

Transfer of Sensitive Samples from FIB to TEM

P.O. Dickerson*, R.M. Dickerson*, T.M. Moore**, G. Amador**, L. Zaykova-Feldman**

*Los Alamos National Laboratory, MST-6 MS G770, Los Alamos, NM 87545

**Omniprobe, Inc., 10410 Miller Road, Dallas, TX 75238

The FIB in-situ lift-out method of TEM sample preparation has become the method of choice for failure analysis during the last decade [1]. During in-situ lift-out in the FIB, a tiny wedge is excised from the sample, attached to a TEM grid, and transferred to the TEM for inspection. However, some samples react with the air and require transfer and handling in a controlled atmosphere [3-5]. There is a need for an integrated system for easy and secure transfer of TEM samples, or any other sensitive samples, to the final destination in an appropriate inert atmosphere.

The Sensitive Sample Transfer System (SSTS) is a low-cost system for integrating the transfer process from the FIB to the TEM or any other analytical instrument. This system was developed in collaboration with the Los Alamos National Laboratory.

The SSTS is composed of three principle components, the FIB interface assembly, the sealed transfer capsule (both shown in Figure 1) and the custom inert atmosphere glove box (Figure 2). The FIB interface assembly mounts to a side port on the FIB and consists of docking hardware, a capsule purge system, an automated gate valve and "smart" vacuum interlock electronics. The transfer capsule is used to safely convey a sample holder carrying two or more TEM grids from the FIB environment to the glove box, holding the samples in an inert environment during the process. At the transfer destination point, a custom inert gas glove box provides a protective environment for transferring the FIB sample from the transfer capsule to a subsequent environmental sample holder for the TEM.

The sensitive sample transfer process begins by loading one or two empty TEM grids onto the special dove-tail sample holder (Figure 3) which is then fastened to the end of the transfer capsule shaft. The transfer capsule is securely attached to the flange of the FIB interface assembly and the FIB is then pumped down to operating conditions. During pump-down, the capsule rod is inserted into the FIB to dock the TEM sample holder onto the FIB XY stage, then the rod is retracted. Once specimen excise and lift-out procedures are completed and the samples have been attached to the TEM grids, the transfer rod is re-inserted into the FIB to recapture the dove-tail sample holder and retract it fully into the capsule. The electronics on the FIB interface assembly automatically sense the rod retraction and close the gate valve, isolating the transfer capsule from the FIB. At this point push button controls can be activated to introduce an inert gas into the transfer capsule. The custom valve on the capsule maintains the inert environment at slightly above atmospheric pressure. The transfer capsule is detached from the FIB interface assembly and placed in a protective carrier for transport to the glove box. The transfer capsule is next docked to the glove box which is purged with the same inert gas

as the capsule. Within the protective atmosphere of the custom glove box the sensitive sample TEM grids are transferred from dove-tail sample holder to a subsequent environmental sample holder, i.e. a TEM sample holder.

The SSTS marks a significant step in the design of secure sample transfer systems. Similar systems can be used for different types of fragile samples which require transfer in a specific environment. This system can be used in a variety of fields such as biotechnology and the space studies, for example, for handling extraterrestrial samples in a controlled atmosphere.

[1] L.A. Giannuzzi et al., “FIB Lift-Out Specimen Preparation Techniques”, in *Introduction to Focused Ion Beams: Instrumentation, Theory, Techniques, and Practice*, L.A. Giannuzzi, F.A. Stevie (eds), Springer-Verlag New York, 201 (2004).

[2] FEI Company UltraView™ NanoLift™ Wafer Biopsy, Sample Transfer Process for the Fab and Lab.

See http://www.feicompany.com/productFiles/documents/Nanolift_DS_0704_v2.pdf

[3] Gatan 626 Single Tilt Cryotransfer System.

See www.gatan.com/holders/626_cryo.html

[4] G. Snell et al., *Structure* 12 (2004) 537.

[5] C. S. Potter et al., *J. Struct. Biology* 136 (2004) 431.

[6] The authors wish to thank to R. Kruger, A. Smith, C. Anderson, A.J. Bava and A. Soekamto for their dedicated efforts.

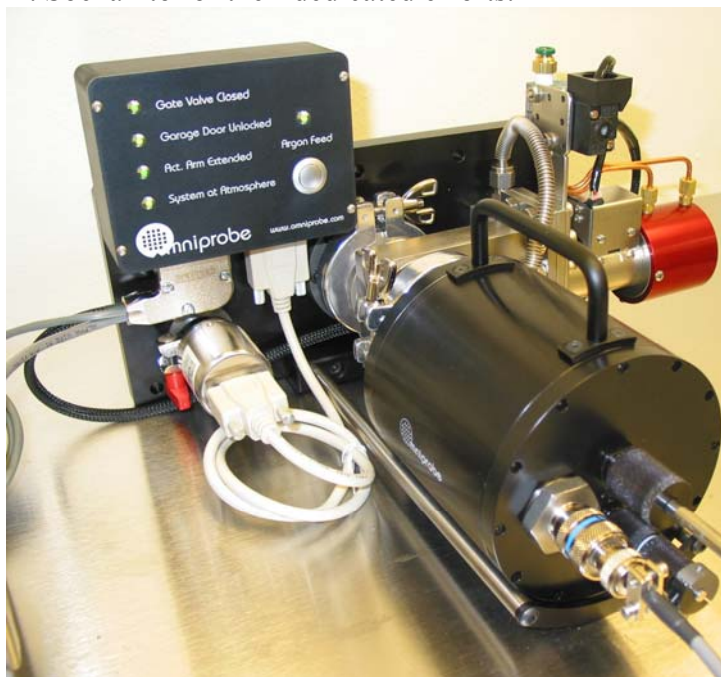


Fig. 1. The Capsule attached to the FIB Interface Assembly



Fig. 2. The custom inert gas Glove Box with video camera and video monitor

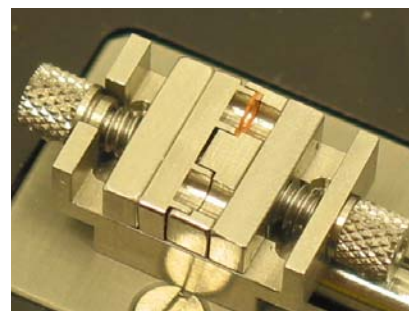


Fig. 3. The Dove-tail Sample Holder with a single TEM grid