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Theory and New Applications of *Ex Situ* Lift Out

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Abstract: The *ex situ* lift out (EXLO) adhesion forces are reviewed and new applications of EXLO for focused ion beam (FIB)-prepared specimens are described. EXLO is used to manipulate electron transparent specimens on microelectromechanical systems carrier devices designed for *in situ* electron microscope analysis. A new patented grid design without a support film is described for EXLO. This new slotted grid design provides a surface for holding the specimen in place and also allows for post lift out processing. Specimens may be easily manipulated into a backside orientation to reduce FIB curtaining artifacts with this slotted grid. Large EXLO specimens can be manipulated from Xe^+ plasma FIB prepared specimens. Finally, applications of EXLO and manipulation of FIB specimens using a vacuum probe lift out method are shown. The vacuum probe provides more control for placing specimens on the new slotted grids and also allows for easy manipulation into a backside configuration.

Key words: *ex situ* lift out, FIB, TEM, specimen preparation, Pick&Place

Introduction

Background on *Ex Situ* Lift Out (EXLO)

Lift out techniques developed nearly 20 years ago have been used extensively for site specific focused ion beam (FIB) specimen preparation (Giannuzzi & Stevie, 2005). The EXLO method is historically the first lift out technique to be implemented for FIB prepared scanning/transmission electron microscope (S/TEM) specimens (Overwijk et al., 1993; Leslie et al., 1995; Herlinger et al., 1996; Giannuzzi et al., 1997) and has been applied to many different material systems (Giannuzzi & Stevie, 2005). The EXLO technique is performed in ambient conditions outside the FIB vacuum chamber using a light optical microscope and micromanipulation station. EXLO is very fast and specimens can be lifted out and manipulated for analysis in only 2–5 minutes per specimen. Greater than 90% success rates may be realized with EXLO when the specimen is correctly FIB milled free.

The basic FIB steps for successful and reproducible EXLO extraction are (Giannuzzi et al., 1997): (i) deposit a Pt (or other) ion beam deposited layer (or electron beam deposited layer followed by ion beam deposited layer) to mark the region of interest and protect the area under this layer from further FIB milling or implantation damage; (ii) FIB mill a specimen to ~1 μm thickness (or thicker) as per usual FIB methods using a large beam current (e.g., probe size) to create large trenches and successively smaller beam currents as the region of interest is approached; (iii) FIB undercut the specimen leaving one or two tabs of material attaching a part of the specimen; (iv) FIB polish the specimen faces using smaller and smaller ion beam currents (e.g., probe sizes) to remove ion implantation damage and redeposition artifacts incurred during FIB imaging and the undercut operation; and (v) achieve final thickness using the smallest beam current and pattern feasible and/or necessary to FIB mill away any tabs of material still holding the specimen. FIB milling the remaining tabs generally takes only a few seconds and will not affect the specimen surface quality if performed using smart FIB practices (e.g., using a small mill pattern and beam current), which avoid redeposition artifacts (Rajsiri, 2002; Rajsiri et al., 2002). To preserve the specimen surface quality, the tabs should never be FIB milled free with an ion beam energy greater than that used to final polish the specimen surface. It is important that the specimen be completely FIB milled free to achieve reproducible lift outs as described below. Once the specimen is FIB milled free, it will move slightly, generally remaining somewhat vertical in the trench. EXLO of specimens still attached to remaining tabs of material are possible, but more difficult, and certainly less reproducible. Specimens may inadvertently remain stuck within the trench by either (i) failing to FIB mill the tabs free, (ii) failing to completely undercut the specimen, or (iii) having sputtered material from the polishing step redeposit inside of the undercuts reattaching the specimen to the trench. To avoid redeposition issues, make sure that the

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undercuts (i) are wide enough, e.g., 1 μm or wider, (ii) mill multiple patterns to create the undercut in parallel mode, and (iii) make sure the undercut is not too close to the bottom of the trench. Lifting out "stuck" specimens requires an advanced skill level (and a bit of luck) to avoid having the specimen ricochet away.

