Transmission Electron Microscopy of Nanostructures

W. Schwinger, G. Hesser, H. Lichtenberger and F. Schäffler
Institute of Semiconductor Physics, University Linz,
Altenbergerstr. 69, 4020 Linz

Transmission electron microscopy (TEM) is a powerful tool for detailed analysis of both crystalline and amorphous structures ranging from the micro to the nanometer scale. TEM is capable to display not only the real but also the reciprocal space of a sample; i.e. the diffraction pattern. In this report we will show – using selected examples – the analytical capabilities of the JEOL FasTEM 2011 with CCD-camera and EDS X-ray detector like the element specific composition of a hetero-bipolar-transistor, the determination of the dislocation density at a Si-Ge interface, the investigation of facets on a prestructured and annealed Si surface, the analysis of size and shape of SiC precipitations and the alignment of self-arranged Ge dots.

Introduction

Transmission electron microscopy is a powerful tool for detailed analysis of both crystalline and amorphous structures ranging from the micro to the nanometer scale. The JEOL 2011 FasTEM can be used for the investigation of interfaces, grain boundaries, nanocrystals, and hetero- and monocrystalline structures as well as for polymeric and organic samples.

TEM is capable to display not only the real but also the reciprocal space of a sample; i.e. the diffraction pattern. The recently installed CCD-camera and the EDS microprobe for qualitative and quantitative element-specific X-ray analysis are an excellent upgrade. The combination of all three devices TEM, CCD, and EDS results in a faster and more comprehensive analysis of samples.

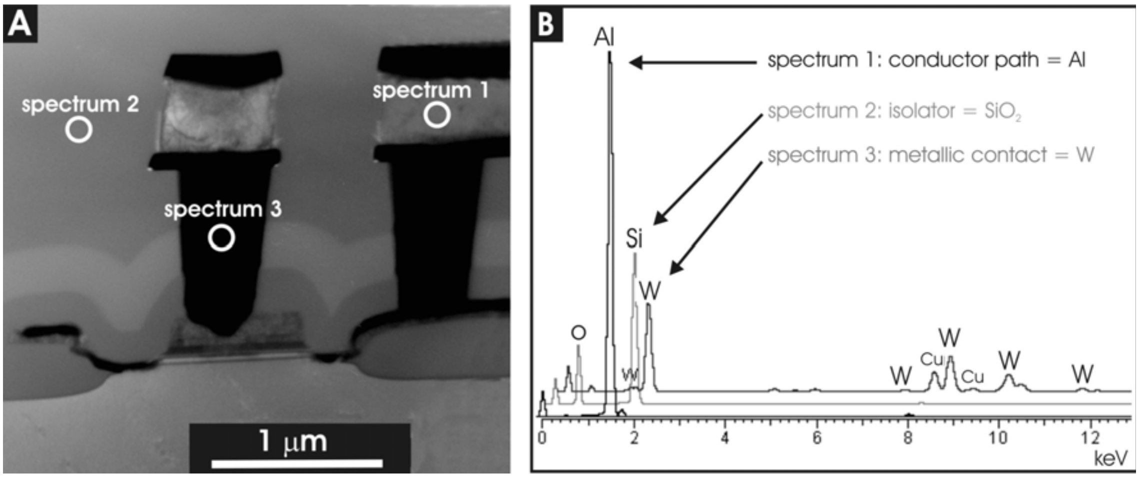

Fig. 1: (a) Cross section image of a transistor produced at austria**micro**systems at medium magnification (sample: 110X). (b) X-ray-spectra reveal the element-specific composition of the sample.

Experimental

Figure 1 (a) shows, at medium magnification, a cross section of a hetero-bipolar-transistor fabricated at austria**micro**systems. Using the EDS microprobe one can evaluate the local composition of the sample (Fig. 1 (b)). The conductor path is made of aluminum; the contacts consist mainly of tungsten and are isolated by SiO_2. The brightness contrast in the TEM-image is due to the different atomic numbers of the elements resulting in different scattering cross section.

When growing heterostructures or other layer sequences the quality of the interface between two layers is of particular interest. With TEM it is possible to investigate interfaces in detail and to determine whether the overgrowth is mono-crystalline, poly-crystalline, amorphous, or disordered. The formation of dislocations is also of particular interest. Tilting the sample increases the contrast of these dislocations. The tilt direction is related to the burgers-vector characterizing the dislocations. One can thus not only determine the density of dislocations at an interface but also the type of dislocations.

