ISTFA 2014: Conference Proceedings from the
40th International Symposium for Testing and Failure Analysis
November 9–13, 2014, Houston, Texas, USA

ex situ Lift Out of Plasma Focused Ion Beam Prepared Site Specific Specimens

Lisa Chan and Jon M. Hiller
Tescan USA, 508 Thomson Park Dr., Cranberry Township, PA 16066

Lucille A. Giannuzzi
EXpressLO LLC, 5483 Lee St, Unit 12, Lehigh Acres, FL 33971

Introduction

ex situ lift out (EXLO) was historically the first lift out technique to be developed for site specific removal and manipulation of focused ion beam (FIB) prepared specimens to a suitable carrier [1]. Early publications simply refer to this technique as "lift out" since further specification was not necessary. EXLO was later coined to distinguish it from lift out methods performed inside the FIB instrument.

EXLO is performed outside of the FIB instrument on a unit basically consisting of a light optical microscope and manipulator.The specimen is usually picked from within the FIB trenches with adhesion forces using a glass probe manipulator. For scanning/transmission electron microscope (S/TEM) characterization, the specimen is then placed on a thin film coated TEM grid [1]. The lift out and manipulation process takes only a couple of minutes [2]. Known advantages to EXLO include ease of use, high throughput and high > 90% success rates [2].

Recently developed methods negate the need for a thin film coated TEM grid [3-8]. A slotted half grid allows specimens to be manipulated to the grid itself. This allows for a better heat sink and electrical path to ground. Since a carbon film may inhibit certain S/TEM techniques such as energy filtered imaging, electron energy loss spectroscopy or electron holography, the absence of this thin film allows all S/TEM techniques to be performed. More importantly, since no thin film support is needed, the specimen can be further processed after manipulation. In addition, a thick specimen can be easily manipulated to the slotted grid into a backside orientation, and then FIB milled to a suitable thickness for S/TEM without producing curtaining artifacts [4,8].

EXLO was developed in the age of Ga^+ FIB columns with ~ 50 nm beam sizes. The modern Xe^+ plasma FIB (PFIB) column operates at about this same 50 nm beam resolution or better. Therefore, it is no surprise that the Xe^+ PFIB is capable of preparing electron transparent specimens for S/TEM [9]. In addition, the PFIB offers inert or other ions of choice for specific applications. Since the PFIB can operate at much higher beam currents (with a smaller probe size) than a Ga^+ FIB, the PFIB's strength is large volume material removal [10,11], which can be exploited for TEM EXpressLO methods [12]. The requirement of producing large lift out specimens increases the FIB time dramatically [13]. For example, it took ~ 4 hours to prepare > 100 µm long TEM specimens prepared via *in situ* lift out via Ga^+ FIB [14]. Thus, in this paper, we combine fast PFIB preparation of large S/TEM specimens with fast conventional EXLO and EXpressLO "pick and place" solutions. The combination of large material removal rates with PFIB and EXLO allows for efficiency and high throughput of FIB lift out specimens.

Experimental Methods

Specimens from a patterned Si wafer were prepared for EXLO and EXpressLO using a Tescan FERA3 Xe^+ PFIB operating at 30 keV. PFIB W deposition marked the region of interest. Each large specimen measured 40 µm x 15 µm x 1 µm and was prepared in well under 30 minutes using successively smaller beam currents of 100 nA, 30 nA, and 10 nA. An example of a final PFIB prepared specimen is shown in the scanning electron microscope (SEM) image in figure 1. Lift out success rates improve dramatically when the specimen is completely FIB milled free (or nearly free as in figure 1). EXLO and EXpressLO examples were performed on an EXpressLO LLC lift out station as shown below in figures 2 and 3.

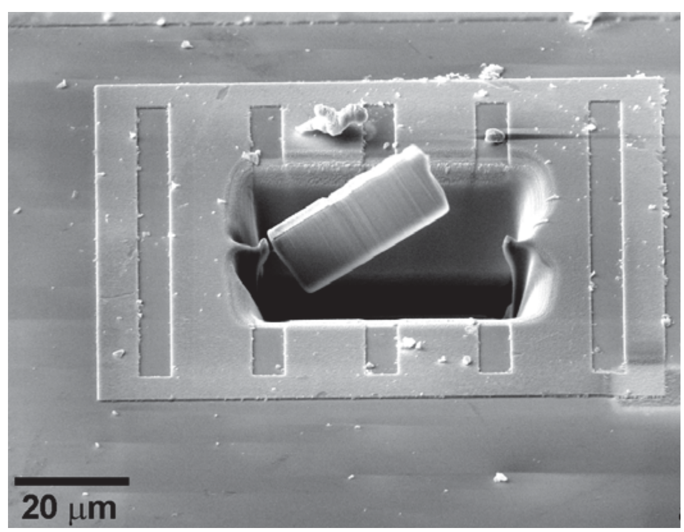

Figure 1: SEM image of PFIB prepared specimen.