Although electron transparent EXLO specimens are most often analyzed by S/TEM, thicker lift out specimens may be viewed by surface-sensitive techniques such as X-ray photoelectron spectroscopy or secondary ion mass spectrometry, or by SEM methods (Stevie et al., 2001; Ferryman et al., 2002; Rossie et al., 2002; Prasad et al., 2003, 2009). EXLO of site specific thick specimens optimizes imaging incident angles and analytical collection angles, which may be compromised or impossible to obtain due to confined trenches of conventional FIB milled cross sections. The speed and ease of use of EXLO is illustrated in Figure 1, which shows a series of time-stamped single-frame digital images acquired from a video of two lift outs manipulated to a 3-mm carbon-coated mesh copper grid performed in under 5 min. The arrows in the last two images in the time series in Figure 1 point to the manipulated specimens.

Adhesion Forces and EXLO

The EXLO technique relies on adhesion forces to pick up the specimen (with a glass needle pulled to ~1–2 μm in diameter) and to place it on a suitable carrier. Pulled glass needles are generally used because they are inexpensive, easy to prepare, and they remain stiff and rigid during lift out. The most common specimen carriers used are thin film coated (e.g., ~5–40 nm carbon, holey carbon, formvar, or other film) 3 mm diameter S/TEM grids where the specimen is manipulated and supported by the film for characterization. Electrostatic forces were previously believed to be the predominant mechanism for EXLO (Giannuzzi et al., 1997); however, a review of all adhesion forces shows that van der Waals and capillary forces dominate the adhesion process for a specimen particle diameter <100 μm (Zesch et al., 1997). Indeed, limiting mutual attraction electrostatic forces by, e.g., metal coating the glass probe can help the release process of a small object from the probe (Petrovic et al., 2002). Alternatively, a metal tip can directly be used for the lift out process (Petrovic et al., 2002).

Adhesion forces are greater than the force of gravity for small masses and this allows direct manipulation of FIB prepared specimens. In particular, van der Waals and capillary forces dominate in ambient conditions and capillary forces increase with an increase in relative humidity (Leite et al., 2012). All adhesion forces are proportional to the contact surface area and increase as the distance between two objects decreases. As shown below, the contact area and forces between a smooth manipulator tip and a smooth FIB milled specimen are indeed suitable for lift out. The adhesion forces associated with the lift out step are greater than any adhesion forces of the freed specimen touching its trench sidewalls as the trench surfaces are rough from FIB milling and the accumulation of redeposited sputtered material. The probe can hold the specimen indefinitely as long as it is not subjected to some other larger force such as excessive vibrations or a strong draft (e.g., via a room air/heating vent). The surface area of the carrier is much greater than the surface area of the probe, thus, manipulation to the carrier is also easily performed. Hence, the FIB milled free smooth specimen can be routinely manipulated in a site specific manner to a flat, smooth, and clean carrier device providing a strong and robust bond between the two.

A plot of adhesion forces including the force of gravity, F_g, of typical FIB specimen geometries is shown in Figure 2. It is assumed that FIB prepared specimens are rectangular with varying thicknesses creating a three-dimensional thin rectangular prism. Two different Si specimen thicknesses are assumed, 100 and 1,000 nm, both having a depth of 5 μm, allowing the specimen length to vary from 5 μm to 1 mm. The force of gravity, F_g, for the two specimen geometries as a function of specimen length is quite small and is shown in Figure 2. For comparison, the force of gravity, F_g, on a cubic block of Si with length varying from 5 μm to 1 mm obviously has a greater slope and is also shown in Figure 2. Thus, this predicts that a rectangular prism shaped specimen over 1 mm in length may be manipulated with adhesion forces.

To calculate the lift out adhesion forces, it is assumed that the glass manipulator tip is a cylinder with 1 μm diameter, the specimen is planar whose length varies as defined above, and the entire tip surface is in contact with the longest length of the specimen. In reality, the probe is conical providing even greater surface area for lift out. The adhesion forces are many orders of magnitude larger than F_g even if the probe only contacts the shortest 5 μm dimension of the specimen. The van der Waals force F_{vdw} for this cylinder plate geometry is given by the following equation:

$$F_{\text{vdw}} = \frac{LH\sqrt{d}}{8\sqrt{2}z^{5/2}}, \tag{1}$$

where L is the specimen length, H the Hamaker material specific constant, d the cylinder diameter, and z the distance between the cylinder and the specimen, which is assumed to be the distance of molecular contact = 0.2 nm (Leite et al., 2012). The Hamaker constant varies between 1×10^{-19} and 1×10^{-21} J and for dissimilar materials the Hamaker constant of each material may be estimated by $H = \frac{2H_1H_2}{H_1+H_2}$. A value of H_1 for silica–silica in air is 6.5×10^{-20} J and H_2 for silicon–silicon in air is 18.65×10^{-20} J, thus, a value of H = 9.64×10^{-20} J was used for these calculations (Leite et al., 2012). From Figure 2, F_{vdw} is more than ten orders of magnitude larger than F_g for typical specimen geometries, which clearly illustrates why EXLO is successful, reproducible, and possible.