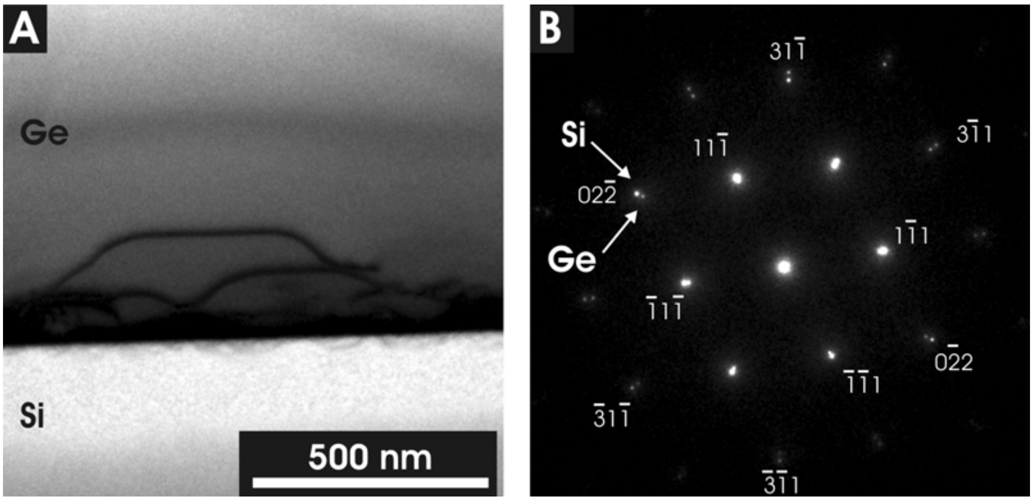

Fig. 2: (a) Dislocation at a Si-Ge interface (sample: 158X). Tilting the sample increases the dislocation contrast. (b) Diffraction image reveals that both the Si- and Ge-layer are mono-crystalline with the same crystal orientation. A split of the diffraction spots is due to the lattice mismatch of about 4% between Si and Ge having the larger lattice constant.

To get atomic resolution the sample has to be aligned along a main crystal axis. When tilting a sample atomic resolution is lost. Figure 2 (a) shows dislocations at an interface between pure Ge deposited on pure Si. Building a multi-image-collage one can thereby determine the defect density and defect depth at the Si-Ge-interface.

The diffraction images of that interface (Fig. 2 (b)) shows that both the Si- and the Ge-layer are mono-crystalline with the same crystal orientation. Since the lattice constant of Ge is about 4% larger than the one of Si, diffraction spots split, with the Ge-spot lying closer to the central (000)-spot (reciprocal space!). This can be especially seen at the higher order spots like the {022} and the {311}. The nonindexed spots result from rescattered spots and thus do not correspond to certain lattice planes and are therefore called forbidden spots.

Two main preparation techniques for solid TEM-sample are common: cross-sectional and plan-view showing either a view along the surface or perpendicular to the surface.

A cross-sectional view not only reveals the surface structures but also the layer sequence, which is not detectable with e.g. AFM.

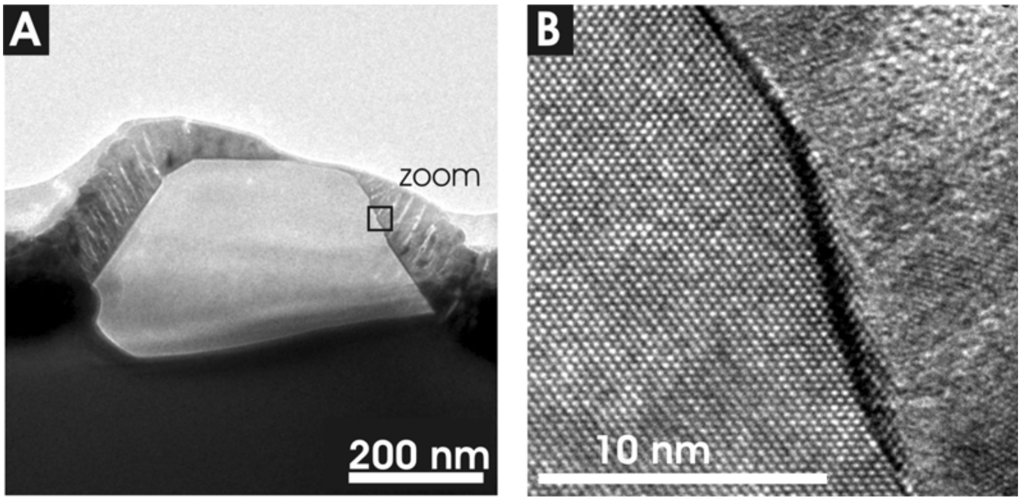

Fig. 3: (a) Cross-section of an originally rectangular Si-wire after annealing and covering with titanium (sample: 082X). Transient-enhanced Si diffusion leads to the formation of stable facets, which can be seen in high-resolution images like (b) with atomic resolution; [1] for more details.

Figure 3 (a) shows a cross-section through a capped wire structure. Originally rectangular Si-wires changed their shape during vacuum annealing and formed facets before covered with titanium. The high-resolution image Fig. 3 (b) shows not only these small facets in detail but also the structure of the deposited titanium. See [1] for more details.

