Optimizing Ar^+-ion etching for TEM cross-section sample preparation

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Samples for transmission electron microscopy (TEM) with thin (< 50-100 nm) plane-parallel regions of interest are advantageous for most TEM techniques and in particular for composition analyses by energy-dispersive x-ray spectroscopy and electron energy loss spectroscopy. However, it is well known that thin films on mono- or polycrystalline substrates with different sputtering yields are challenging to prepare with a homogeneous and broad thinned region of interest. Ar^+-ion etching as the final step of preparation turned out as one of the most important steps. The common preparation procedure including sandwich gluing, grinding, polishing and Ar^+-ion etching [1] often fails, if the unsuitable parameters for ion milling are selected. Depending on ion energy, geometrical (sputtering angle) and devicespecific parameters (sector selection), a large variety of inappropriately etched TEM samples can be obtained. Much time is wasted in the preparation of samples with large wedge angles or even cross-section samples, where the film of interest is preferentially etched and disappears during ion milling [2]. Therefore, an improved procedure for Ar^+-ion etching by means of a PIPS (Gatan) is presented, with which high-quality cross-section samples can be prepared reliably and reproducibly.

The main difference between single-sector and double-sector selection for etching samples is illustrated in Fig. 1. Fig.1a shows a TEM bright-field image of an epitaxially grown $EuTiO_3$ (ETO) thin film on single-crystalline $SrTiO_3$ (STO) obtained in (002) two-beam condition (extinction distance $\xi_{(002)}$ = 52 nm). This sample was thinned by selecting the double-sector mode with the incident ion beam perpendicular to the bonded surfaces (incident beam angle of 4° and 4 keV ion energy), resulting in a wedge-shaped sample profile which is indicated by the thickness contours parallel to the sample edge (wedge angle of 12°). To enlarge the electron transparent regions, the sputtering procedure was optimized. Fig. 1b shows a cross-section TEM sample of the same material (ETO/STO), mechanically prethinned by the same procedure but etched by selecting the single-sector mode. It possesses an area with a width of several ten µm and a depth of about 800 nm from the surface. The thickness is rather homogeneous in the marked region with a thickness change of less than one extinction distance.

To understand the difference in the behavior, the obtained topography was evaluated by means of qualitative Monte-Carlo sputtering simulations which are based on the angular dependency of the sputtering yield described by Eckstein and Preuss [3]. Fig. 2 shows the simulated topography of cross-section samples achieved by double- and single-sector ion bombardment, respectively. The comparatively large angle of the wedge-shaped samples prepared by double-sector etching can be reproduced by the simulations as can be recognized in Fig. 2a. The angular dependency of the sputter yield [3,4] in combination with the singlesector mode on the other hand induces a topographical step on the surfaces on the side of the sandwich opposite to the activated ion gun as visualized in Fig. 2b. With increasing

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sputtering time, this step is driven away from the bonding interface, leaving an area with nearly flat topography behind. When the step reaches a distance of about 10 µm from the glue layer the sample is rotated by 180° to apply the Ar^+-ion bombardment to the opposite side (Fig. 2b) which leads to a large thin region on one side of the sample sandwich (see Fig. 2b). The thickness is larger on the other side of the glue layer, but it may still be well electron transparent. One advantage of this technique is that the thin films on the faces of the embedded pieces are always protected by the shadow of the side that is facing the activated ion guns (see Fig. 2b).

The proposed procedure allows the preparation of high-quality TEM samples with large electron-transparent region with homogeneous thickness by common preparation techniques. This is possible for materials with inhomogeneous sputtering behavior which usually requires preparation by the focused-ion-beam technique. For the latter technique, low-kV ion etching is often necessary to remove amorphized surface layers due to the Ga-ion bombardment.

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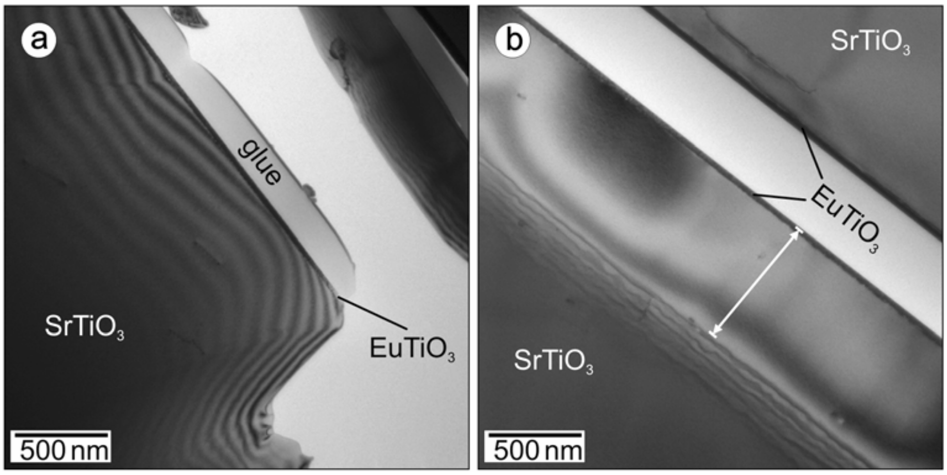

Figure 1. TEM cross-section brightfield images of epitaxially grown $EuTiO_3$ on $SrTiO_3$ in (002) two-beam conditions: a) double-sector etching, b) single-sector etching.

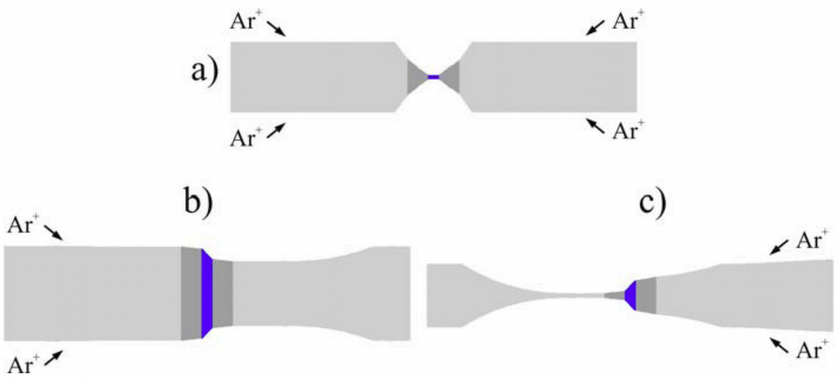

Figure 2. Monte-Carlo sputtering simulations of the topography by a) double-sector etching b) single-sector etching (first side), c) after rotating the sample by 180°, (height rescaled for better visualization). The dark blue region corresponds to the glue layer.

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