An **Oxford Instruments** Company

APPLICATION NOTE

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A Short-Cut™ for TEM & Atom Probe Sample Preparation

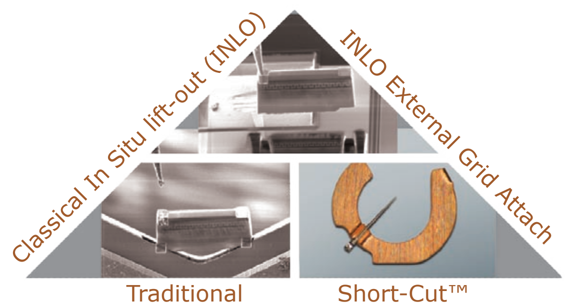
INTRODUCTION

In many applications, atom probe and TEM analyses are desired at highly site-specific positions. Examples include phase-segregated regions in alloys, grain boundaries in ceramics, individual devices in semiconductor components, or even mechanical failures, such as crack tips and indentations. In some cases, the standard FIB lift-out specimen preparation is easily applied. For other cases, it may be difficult to properly align the feature of interest axially on the post and retain the feature of interest at the specimen tip after FIB milling. This is especially true for materials that have different sputtering rates, such as oxides in metallic matrices and the widely varying compositions present in semiconductor devices.

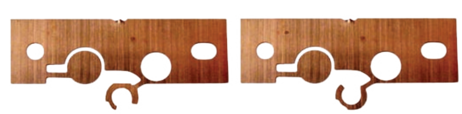
Atom probe analysis can be significantly inhibited by the presence of insulating materials within the specimen. Dielectric layers can lead to field concentrations, and ultimately specimen fracture during analysis. This effect is significant in semiconductor devices processed on silicon-on-insulator (SOI) wafers. Backside specimen preparation eliminates this issue by placing the substrate side towards the atom probe detector, so the SOI layer is accessible to FIB mill away before atom probe analysis. Typically, this requires ex-situ specimen and nanomanipulator rotations that are time consuming and unreliable. A solution to this problem is provided by a simple adaptation to Omniprobe's Short-Cut™ technique [***1***] and grid geometry [***2***]. The result allows expedited site-specific, backside atom probe specimen preparation with an efficiency that enables routine use.

SHORT-CUT™ OVERVIEW

Omniprobe's Short-Cut™ pneumatic benchtop press was developed to simplify the step of transferring a lift-out sample to a suitable holder, like a TEM grid, such that fast and easy transfer can be readily accomplished by both new and experienced analysts alike. The Short-Cut™ provides reliable attachment and is sensitive to FIB resource constraints by performing attachment ex situ. Traditionally, the in situ lift-out method requires several in situ manipulations to transfer the sample from the probe tip to a suitable holder. These steps involve retracting the needle that holds the sample to a safe position, maneuvering the stage to bring the holder into the field of view, reinserting the needle and precisely positioning it so the sample touches or is very close to the holder, welding the sample to the holder by FIB assisted deposition, and then ion beam milling to release the needle.

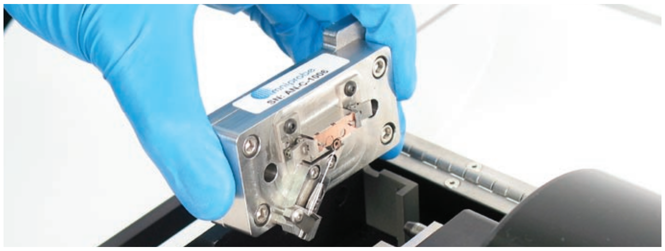


Traditional INLO vs. Short-Cut™ assisted INLO Although some indicate these in situ handling steps can be efficiently performed to complete the entire transfer sequence in approximately 10 minutes [***3***] others report it can take as long as 45 minutes [***4***]. Many factors play into these time differences, including user expertise, FIB and manipulator hardware, and sample geometries. The Short-Cut™ transfer process levels the playing field and results in repeatable transfer times, regardless of these differences. The Short-Cut™ pneumatic bench-top press includes several accessories that help easily and reliably transfer the probe tip and sample to a sample holder. The sample holder starts out as a preform, or coupon. There are two coupon designs for TEM sample preparation to facilitate FIB thinning from either the top of the sample (frontside) or from the bottom of the sample (backside).



