DOI: 10.1017/S1431927605504872
Microsc Microanal 11(Suppl 2), 2005
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# Shadow FIBing - Using Geometry to Prepare TEM Samples

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Focused ion beam (FIB) instrumentation has proven to be extremely useful in preparing cross-sectional samples for transmission electron microscopy (TEM) investigations. The two most widely used methods involve either milling a trench in a bulk material while leaving behind an electron-transparent window or the so-called "lift-out" method [1]. While these two methods are very powerful in their versatility and ability to make site-specific TEM samples, they also rely on using a sacrificial layer to protect the surface of the sample as well as the removal of a relatively large amount of material, depending on the size of the initial sample. While neither of these two characteristics may be typically seen as drawbacks, some samples prohibit the use of a sacrificial layer or long milling times.

Shadow FIBing is a technique that relies on positioning a corner of a sample at an angle of incidence not normal to the electron beam. As seen in Figure 1, the thin film is positioned so that it is in the "shadow" of the ion beam. Subsequently, the focused ion beam is used to mill either side of the sample at the corner until a fin-shaped structure (shown in Figure 2a) arises on the film-side of the sample. By using this milling geometry, cross-sectional samples with 50 nm in thickness can be produced from virtually any material system without the use of a sacrificial layer (Figures 2b,c). In addition, by preparing the sample before milling with a cleaving technique, the initial tip of the sample is near electron transparency and requires significantly shortened milling times, supporting the reduction of the incorporation of Ga ions dramatically. Besides, the incident angle of the Ga ions with respect to the sample surface is virtually zero degrees minimizing sample modification and Ga ion contamination. In addition, the advantage of milling the sample from the backside of the area of interest is the fact that the inclusion of Ga ions is limited to the region (such as the substrate) far from the area of interest. As a result, cross-sectional TEM samples prepared by this technique are of very high quality and prepared in a very short amount of time.

The shadow FIBing technique is extremely versatile with regard to the different material systems it can address. There are systems which do not allow for traditional thinning techniques, and standard FIB cross-sectional or lift-out samples. For instance, the shadow FIBing technique was used to make a cross-sectional sample of a $VSe_2$ layered crystal that could not withstand the deposition of any sacrificial layer [2]. In this case, the $VSe_2$ surface had been previously altered by the deposition of 3 nm of Cu and it was undesirable to alter the surface any further. In another example, individual nanostructures (Ge islands) had been grown on a Si surface and the shadow FIBing technique was employed in order to preserve the structures on the surface unaltered, so that the possible sacrificial layer materials (in our case Pt or $SiO_2$) would not obscure sensitive chemical analysis [3]. In addition, multilayer complex oxide thin films which were unfeasible to prepare using traditional techniques owing to delamination issues was instead pre-cleaved and thinned by shadow FIBing. These and other examples of TEM samples prepared by the shadow FIB technique will be presented.

https://doi.org/10.1017/S1431927605504872 Published online by Cambridge University Press

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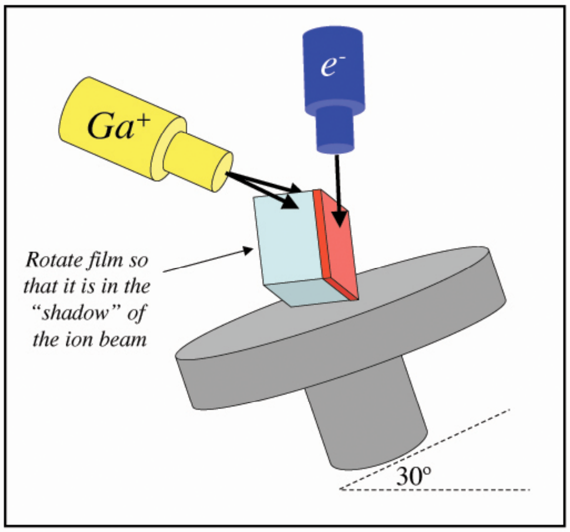


FIG. 1. Schematic of the Shadow-FIB technique for cross-sectional TEM sample preparation. In this case, a thin film (red) is tilted so that the surface is in the "shadow" of the ion beam. This geometry allows for the ability to cross-section a sample without using a sacrificial layer or long milling times. The ion beam is focused on either side of a corner of the sample, resulting in an electron-transparent wedge ending on the film surface. The angle of incidence of the ion beam can be varied depending on the circumstances, as long as it is directed from the back, substrate-side.

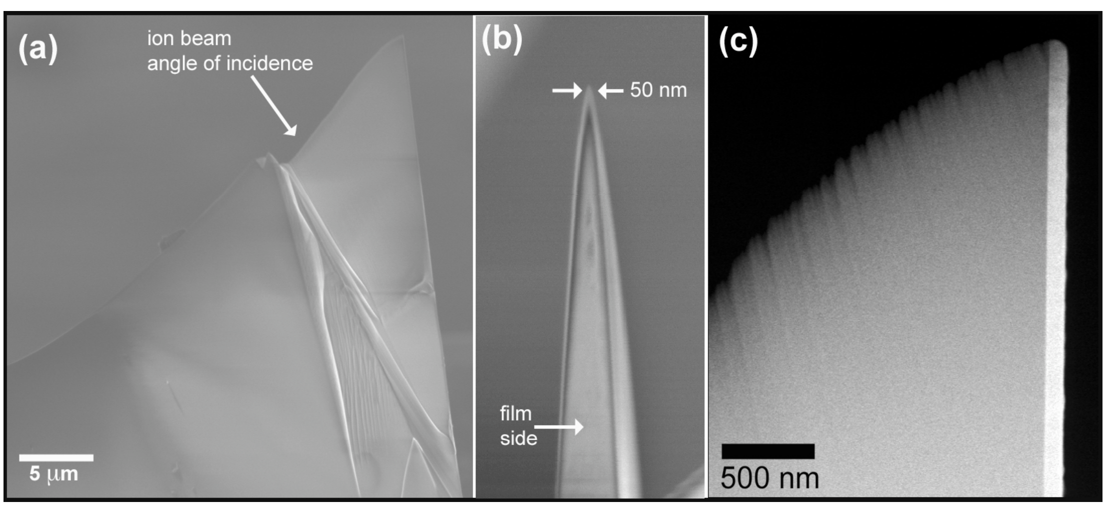


FIG. 2. An example of a TEM sample prepared by the shadow-FIB technique. In this case, an $HfO_2$ thin film on zirconia was cleaved prior to ion milling. (a) SEM picture from the side of the thin wedge showing the fin-like geometry of the sample corner. The film surface is on the far-right side of the sample, parallel to the viewing angle show here. (b) SEM image of the tip of the wedge shown in (a) rotated 90 degrees so the viewing angle is directly at the film side of the sample. (c) High angle annular dark field STEM image of the same sample, showing the electron transparent part of the wedge and the thin film.