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"H-Bar Lift-Out" and "Plan-View Lift-Out": Robust, Re-thinnable FIB-TEM Preparation for Ex-Situ Cross-Sectional and Plan-View FIB Specimen Preparation

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Focused ion beam (FIB) specimen preparation for TEM has become the standard for semiconductor and high-end materials science TEM specimen preparation. The development of the "Lift-Out" technique [1,2] greatly facilitated rapid specimen preparation and reduced spurious bulk x-ray signal by physically removing an electron transparent cross-sectioned membrane from the bulk sample and placing this onto a thin carbon film for examination in the TEM. Further development of in-situ based techniques by vendors (Hitachi High-Technologies, OmniProbe, etc.) [3] and ex-situ based techniques [4] have been published detailing how to produce re-thinnable specimens.

Lacking an in-situ FIB micromanipulator, we undertook to follow the innovative technique of Rossie et al. [4], producing "bulk" lift-out cross-sectional membranes approximately 20 μm x 10 μm x 3 μm thick, and utilizing a 1000 mesh grid as the "carrier" for the bulk membrane. Using the suggested "SK-9" UV curable adhesive we were successful in reproducing their technique, but ran into a number of difficulties:

- the SK-9 UV curable adhesive was oxygen-inhibited and did not cure readily in air (nor did a myriad of other UV-curable adhesives we experimented with)
- the 1000 mesh membrane was difficult to cut without inducing warpage in the critical bars at the edge of the cut grid
- positioning the membrane at the edge of the mesh grid was often challenging and could result in significant manipulation of the membrane and resultant mechanical stress (especially significant with metallurgical specimens)
- even when successful, the specimen was quite fragile, and repeated mounting or unmounting of the specimen between FIB and TEM holders put stresses on the electron transparent sample which readily lead to tearing at worst or bend contours at best

As many of our specimens are fragile corroded grain boundaries or cracks, the stress that we observed during handling was detrimental to the specimens, frequently tearing them apart. Even "robust" specimens suffered due to grid flexure when repeatedly loaded into the TEM holder. Consequently we undertook minor modifications to the Rossie technique to produce more robust carriers that also protected the specimens and also made them robust for "shipping and handling".

Preparation of the base carriers for what we have dubbed "H-Bar Lift-Out" (HBLO) and "Plan-View Lift-Out" (PVLO) is straightforward and requires only a few low-cost items that are common to most microscopy laboratories. The preparation method for a copper carrier is as follows. A 3 mm copper aperture grid with a 600 to 800 μm diameter aperture is cut using a razor blade just above its halfway point. The smaller portion of this grid is mounted onto a polishing stub and a 30^o bevel is polished into the dull or rougher side of the grid. The polished piece of copper can then be removed from the stub and adhered to the opposite, larger side of the original TEM grid (bevel side out) using silver epoxy (Figures 1, 2). The knife edge of the beveled piece should be recessed from the top of the larger half-grid. The specimen is then mounted in the recessed area of the beveled piece exposed by the hole on the "front" side of the carrier, where it is best protected against impact with external objects. This technique has been applied successfully to Cu, Si, and Au carrier bars, but has the potential to be applied to a wide range of materials. Results from experiments with B_4C carrier bars designed for low EDX background will be presented.

To produce a cross-sectional FIB specimen (HBLO) a similar approach to conventional lift-out is performed, but the specimen is removed from the trench at a thickness of ~ 3 μm. At this thickness, the specimen is quite robust, and is easily placed on the carrier and slid into position without fear of "capsizing" as was common when we attempted the grid method of Rossie et al. To produce a plan-view specimen (PVLO) a more complex series of FIB cuts are performed to isolate and cut free a section of the specimen shaped like an inverted barn [5].

To avoid potential carbon contamination from the adhesive, we have found it feasible to position the thick membrane or barn on the carrier without the use of any adhesive and return the carrier to the FIB where the thick membrane can fixed in place by FIB deposition (in what was a surprise to us, the membranes stay firmly affixed during pump-down and reentry into the FIB chamber and during subsequent imaging and deposition).

