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Sample Repositioning Solutions for in situ Preparation and Analysis

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Since the world's first sale of commercial equipment designed specifically for FIB in situ lift-out (INLO) for TEM sample preparation in 1999, INLO has grown across many industries and established itself as a dominant method not only for TEM sample preparation, but also for atom probe analysis and other types of analyses that occur in an electron microscope. Reasons for this are numerous including site specific removal of features with small dimensions compatible with FIB spatial accuracy of 20nm [1], ability to prepare samples without destroying the bulk sample, production of samples compatible with a clear view at all angles for TEM inspection, and improved speed compared to other site specific preparation methods.

More recently, variations of INLO processes have been developed to meet the needs for better final sample quality and for alternate FIB milling angles. With the improved imaging capabilities of aberration corrected and high resolution electron microscopes come more stringent requirements on sample preparation to produce an adequately thin and uniform sample free of artifacts. Some of the applications driving these approaches include 1) elimination of curtaining artifacts on heterogeneous samples, including but not limited to semiconductors [2]; 2) requirements to obtain uniformly thin lamellae from samples with structures having high aspect ratios over a long depth, especially when two or more materials of dramatically different milling rates are present [3]; 3) requirements to conserve stresses so TEM strain measurements are representative of the sample in its native state [3]; 4) requirements to create planar vs. cross-sectional TEM samples for identifying rare defects or defect originations over a large area; and 5) requirements to produce an adequate sample for high resolution electron microscopy (sub-50nm lamellae thickness, uniformly thin, free of artifacts and sample damage).

The traditional INLO method accomplished milling the INLO sample with the beam impinging orthogonal to the sample top surface (also known as frontside milling). Although frontside milling in many cases provides adequate samples, applications mentioned above require milling a sample that has been rotated 90 (sideways thinning) and 180 degrees (backside thinning) relative to the normal frontside position. Methods to reorient a sample for FIB thinning are varied and not necessarily intuitive. Simply rotating an INLO sample held on the end of a probe needle will usually not achieve the desired end position for milling without additional steps, and the different approaches that do work can vary significantly with respect to the number of steps, length of time, difficulty, and expertise required to accomplish the task.

References

[1] J. Mayer et al., *MRS Bulletin,* 32 (May) (2007) 400.
[2] L.A. Giannuzzi and F.A. Stevie, *Introduction to Focused Ion Beams, Instrumentation, Theory, Techniques and Practice*, Springer, New York (2005).
[3] R. Irwin et al., *JVST A* 27(6), Nov/Dec (2009) 1352.
[4] The aid of Leigh Ann Robidoux for the figure illustrations is gratefully acknowledged.

Different approaches to orient and prepare samples for further FIB processing or analysis will be presented and compared, including new efficient methods that allow all manipulation to be accomplished in situ, without requiring removal of the sample from the chamber for repositioning.

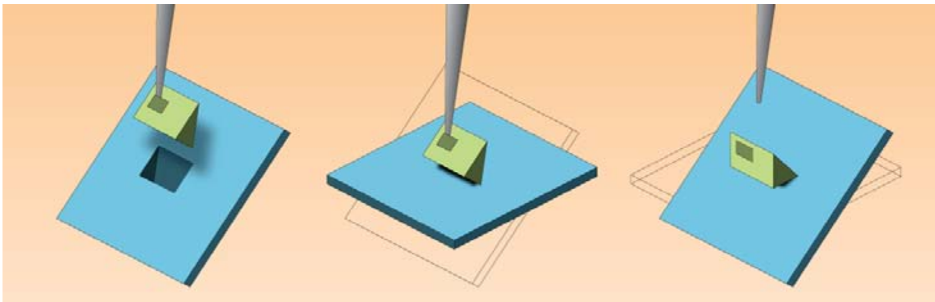

FIG. 1. This illustration of the “double lift-out” method for planar sample preparation shows the requirement to manipulate the stage tilt from 45 degrees to zero back to 45 degrees with two “attach and remove” lift-out steps in order to achieve the 90 degree orientation required for milling a final lamellae that is within a plane parallel to the sample surface.

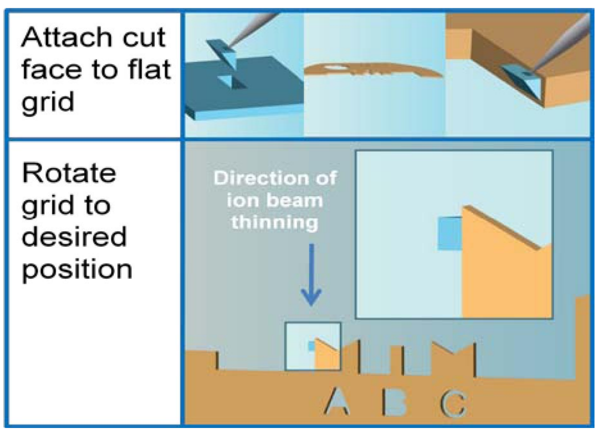

FIG. 2. In the “rotated grid” method for planar preparation, the grid is repositioned after attaching the sample while the grid is laying flat.

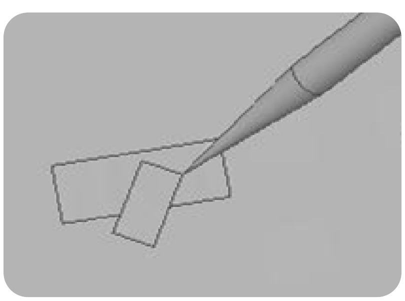

FIG. 3. In the stage-shaft rotation method, mathematical transformations based on the conversion from angle-axis representation to rotation-matrix representation are calculated to determine stage tilt and rotation, combined with manipulator shaft rotation, to achieve placement for planar sample preparation in one lift-out step without requiring venting.

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