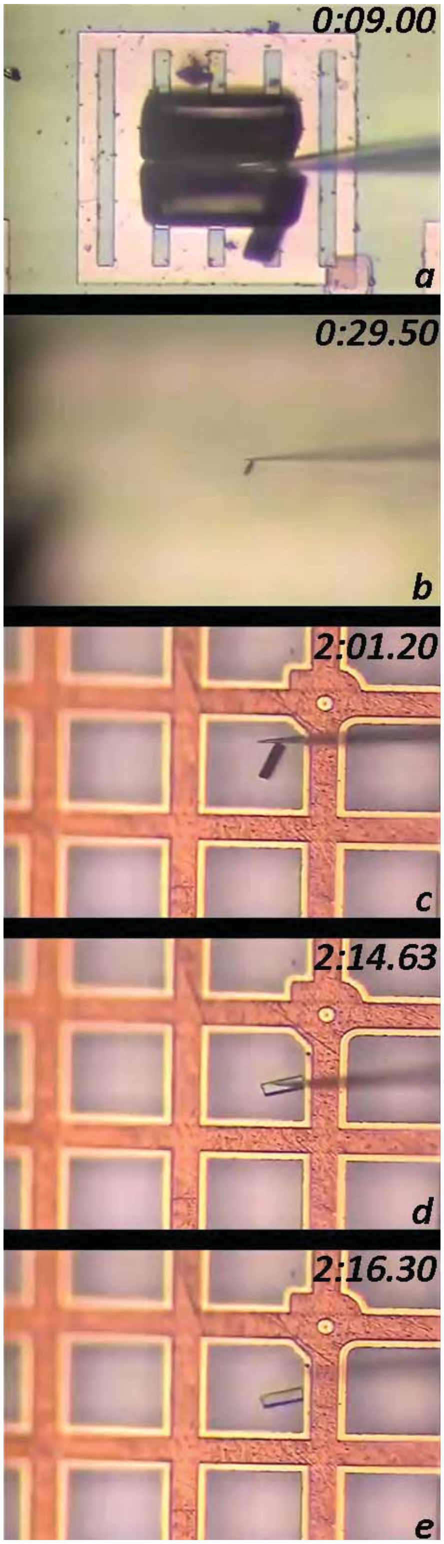

Figure 2: Sequence of EXLO steps for a PFIB prepared specimen from lift out to manipulation to a formvar coated Cu TEM grid. The PFIB specimen length is 40 μm.