Capillary forces arise from a liquid film between surfaces under ambient conditions and increase with humidity (Leite et al., 2012). For hydrophilic surfaces, the capillary force F_{cap} may be estimated by the following equation:

$$F_{\text{cap}} = 2\pi Ld\gamma, \tag{2}$$

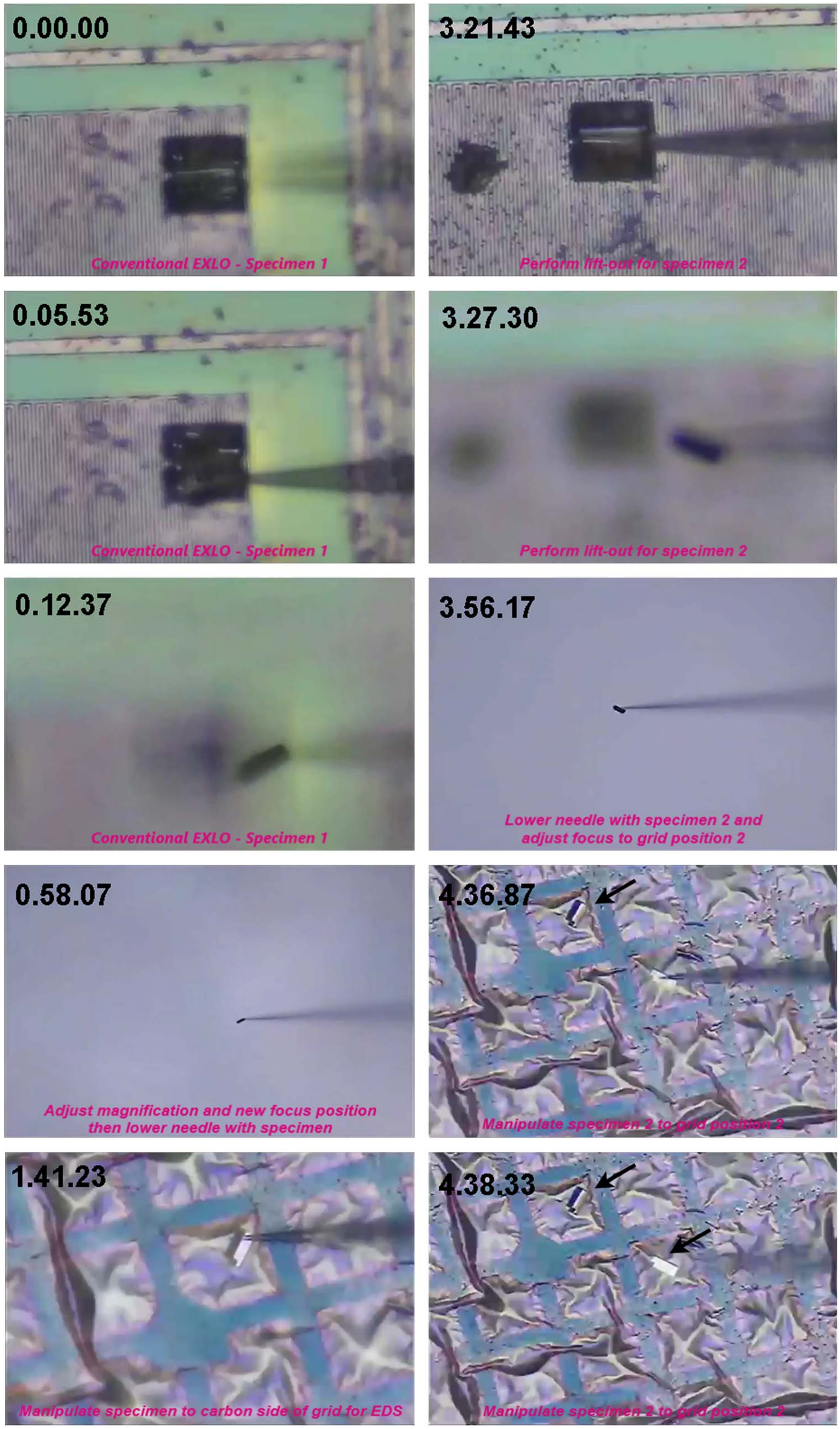

Figure 1. Still images with time stamps acquired from a video in real time showing two lift outs (shown by arrows) performed in <5 min. For reference, each specimen is ~12–15 μm long and the grid square openings are ~40 μm wide.

where γ is the surface tension of bulk water at 20°C = 7.28 N/m^2 (Adamson & Gast, 1997). Figure 2 shows that F_{cap} is also several orders of magnitude greater than F_g at these dimensions. It should be noted that even with diminished capillary effects (e.g., low relative humidity conditions common in laboratory environments), F_{vdw} by itself is quite sufficient for lift out. Note, it is well known that plasma cleaning not only removes carbonaceous contamination, but also improves the hydrophilic nature of surfaces (O'Kane & Mittal, 1974).

The electrostatic force, F_{elec}, for a cylinder-plane model where there is no charge between contacting objects is given by the following equation (Leite et al., 2012):

$$F_{elec} = \frac{L\pi\epsilon_0\epsilon_R\sqrt{RV^2}}{2\sqrt{2z^{3/2}}}, \quad (3)$$

where ϵ_0 and ϵ_R are the permittivity of free space (8.85×10^{-12} s^4 A^2/m^3 kg) and relative permittivity of air (~1), respectively, R the cylinder radius, and V the potential difference. As there is no applied potential, $V \sim 1$. F_{elec} is also plotted in Figure 2 and is the weakest of the adhesion forces in air, indicating that the EXLO process is dominated by van der Waals forces.

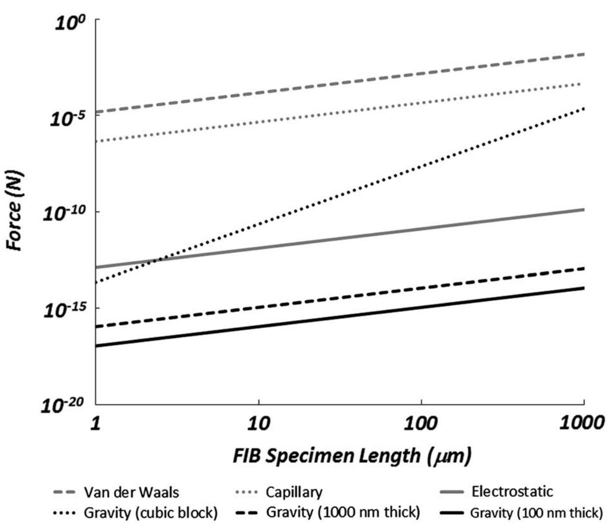

