated, respectively. Using the resulting thickness map and the measured intensities local compositions are determined for the entire STEM image [2].

4. Results

4.1. Thickness determination in a pure Si wedge sample

To validate the thickness measurement by HAADF-STEM with the simulated reference, a wedge specimen of pure Si was FIB-prepared in [110] zone axis and micrographs were acquired by HAADF-STEM and by dark-field conventional transmission electron microscopy (DF-CTEM) with the (004)-beam under imaging conditions that allowed the detection of Pendellösung thickness contrast fringes. From these fringes the specimen thickness was determined by comparison with Bloch-wave simulations conducted for the same sample orientation as used for the acquisition.

Figure 1 shows a comparison of the measured specimen thickness from both methods. The values from HAADF-STEM show a good agreement with those from DF-CTEM; the deviation is below 10 nm. This coincides with the accuracy of prior works on quantitative STEM comparing simulations and experiment, e.g. reference [5].

4.2. Concentration evaluation of a calibration sample

Furthermore, a purchased commercial calibration sample, MAG*I*CAL® from Norrox Scientific Ltd., containing multiple GeSi-layers with a known nominal Ge concentration of 19% embedded in Si and conventionally prepared in [110] zone axis was examined. The determined Ge concentration was (18±4)%, which is in good agreement with the nominal value. The evaluated composition of single layers from high resolution HAADF-STEM images coincided with the results of strain state analysis of high resolution CTEM micrographs which were evaluated for comparison.

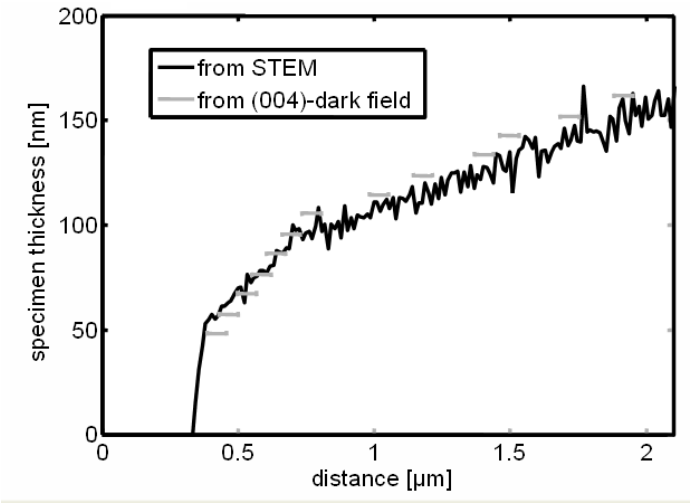

Figure 1. Comparison of specimen thickness of the pure Si wedge sample determined from HAADF-intensity and from Pendellösung thickness fringes. A good agreement is found; the deviation is below 10 nm.

4.3. Investigation of a GeSi/Si-MOSFET structure

In addition we investigated a Si-based p-MOSFET structure in which source and drain contain Ge to improve charge carrier mobility by straining the Si in the gate region [1]. The resulting two-dimensional composition map from the HAADF signal is depicted in figure 2. The mean Ge concentrations in the lower and upper regions are (22±3)% and (28±3)%, respectively. These are slightly higher than the nominal values of 19% and 27%, but fall within statistical error.

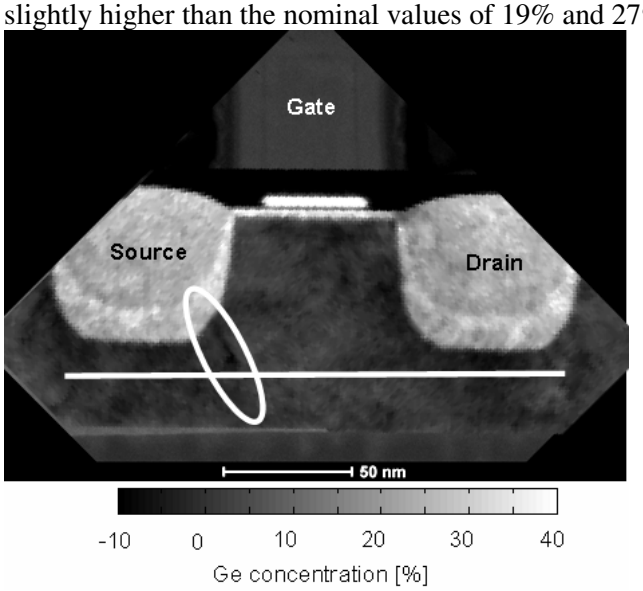

Figure 2. Concentration map of a GeSi/Si-MOSFET structure with two regions of different Ge concentrations in source and drain. The line marks the position of the linescan shown in figure 3. In the encircled area an artificial negative Ge concentration caused by a stacking fault is observed.

