TEM lamella preparation with in situ low energy Ar-milling

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Lamellae for transmission electron microscopy (TEM) can effectively be prepared with focused ion beam instruments (FIB). Using low voltage Ga-ion-polishing as final step (ion energy 2 - 5 keV), the lamellae can achieve thicknesses below 30 nm, sufficient for high resolution investigations (HRTEM) [1]. A problem using FIB-samples, especially for HRTEM and energy loss spectroscopy (EELS) is the amorphous layer occurring during sample preparation. The high-energy (typically 30kV) Ga-ion-beam used to cut the TEM lamella implants Ga into the sample and alters the material up to 30 nm beneath the surface into an amorphous layer [2, 3].

Noble gas ion polishing at low voltages in the range of 500 eV is capable for removing the amorphous layer of FIB-lamellae. Ion milling typically done in dedicated preparation tools applying a broad (100 µm – 5 mm) Argon ion beam. In this study, we used a low energy Ar-ion gun (Penning Ion Gauge, PIG) which has been implemented into a two-beam focused Ion beam instrument (ZEISS, NVision40 Argon). The acceleration voltage for the Ar-ions can be adjusted from 500 to 1000 V. They are collimated and focused with an electrostatic condenser and electrostatic objective lens. The Ar beam current is typically set to 10-12 nA, at a beam diameter of about 100 µm. By choosing different gun and condenser settings, a beam diameter below 50 µm can be achieved, at the expense of available beam current. Electron-, focused Ga-ion-, and Ar-beam are coincident in the same point at the sample surface which enables real time SEM imaging of the FIB- and the Ar-milling process. The duration of the irradiation depends on the thickness of the damage layer to be removed, the beam energy and incidence angle. A few minutes of polishing per lamella side are usually sufficient.

The usability of in situ Ar-milled TEM-lamellae was investigated by high resolution imaging. Figure 1 shows a TEM-lamella prepared from a YAG-crystal [4] before and after in situ Ar-polishing. After the final Ga-Ion-milling step (Fig. 1 upper image), Ar-polishing (1kV Argon, ca. 5 min both sides, incident angle 5°) was applied. The removal of the amorphous layer is clearly visible.

Figure 2 and 3 show high resolution TEM and STEM images of the sample recorded at ZEISS Libra 200 microscopes with image CS-corrector for the HRTEM and illumination CS-corrector for the HRSTEM image. In the HRTEM image recorded at the edge of the crystal nearly no contamination could be detected. The fast fourier transform transfers information down to 80 pm.

The high angle annular dark field (HAADF) images in figure 3 were recorded several nm from the edge of the lamella. The sample was slightly thicker in this region. The amorphous layer as well as some sample damage by the Ga-ions are visible. The sample quality is anyhow sufficient for high resolution imaging.

These results proof that in situ Ar-Ion-polishing is a powerful enhancement to TEM-sample preparation in a focused ion beam instrument.

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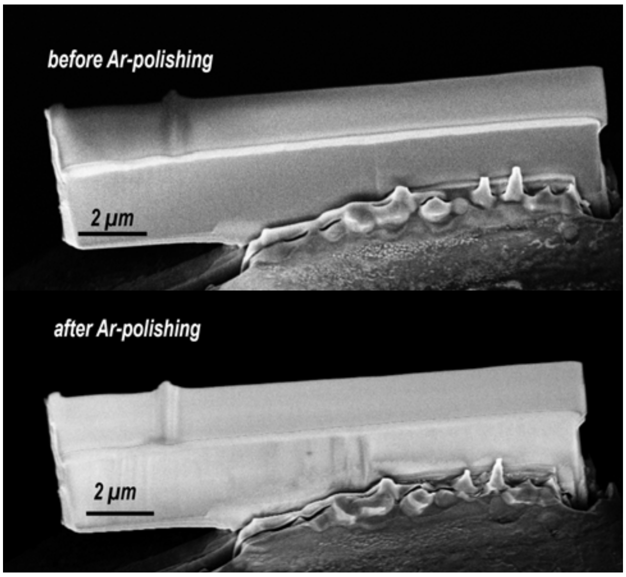