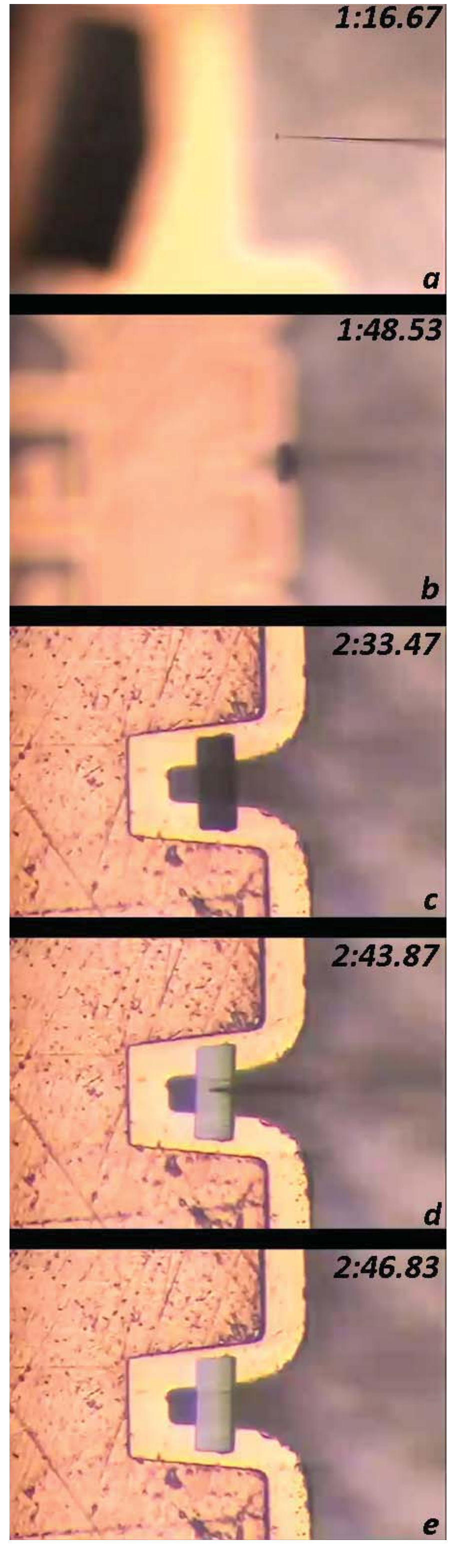

Figure 3: Pick&Place sequence of EXpressLO steps for a PFIB prepared specimen from lift out and manipulation to a slotted Cu TEM grid [8]. The PFIB specimen is 40 μm long.

Results and Discussion

Conventional EXLO

Figure 2 shows a sequence of images during the EXLO of a PFIB specimen and manipulation to a formvar coated Cu TEM grid. The time stamp from the video acquisition is noted on each image. As shown, the entire process takes just over a couple of minutes. Figure 2a shows the PFIB specimen secured on the probe tip in under 10 s. Figure 2b shows a low magnification image of the specimen attached to the probe tip. In figure 2c the grid has been centered and the specimen has been lowered close to the desired grid square. In figure 2d, the probe tip gently touches the top of the specimen, to ensure it is flat and secure. The probe tip is raised and the specimen transfer is completed in figure 2e in just over 2 minutes.

EXpressLO Pick&Place

Figure 3 shows a sequence of images during EXpressLO of a PFIB specimen and manipulation to a slotted Cu TEM grid. The time stamp from the video acquisition in figure 3 shows the elapsed time after the specimen has been picked out of the trench. As shown above, the specimen "pick" generally takes ~ 10s if the specimen is completely FIB milled free. The entire "place" process to the slotted grid after the specimen "pick" in figure 3 takes just under 3 minutes.

Figure 3a shows the PFIB specimen positioned on top of the probe tip and just above the EXpressLO Cu grid. If the "pick" does not result in this preferred orientation of the specimen for the EXpressLO process, the probe can be easily rotated to orient the specimen on top of the probe. Figure 3b shows the specimen and grid close to the same focal plane. When the probe and grid both start to appear in focus as in figure 3b, the probe is very close to the grid surface. At this point, the probe with the specimen is centered over the slot opening in the EXpressLO grid. In figure 3c the probe is lowered and manipulated through the slot opening such that the specimen comes to rest and is "placed" on the grid support structure. The probe then gently touches the specimen to ensure it is flat and secured to the grid (figure 3d). After correct placement, the specimen reflects brightly in the light optical image as per figure 3d. The probe is raised and the specimen "place" is finished completing the "Pick&Place" procedure as per figure 3e.

Figure 4 shows an SEM image of the specimen placed on the EXpressLO grid after ion beam assisted W deposition (indicated by the arrow) was used to further secure one side of the specimen. Certainly, this W deposition step is not needed for careful handling as the adhesion forces are sufficiently large to successfully transfer the specimen attached to the grid from the lift out station in ambient conditions into the FIB/SEM vacuum (figure 4). Indeed, electron transparent specimens manipulated to an EXpressLO grid have been transferred directly to a TEM for analysis (without further deposition to secure the specimen) [3]. This added W deposition is performed as a precautionary measure to secure the specimen during further FIB milling. At this point, it is not known if this added W deposition step is absolutely necessary prior to further FIB milling, however, the added time required for the W deposition step is minimal compared to the alternative scenario of having to re-FIB mill another lift out specimen.

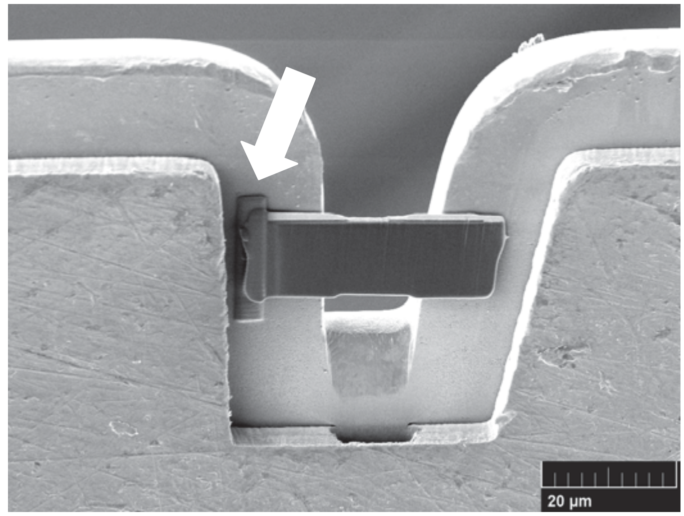

Figure 4: SEM image of the Pick&Place specimen. The arrow indicates PFIB deposited W.