Figure 2. Graph showing adhesion forces for Si are orders of magnitude larger than the force of gravity for typical lift out specimen geometries. The rectangular prism shaped specimens are assumed to vary in length, are either 100 or 1,000 nm wide with constant depth = 5 μm.

Over the years, EXLO has worked well for many types of specimens even with the use of an insulating glass probe tip. However, there is a chance that either the insulating probe tip or the insulating or semiconducting lift out specimen can store a net electrical charge. In this case, the electrical force between the probe and specimen may dominate the adhesion forces. The electrical force may be either strongly repulsive and make it impossible for the specimen to adhere to the glass tip, or strongly attractive and make it difficult to release the specimen from the probe. To reduce any potential electrical interactions between the probe tip and the specimen, the glass probe may be metal coated (Petrovic et al., 2002).

To avoid excessive attractive forces between the glass probe tip and FIB prepared InGaAs lift out specimens, the glass probe tip was coated with a thin Au sputter coating. The glass rod was coated for ~30 s on one side and then spun ~120°, repeating the process until all sides of the probe were coated. Figure 3 shows light optical micrographs of the lift out of an FIB prepared InGaAs using the Au-coated glass probe tip. Note the probe is no longer transparent in Figure 3 due to coating (compare with Fig. 1). The specimen is lifted out in Figure 3a and manipulated to a carbon coated TEM grid in Figure 3b. The probe is released and the specimen remains stuck to the grid carrier in Figure 3c. The Au coating allows for successful and fast lift out similar to that shown in Figure 1.