Once the specimen is returned to the FIB chamber, the portion of the carrier that would obscure the line of sight in the TEM is then milled away using the FIB (Figure 3). This is facilitated by the 20° to 30° bevel that is polished into the backside cross-piece of the carrier, and requires only 20 to 30 minutes with a 20 nA beam current. During FIB thinning, some carrier material must be left on either side of the specimen so that it remains securely mounted, but sufficient material is removed to allow 40° of tilt in all directions. Finally, the specimen itself is thinned down to electron transparency. The combination of carrier and specimen can then be taken to the TEM and imaged. If further thinning is required, the specimen is returned to the FIB as needed.

References
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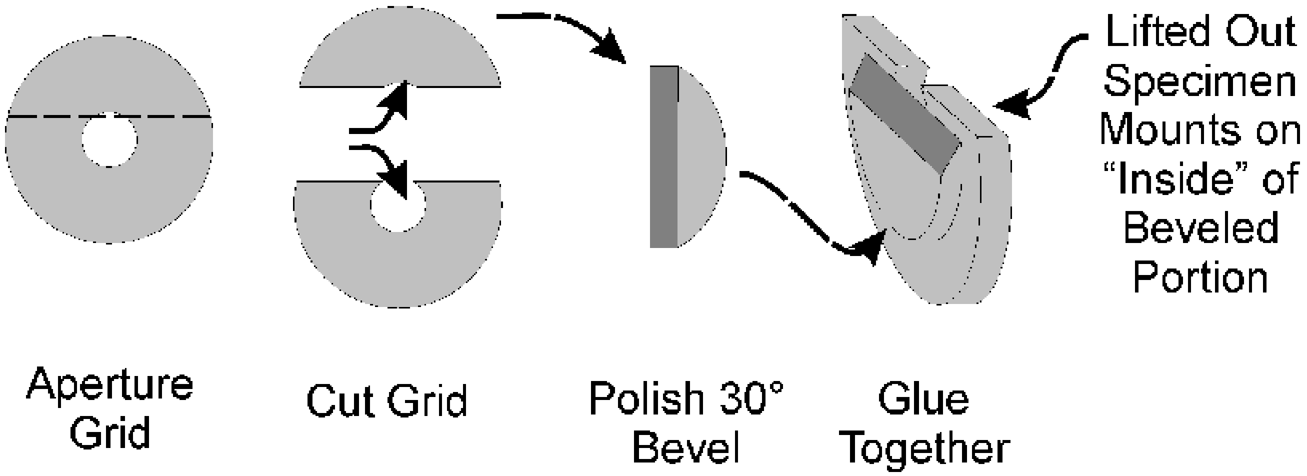

FIG. 1. Schematic representation of the preparation of a PVLO or HBLO "Carrier". During mounting the specimen is easily slid into position. Once mounted on the inside surface of the beveled bar the specimen is well protected.

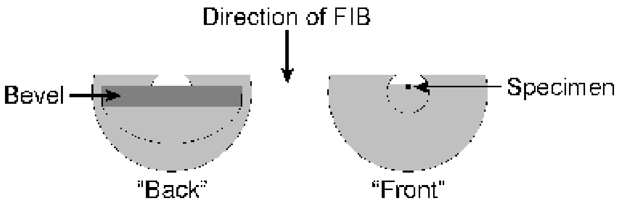

FIG. 2. Schematic front and back views of the carrier with a specimen in place.

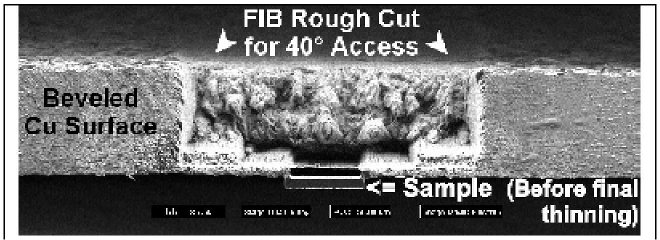

FIG. 3. FIB secondary electron image after rough cutting the carrier (~ 20 to 30 minutes at 20 nA beam current).