At this point the specimen in figure 4 can be directly analyzed via SEM or other technique. In addition, this specimen can be further thinned to electron transparency for S/TEM via standard Ga^+ FIB methods or via Xe^+ PFIB methods [9,13]. Indeed, very large > 100 μm specimens have been prepared via *in situ* lift out and thinned to electron transparency via Ga^+ FIB, albeit at times ~ 4 hours [14]. More recently, very large > 100 μm specimens were thinned to electron transparency via Xe^+ PFIB [13]. Thus, once a specimen is manipulated to a suitable grid as in figure 4, thinning to electron transparency is routine via established methods described above.

Conclusions

Large (40 μm) PFIB prepared site specific specimens were milled, lifted out ("picked") and manipulated ("placed") quickly for direct characterization using EXLO. With conventional EXLO, thick or electron transparent specimens can be picked and placed on thin film coated grids or other appropriate supports for either S/TEM or other characterization. Pick&Place of specimens directly to EXpressLO grids enable further FIB or other processing prior to S/TEM or alternative technique. The combination of PFIB milling and EXLO increases throughput for specimen preparation.

References

[1] L.A. Giannuzzi et al., "Focused Ion Beam Milling and Micromanipulation Lift-Out for Site Specific Cross-Section TEM Specimen Preparation," Mat. Res. Soc. Symp. Proc. Vol. 480 (1997), MRS, pp. 19-27.

[2] L.A. Giannuzzi and F.A. Stevie (eds.) Introduction to Focused Ion Beams, Springer (New York 2005) pp. 201-228.

[3] L.A. Giannuzzi, "EXpressLO™ for Fast and Versatile FIB Specimen Preparation," *Microsc. Microanal.* Vol. 18, S2 (2012) pp. 632-633.

[4] L.A. Giannuzzi, "Routine Backside FIB Milling With EXpressLO™," Proc. ISTFA (2012) pp. 388.

[5] L.A. Giannuzzi, "Enhancing Ex-Situ Lift-Out with EXpressLO," *Microsc. Microanal.* Vol. 19, S2 (2013) pp. 906-907.

[6] N. Bassim, K. Scott, and L.A. Giannuzzi, "Recent advances in focused ion beam technology and applications," *MRS Bulletin* Vol. 39, No. 04 (2014) pp 317-325.

[7] F.A. Stevie and L.A. Giannuzzi, "FIB Applications: A Historical Perspective," to be published, *Microsc. Microanal*. Vol. 20, S2, (2014)

[8] Lucille A. Giannuzzi, U.S. Patents 8,740,209 and 8,789,826.

[9] L. Giannuzzi and N. Smith, "TEM Specimen Preparation with Plasma FIB Xe^+ Ions," Microsc. Microanal. Vol. 17, S2, (2011) pp 646-647.

[10] N.S. Smith, W. P. Skoczylas, S. M. Kellogg, D. E. Kinion, P. P. Tesch, O. Sutherland, A. Aanesland and R. W. Boswell, "High brightness inductively coupled plasma source for high current focused ion beam applications," *J. Vac. Sci. Technol. B* Vol. 24, No. 6, (2006) pp. 2902-2906.

[11] Paul Tesch, Noel Smith, Noel Martin, Doug Kinion, Proc. ISTFA, (2008) pp.7-13.

[12] Lucille A. Giannuzzi and Noel S. Smith, "*ex situ* lift out of PFIB prepared TEM Specimens," to be published *Microsc. Microanal*. Vol. 20, S2, (2014).

[13] A Delobbe, O Salord, P Sudraud, T Hrncir, F Lopour, and A David, "High Speed TEM Sample Preparation by Xe FIB," to be published, *Microsc. Microanal,* Vol. 20, S2, (2014).

[14] S.M. Schwarz, B.W. Kempshall, L.A. Giannuzzi, "Effects of diffusion induced recrystallization on volume diffusion in the copper-nickel system," *Acta Materialia* Vol. 51 (2003) pp. 2765–2776.