Frontside & Backside thinning coupons

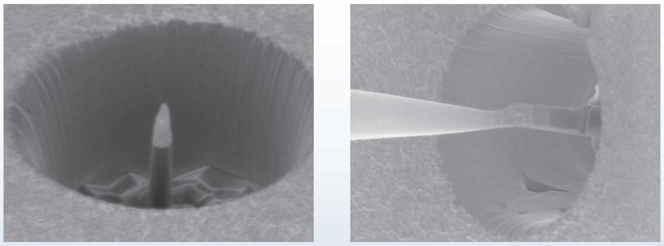
The coupon is mounted on a mechanical anvil, along with the probe needle that holds the sample. The anvil design ensures automatic alignment of all components. The loaded anvil is placed in the Short-Cut™ press, and with the simple push of a button, the probe needle, with sample attached, is automatically aligned and transferred to a TEM-compatible grid-like holder.



Short-Cut™ anvil, loaded with coupon and needle

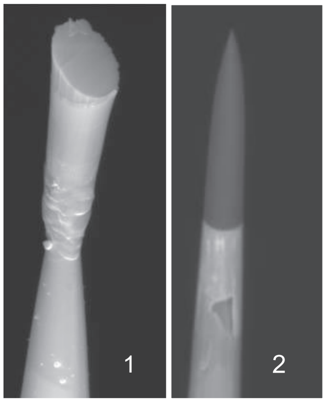
BACKSIDE ATOM PROBE ANALYSIS

In addition to being site-specific and eliminating layers that would normally cause difficulty during atom probe analysis, backside atom probe preparation facilitated with Omniprobe's Short-Cut™ is also more rapid compared with the standard lift-out technique. It requires less FIB time during initial metal deposition and specimen removal, as well as during final preparation FIB milling. As in standard lift-out techniques, the new site-specific atom probe preparation leveraging the Short-Cut™ requires the area of interest to first be protected using a deposited material. The major difference in the new technique is in the amount of area defined. For the traditional lift-out technique, an area approximately 2 × 20 µm is prepared, whereas the atom probe site-specific technique only requires a circular area approximately 500 nm in diameter. After the area of interest has been defined and protected by metal deposition, a cylindrical post is cut from the sample. The specimen is then rotated until the nanomanipulator is parallel to the long axis of the cylinder. Again using FIB deposition, the nanomanipulator is attached to the cylinder, and the cylinder cut loose.



A cylindrical sample is created, tilted, and attached to a probe needle

Once extracted from the sample, the specimen is left attached to the nanomanipulator and loaded into the Short-Cut™ press. A punch operation subsequently cold forms the nanomanipulator needle into the grid material while concurrently sectioning the needle. The newly created assembly can be introduced back into the FIB for final specimen shaping.



Sample on probe tip after release (1) & final shaping (2)

The final specimen form gives line of sight access from the specimen tip to the atom probe optics. A specific advantage of forming the manipulator tip to the grid material is that TEM examination of the atom probe specimen is still possible. If TEM is not going to be used, the nanomanipulator needle itself can be loaded into the atom probe. It should also be noted that since this technique produces only a single specimen, local electrode atom probe is not required.

The Short-Cut™ makes triple-compatible atom probe/TEM/tomography samples

The geometry of the specimens when using these site-specific techniques is particularly advantageous for TEM tomography. Cylindrical specimens do not change thickness with tilt angle, and the geometry of the grid and specimen are such that the grid will not enter the specimen field of view during tilting, making 360° rotations possible [**5**].

REFERENCES

1.*US Patent 7126132. (2006)*

2.*US Patent 7115882. (2006)*

3.*Langford RM, Rogers M. In situ lift-out: Steps to improve yield and a comparison with other FIB TEM sample preparation techniques. Micron. 2008.*

4.*Delaye V, Andrieu F, Aussenac F, Faynot O, Truche R, C. Carabasse, et al. In-line transmission electron microscopy for micro and nanotechnologies research and development. Microelectronic Engineering. 2008;85:1157-61.*

5.*Gorman BP, Diercks D, Salmon N, Stach E, Amador G, Hartfield C. Hardware and Techniques for Cross-Correlative TEM and Atom Probe Analysis. Microscopy Today. 2008 July 2008:42-7.*