Type of presentation: Oral

ID-10-O-2545 Changes of Materials' Properties at the Surface of TEM-Samples Due to Low-kV Argon Ion Milling

Müller E.[1]

[1]Electron Microscopy Facility, Paul-Scherrer-Institute Villigen, Villigen/PSI, Switzerland

Email of the presenting author: elisabeth.mueller@psi.ch

Abstract

The availability of Cs-corrected transmission electron microscopes (TEM) has strongly increased the quality requirements for TEM samples. Especially, the amorphous layers at the sample surface have to be as thin as possible. If Ar-ion milling is applied for cross-section TEM sample preparation, finishing the ion-milling procedure with a voltage as low as a few hundred volts may account for this [1-6]. This study, however, reveals 3 additional effects of the low-voltage Ar-ion treatment.

For the present study, a Precision Ion Polishing System (PIPS, Gatan) equipped with low voltage option was used for ion milling (final milling at 0.5kV for 5 minutes; etching angles 3 - 5°). Three specimen types (pure silicon, III-V semiconductors and SrTiO3) were investigated in a Philips CM12 (100kV) and a FEI Tecnai F30ST (300kV) TEM as well as a Hitachi HD-2700Cs (200kV) dedicated STEM.

The fine polishing at low acceleration voltage resulted in a clear reduction of the thickness of the amorphous surface layers (Fig. 1). This was, however, not the only effect observed: all three sample types reproducibly showed severe charging in all three microscopes. This effect was – especially in the dedicated STEM – present with and without subsequent plasma-cleaning for reduction of contamination. The low-voltage milling furthermore caused some re-deposition of Cu-particles onto the sample from the Cu-tube into which the material had been embedded. The Cu was observed at material boundaries and at the sample edges. It is assumed that this was due to electrostatic fields built up by charging effects already during the milling process (Fig. 2). Last but not least, another important property of the material – its oxidation behaviour – appeared to have changed drastically: While in a TEM sample the AlAs layers of an AlAs/GaAs superlattice delaminated after few hours due to oxidation, if the sample was milled at 3.5kV, the oxidation was almost fully suppressed for several weeks after low-kV milling (Fig. 3). Only along dislocations oxidation was still observed to occur.

References

[1] T. Schuhrke, M. Mandl, J. Zweck, H. Hoffmann, Ultramicroscopy 41 (1992), p. 429

[2] A. Barna, B. Pécz, M. Menyhard, Ultramicroscopy 70 (1998), p. 161

[3] A. Barna, B. Pécz, M. Menyhard, Micron 30 (1999), p. 267

[4] J.P. McCaffrey, M.W. Phaneuf, L.D. Madsen, Ultramicroscopy 87 (2001), p. 97

[5] J. Mayer, L.A. Giannuzzi, T. Kamino, J. Michael, Mrs Bulletin 32 (2007), p. 400

[6] M.J. Süess, E. Mueller and R. Wepf, Ultramicroscopy 111 (2011), p. 1224.

Acknowledgement: The support with sample preparation by Eszter Barthazy and the possibility to use the infrastructure of the Microscopy Centre at ETH Zürich, ScopeM, are gratefully acknowledged.

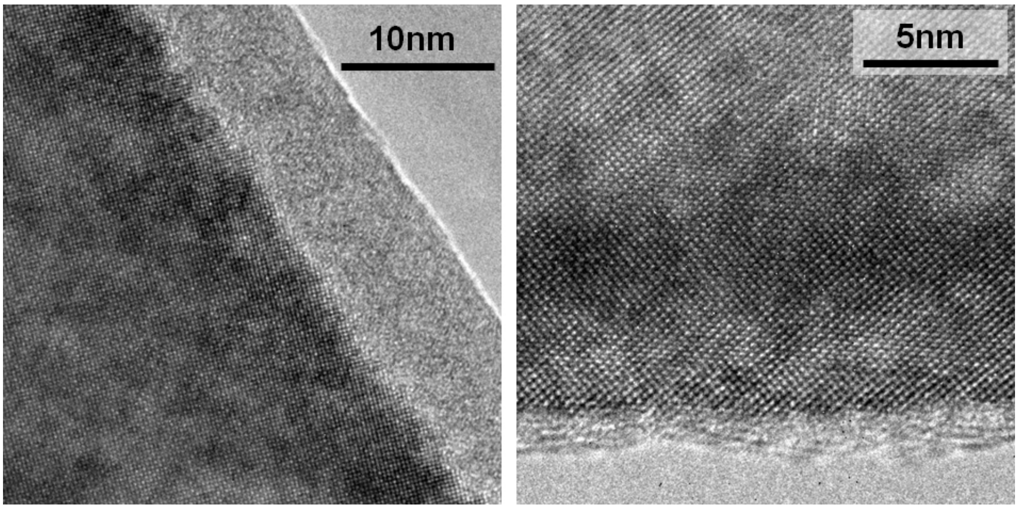

Fig. 1: HRTEM images of SrTiO3 show an amorphous layer at the sample edge of about 8nm after ion etching at 4kV (left). The amorphisation is reduced to below 2nm after a low-voltage etch of 0.5kV (right).