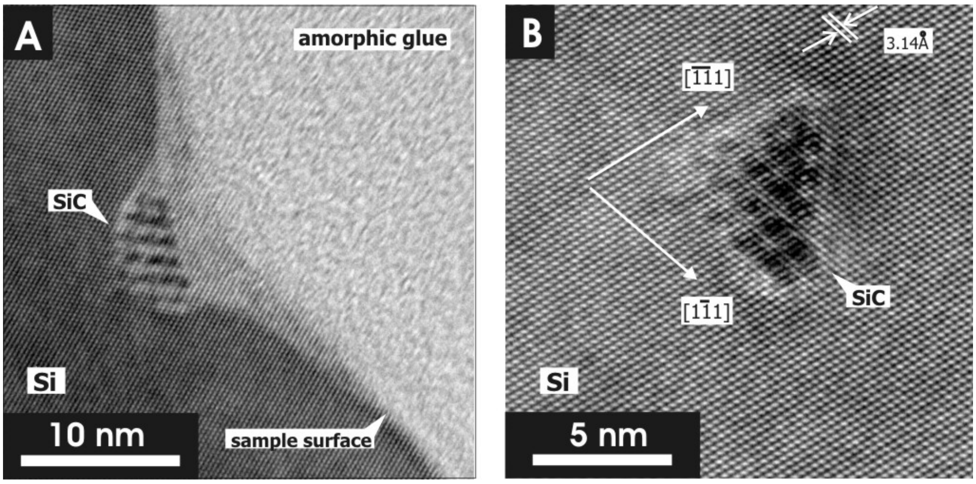

Fig.4: A: High-resolution image showing a cross-section of a SiC-precipitate, which disrupted further overgrowth (sample: 47X). B: High-resolution image showing a buried SiC precipitate (sample: 47X).

Annealing a submonolayer of Fullerene-molecules deposited on Si(100) and buried with Si leads to the formation of SiC precipitates. High resolution TEM-images reveal Moiré-fringes resulting from the overlap of crystalline Si and SiC as shown in Figs. 4 (a) and 4 (b). Knowing the lattice constant of Si and measuring the period of the Moiré-pattern one can calculate the lattice constant of SiC and thus can determine whether the precipitates are relaxed or strained. One can also see that large precipitates disrupt further overgrowth leading to a funnel-shaped surface (Fig. 5 (a)) whereas small precipitates are covered by crystalline Si (Fig. 4 (b)).

Figure 5 shows the arrangement of Ge-dot in 3layer-sequence. TEM images show the perfect self-arrangement of the dots. In Fig. 5 (a) in addition to the layers structure also the tension contrast can be seen. Figure 5 (b) displays mainly mass contrast between Si and Ge due to their different atomic mass.

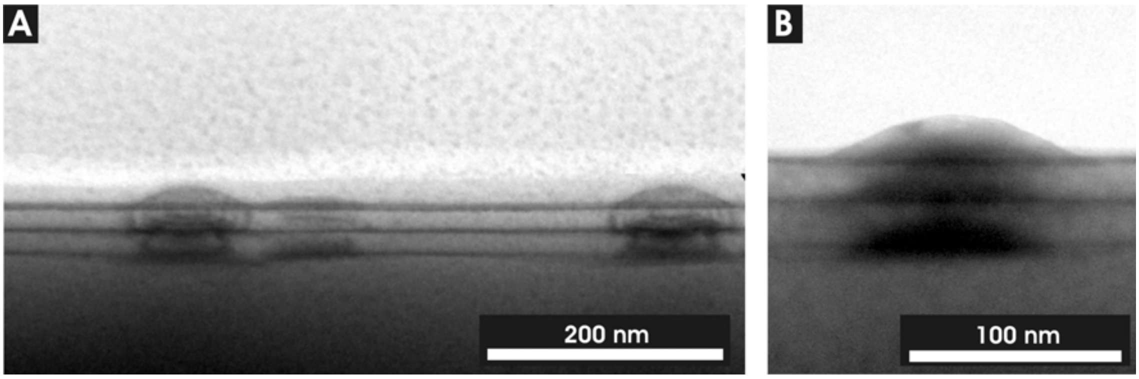

Fig. 5: (a) 3-layer-sequence of self-arranged Ge-dots on Si. Bows between the layers indicate tension; (b) Detail of (a) focused on mass-contrast; [2] for details (131X).

Conclusion

These examples show that TEM is a powerful tool for the detailed analysis with atomic resolution of crystalline mono- and hetero-structures revealing details of the sample composition since in addition to an image of the sample in real space also the reciprocal space of the sample, i.e. the diffraction pattern – which gives information of the crystal growth in more detail – can be displayed. The additional EDS X-ray detection system is a perfect upgrade allowing a qualitative and quantitative element specific analysis of the sample composition. The modern CCD-camera allows electronic image processing giving instant results.

References

[1] H. Lichtenberger, M. Mühlberger, “Transient-enhanced Si diffusion on native-oxide-covered Si(001) nanostructures during vacuum annealing”, Appl. Phys. Lett. **82**, 3650 (2003)

[2] Zhenyang Zhong, A. Halilovic, “Positioning of self-assembled Ge islands on stripe-patterned Si(001) substrates” , J. Appl. Phys. **93**, 6258 (2003)

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A. Bonanni, H. Sitter, Institut für Festkörperphysik, JKU Linz

fke - Institut für Festkörperelektronik, TU Wien

AMS - austriamicrosystems AG, A-8141 Schloss Premstätten

E+E - Elektronik Ges.m.b.H., A-4209 Engerwitzdorf