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Flexible Grid Holder Enabling FIB-SEM Sample Prep and Analysis

C. Hartfield[1] and F. Bauer[2]

[1.] Oxford Instruments America, Concord, MA, USA
[2.] Oxford Instruments GmbH, Wiesbaden, Germany

There are a number of challenges for preparing samples for transmission electron microscopy (TEM) and atom probe tomography (APT) using focused ion beam – scanning electron microscopes (FIB-SEM). Some of these include navigating in 3D space using only 2D images, manipulating a sample to a specific orientation using equipment having limited degrees of freedom, and processing samples into optimized geometries with minimal artifact [1, 2]. The FIB-SEM most commonly prepares TEM and APT samples into geometries that include rectangular thin sheets (lamella), pentahedron shapes, and micropillar or sharp needle shapes. Prior to analysis, all of these samples are placed on a carrier to aid final processing or transfer to a desired instrument. A common carrier is a <100um thin, 3mm wide half-disc of Cu or other metal, known as a "lift-out grid" or "half-grid". Due to their size and fragility, grid holders are used to aid handling. There are many types of grid holders. Examples here show strategies for the efficient preparation and analysis of high quality samples by implementing a new pivoting grid holder having one degree of freedom. This aids many techniques including TEM and APT sample preparation, SEM imaging, and electron backscatter diffraction (EBSD) and transmission kikuchi diffraction (TKD) analysis.

Grid holder pivot positions of 0°, 45°, and 90° are all that are required to enable effective preparation and analysis across the above-mentioned applications. In the 0° and 90° positions, the grid is held horizontally or vertically with respect to the base of the grid holder, respectively (fig. 1). These latter two positions are useful for creating plan-view as well as backside TEM samples. In both cases, the holder's 0° pivot position is used to place the grid horizontally prior to lifting out and attaching the TEM sample. After the sample is attached, the holder is moved to the 90° pivot position, which places the grid in the standard "vertical grid" position that is commonly used in lift-out practice for the final thinning of TEM lamella. By combining this holder with a nanomanipulator having a rotation axis, a backside sample can be rapidly created with a single lift-out step (fig. 2). The 0° and 90° positions also facilitate EBSD and TKD analysis respectively, after attaching the pivoting holder to a fixed-angle holder which allows achieving the desired analysis angles. The design of the pivoting holder is compatible with the requirement to avoid any shadowing of the signal.

The 45° position aids creation of APT samples and SEM imaging. Assuming a FIB having an ion column at 52°, it is simply a matter of tilting the sample stage to 7° to position the sample's top surface orthogonally to the ion beam for needle sharpening, and then rotating the stage 180° so the sample's side surface is orthogonal to the beam for flattening surfaces and slicing off capping layers (fig. 3). The 45° position also aids holding the sample for SEM imaging at 1mm working distance when the FIB-SEM stage is tilted to 45°.

In summary, a grid holder pivoting with one degree of freedom assists sample preparation and imaging over many applications, including in situ lift-out of plan-view and backside TEM samples, atom probe samples, SEM analysis at 1mm working distance, and EBSD and TKD analysis. It is expected that additional new uses will appear.

References:

[1] C. Hartfield *et al*, Microscopy and Microanalysis, 16 (Suppl.2) (2010) p. 16.
[2] C. Hartfield *et al*, Electronic Device Failure Analysis Magazine, 13:3 (2011) p. 18.

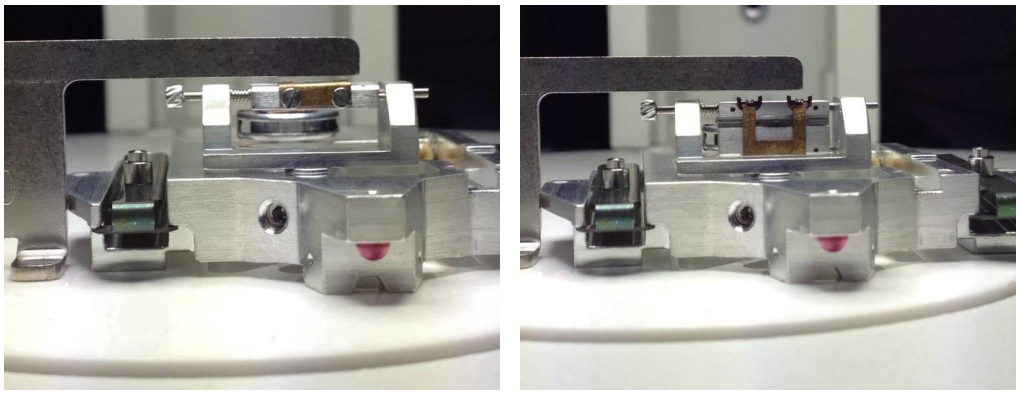

Figure 1. Pivoting holder positioned at 0° (left) and 90° (right) pivot positions

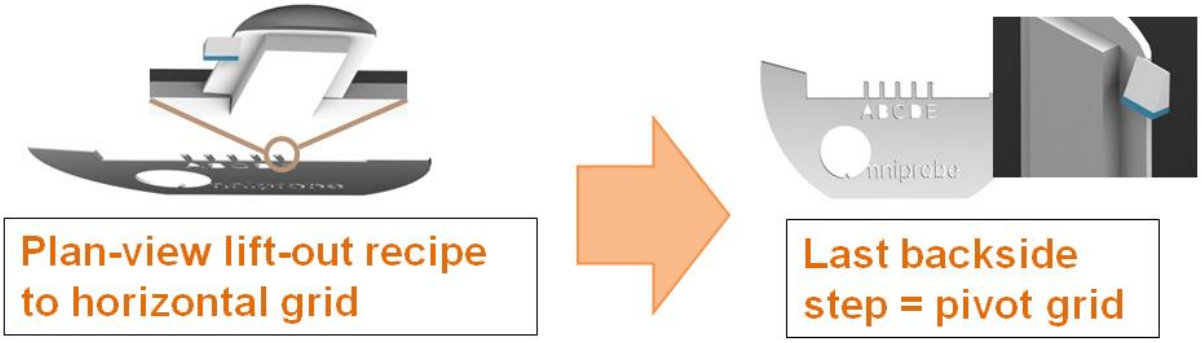

Figure 2. A backside TEM sample can be made in one lift-out step by combining the pivoting holder with a rotating nanomanipulator workflow typically used to create plan-view sample.

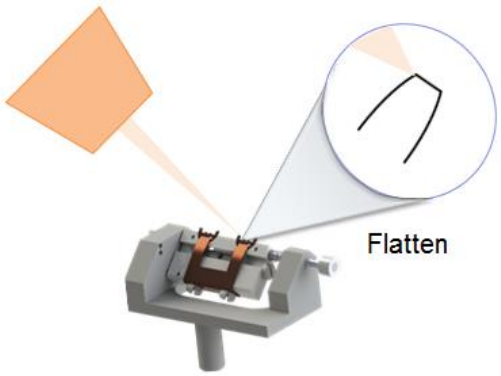

Figure 3. The 45° pivot position combined with sample stage tilt and rotation helps with the creation of flat surfaces and the slicing off of capping layers from atom probe tomography samples.

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