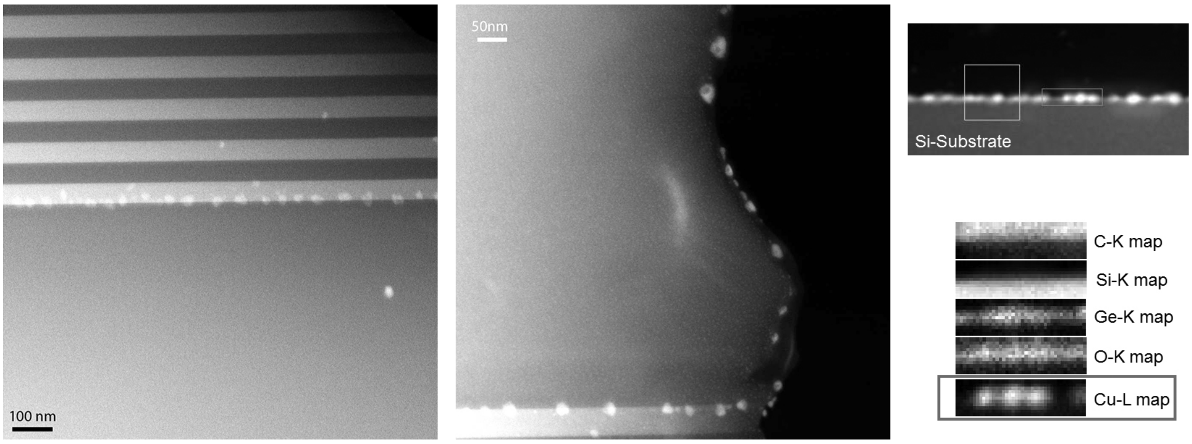

Fig. 2: HAADF STEM images of a SiGe/Ge superlattice on Si(001). Small particles are decorating the interface between the superlattice and the substrate as well as the edge of the sample (left and central image). EDX maps of the area shown in the HAADF STEM image at top right identified the particles to consist of Cu (right).

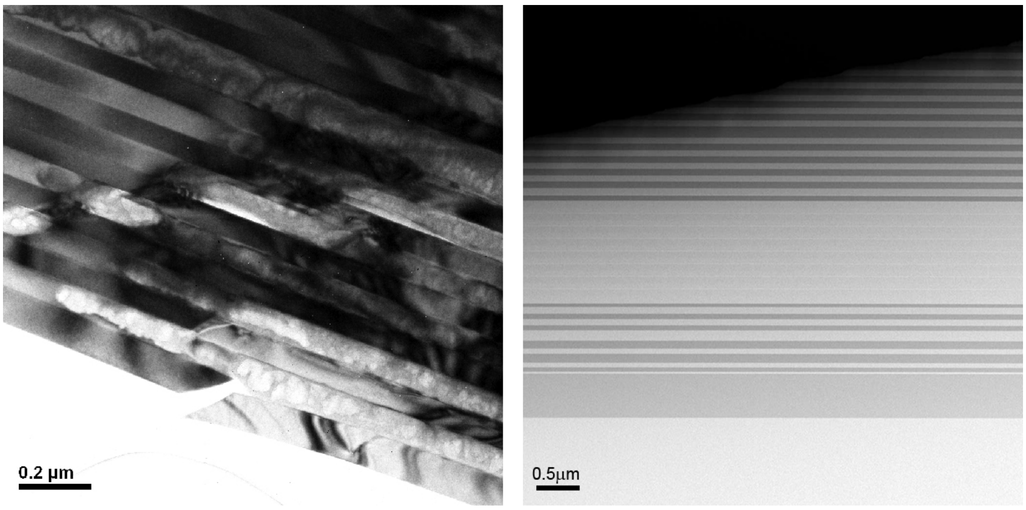

Fig. 3: GaAs/AlAs superlattice with final Ar-ion etching step at 3.5kV (left, TEM) and 0.5kV (right, HAADF STEM), respectively. While oxidation of the AlAs layers had caused a severe degradation of the sample within less than 1 day after etching at 3.5kV, the layers were still perfectly preserved 3 weeks after low-voltage milling.