doi:10.1017/S1431927618004701
Microsc. Microanal. 24 (Suppl 1), 2018

A Method for Plan-View FIB Liftout of Near Surface Defects with Minimal Beam-Induced Damage

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Focused Ion Beam (FIB) is a widely used technique in preparing traditional lamella type Transmission Electron Microscopy (TEM) samples with a cross sectional geometry [1]. However, additional challenges exist when preparing plan-view (i.e., with TEM sample parallel with surface) specimens of near surface, fine-scale features, and ensuring minimal damage is caused by the ion beam. Among these potential challenges are, maintaining the area of interest in the center of the liftout, ensuring that the near surface region remains in the thinned region of the lamella, and working with the plan-view geometry that is quite different from the normal cross-section geometry. Presently there are several plan-view TEM preparation techniques described in the literature, for example reference [2]. Here, we investigate how to minimize Ga-induced beam damage and prepare a site-specific plan-view specimen of a near-surface feature (<50 nm from the surface).

The sample chosen was a Cu tensile specimen strained just to the point of void nucleation in the necked region as a result of microvoid coalescence. First, an EBSD map was acquired on the sample surface in order to find areas with interesting orientation relative to the tensile axis (Fig1a). Next, near-surface voids were chosen in these regions for TEM investigation of the dislocation structure around them. These regions were protected with an e-beam-deposited C layer, then a subsequent ion-beam deposited Pt, and ion-beam deposited C layer (fig 1c). The resulting cuboid liftout was removed (fig 1d) and placed onto a TEM grid (Fig1e) with the grid mounted parallel to the original sample surface. After attaching the liftout, a layer of protection was also applied to the top of the liftout in order to protect the sample from beam damage during the cutting/thinning steps perpendicular to the area of interest. The sample was then thinned down to the appropriate (near transparent) thickness for TEM.

The resulting thin sample is shown in figure 2. Figure 2a) shows a 30 kV STEM-dark field image taken in the FIB. The grain structure and individual dislocations are clearly visible in the image. A 300 kV STEM image is shown in Figure 2b), where the void of interest can be seen, and individual dislocations around the void are easily visible. A high-quality result is highly dependent on stopping the thinning process as soon as the protective layer has been removed such that low-kV polishing can still occur on the near-surface feature and remove any high-kV beam damage. This technique provides high-quality TEM results and can be used to study near-surface defects in a plan-view orientation. This technique can also be broadly applied to other material and defect types [3].

References:
[1] L.A. Giannuzzi, F.A. Stevie, Micron **30(3)** (1999), p. 197.
[2] Chen Li *et al,* Ultramicroscopy **184(A)** (2018), p. 310.
[3] Sandia National Laboratories is a multimission laboratory managed and operated by National Technology and Engineering Solutions of Sandia LLC, a wholly owned subsidiary of Honeywell International Inc., for the U.S. Department of Energy’s National Nuclear Security Administration under contract DE-NA0003525.