4.4. Influence of strain on measured concentrations and thicknesses

In the concentration map regions of artificial negative Ge concentrations also appear especially near GeSi/Si interfaces. This is partially caused by stacking faults as the one marked in figure 2, which occur in this highly strained structure. A linescan below the transistor in the pure Si region, as shown in figure 3, however, reveals artificial Ge concentration fluctuations of ±4% that cannot totally be caused by dislocations.

Another possible reason are effects of strain in the Si induced by the GeSi-regions: For the reference simulations described in section 2 pseudomorphic growth of the GeSi on bulk Si was assumed, which is legitimate for layer samples, but in this highly strained structure, the GeSi in source and drain could actually cause strain in the Si. This mismatch between simulations and experiment would cause a systematic error. To investigate its influence, simulations were conducted for strained Si supercells. The results showed a significant intensity difference to unstrained Si, that can lead to an

underestimation of the specimen thickness of up to 10 nm for 100 nm thick specimens or, if the thickness was estimated correctly, negative Ge concentrations in regions of pure Si of up to -2%. This systematic error is unavoidable as long as the strain state of both the investigated and the reference area is unknown. It partly explains the observed fluctuations. The remaining artifacts are most likely caused by surface strain effects as described in reference [2].

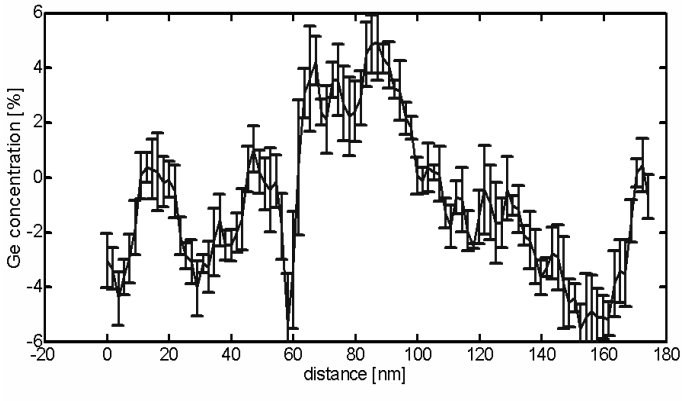

Figure 3. Linescan of 10 nm width from the concentration map of the MOSFET shown in figure 2 taken in the pure silicon area below the transistor (red line in figure 2). A HAADF-intensity corresponding to a Ge concentration in the range of ±4% is measured. Below source and drain artificial negative concentrations are obtained. This is caused by strain effects and, as in the dip at 60 nm, by dislocations.

5. Summary

The applicability of chemical composition evaluation with normalised HAADF-STEM intensity by comparison to multislice simulations in the GeSi/Si material system was investigated. The measurement of thickness in a pure Si wedge specimen and the concentration determination in a layer structure yielded results which are in good agreement with both reference measurements and nominal values.

For a GeSi/Si MOSFET structure two-dimensional composition maps were determined. The concentrations were also in good agreement with nominal values. Within these highly strained structures, however, artificial Ge concentration fluctuations were observed in pure silicon regions, which could partly be explained by strain effects. To avoid these, the simultaneous measurement of strain as suggested by reference [14] would be necessary.

References

[1] Auth M et al. 2008 *Intel Technology Journal* **12** 77-85
[2] Rosenauer A et al. 2011 *Ultramicroscopy* **111** 1316-1327
[3] Carlino E and Grillo V 2005 *Phys. Rev B* **71** 235303
[4] Rosenauer A, Gries K, Müller K, Pretorius A, Schowalter M, Avramescu A, Engl K and Lutgen S 2009 *Ultramicroscopy* **109** 1171-1182
[5] Grieb T, Müller K, Fritz R, Schowalter M, Neugebohrn N, Knaubb N, Volz K and Rosenauer A 2012 *Ultramicroscopy* **117** 15-23
[6] Walther T and Humphreys C J 1999 *Journal of Crystal Growth* **197** 113-128
[7] Rosenauer A and Schowalter M 2007 *Microscopy of Semiconducting Materials 2007* Cullis A G, and Midgley P A (Springer Netherlands) pp 170-172
[8] Schowalter M, Rosenauer A, Titantah J T and Lamoen D 2009 *Acta. Cryst. A* **65** 5-17
[9] Grillo V, Carlino E and Glas F 2008 *Phys. Rev. B* **77** 054103
[10] Tersoff J 1988 *Phys .Rev. B* **37** 6991-7000
[11] Plimton S 1995 *J. Comput. Phys.* **117** 1-19
[12] Mehrtens T, Bley S, Satyam P V and Rosenauer A 2012 *Micron* **43** 902-909
[13] Le Beau J M and Stemmer S 2008 *Ultramicroscopy* **108** 1653-1658l
[14] Müller K, Rosenauer A, Schowalter M, Zweck J, Fritz R and Volz K 2012 *Microsc. Microanal.* **18**, 995-1009