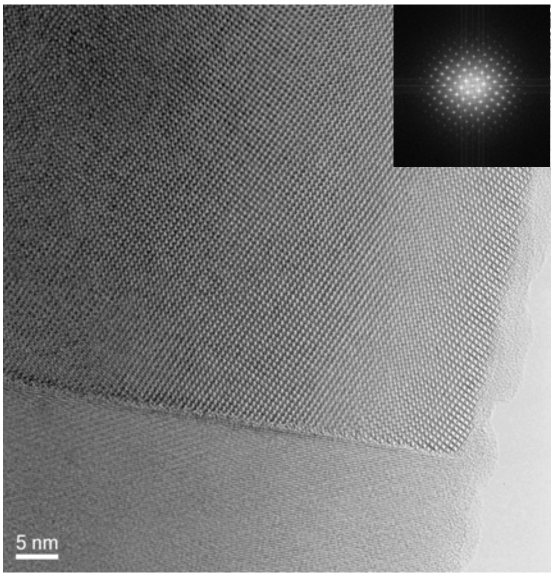

Figure 1. SEM images of a TEM lamella from a YAG crystal before (upper) and after (lower) Ar-ion-beam polishing. Removal of the amorphous surface layer after polishing is visible.

Figure 2. HRTEM image (CS=2 μm, DF = 5 nm) of the YAG crystal at the boundary side. Nearly no amorphous layer is visible. The inset shows the fast fourier transform of the image.

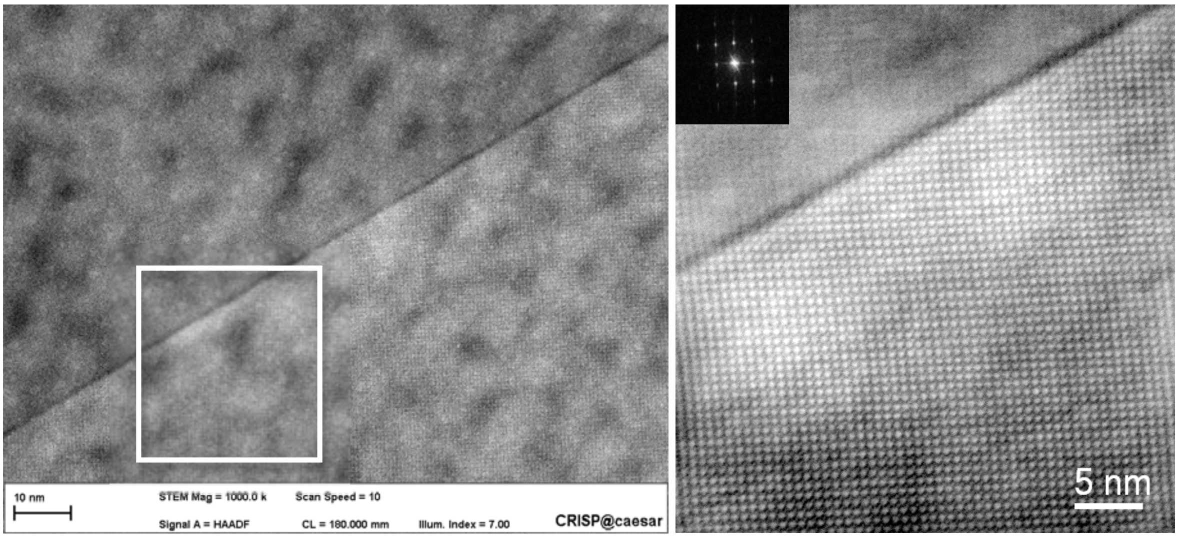

Figure 3. High-angle-annular-darkfield (HAADF) images of a YAG-bicrystal at the boundary of both crystals. The right image shows a detail at higher magnification . The inset shows the corresponding FFT.

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