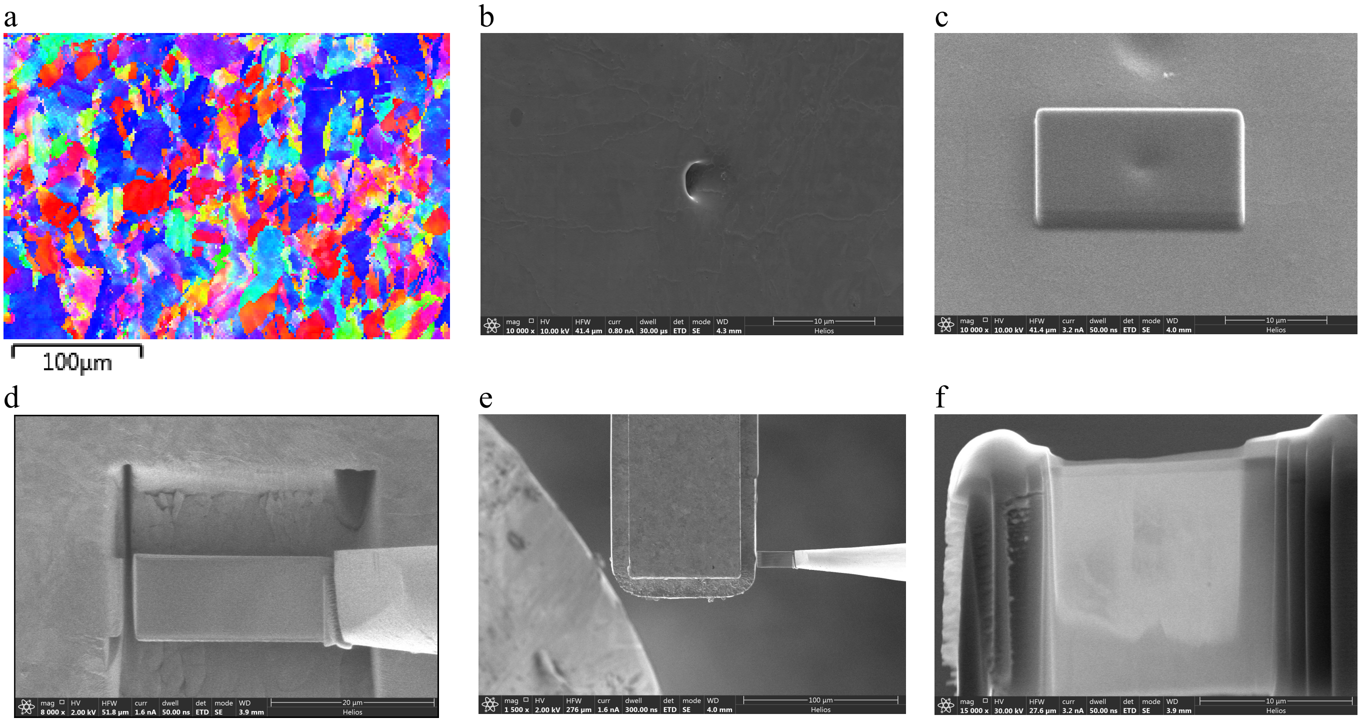

Figure 1. (a-f) Images of "area of interest". (a) EBSD Map (b) SEM image of "Void". (c) SEM image of protective layer (Pt and C) on "Void". (d) SEM image of lift-out on needle. (e) Lift-out attached to TEM grid. (f) Final thinned section for TEM

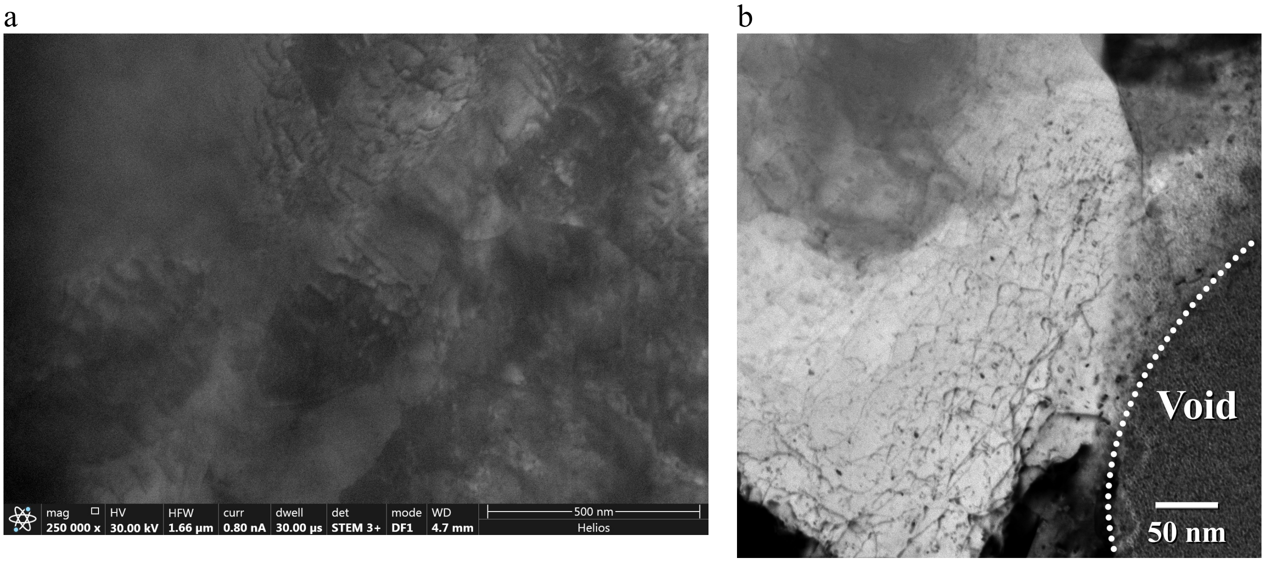

Figure 2. (a-b) Images of final thinned section. (a) 30kV STEM image. (b) 300kV STEM image of dislocations near "void" edge.