Rescuing Specimens

If the specimen is not completely FIB milled free when probed, the specimen can fling away up to several millimeters from its trench. If the sample surface is polished flat or is a semiconductor or other wafer device, the specimen may often be located by carefully searching the sample surface. The specimen can be successfully manipulated from this new position. However, the first place to look when the specimen flings away is the manipulator probe itself. At times, the specimen will just climb up to a thicker part of the needle where there is greater surface area, making it nearly impossible to manipulate from this position. However, as shown in Figures 4a–4d, the specimen can be rescued and repositioned onto the probe tip for successful manipulation

Figure 3. (**a**) Lift out of a focused ion beam prepared InGaAs specimen with a gold coated glass probe tip, (**b**) manipulation to a carbon coated transmission electron microscope grid, and (**c**) release of the probe.

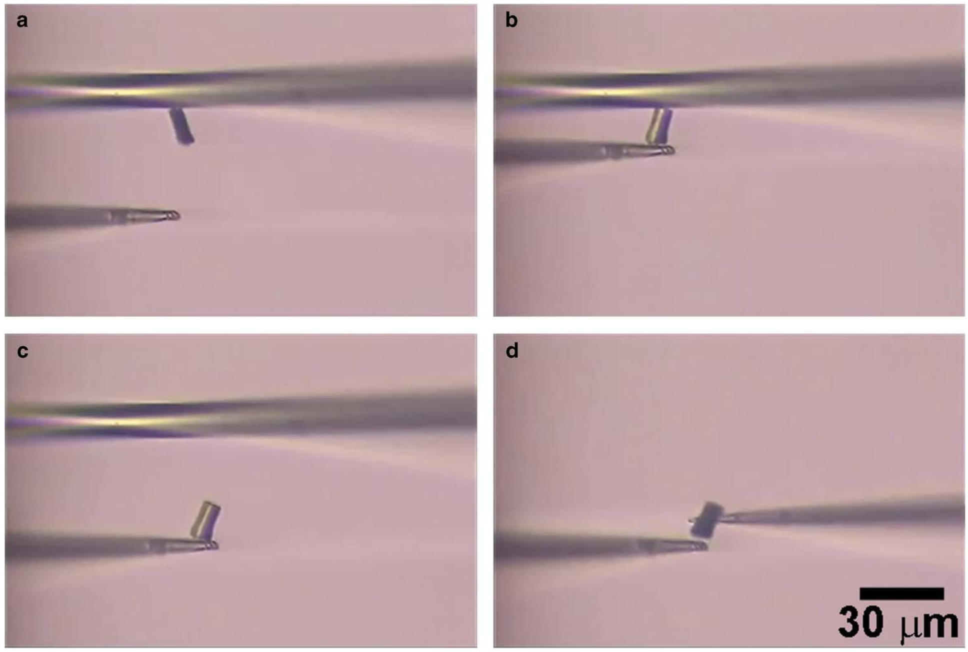

Figure 4. Light optical micrographs of (**a**) a specimen that has climbed too far up the shaft of a right-handed manipulator probe that cannot be easily manipulated to a carrier. (**b**) A second left-handed probe is used to pick the specimen from the first probe. (**c**) The specimen can be directly manipulated from the left handed probe or (**d**) transferred back to the right-handed probe for manipulation.

in about 1 min. Figure 4a shows a specimen clinging via adhesion forces to a thick portion (e.g., well up the conical shaft) of a right-handed manipulator probe. A second left-handed probe is positioned within the field of view (Fig. 4a), brought into contact with the specimen (Fig. 4b), and the specimen is transferred to the left handed probe (Fig. 4c). At this point, the specimen can be manipulated to a suitable carrier using the left-handed probe or transferred back to the right-handed probe for final manipulation (Fig. 4d). In lieu of a second manipulator, a static probe held in a small vice grip may be used for the rescue and transfer.

Specimen Preparation for *In Situ* S/TEM via Microelectromechanical Systems (MEMS) Carrier Devices

Dynamical S/TEM studies are important because phase transformations or chemical reactions may be viewed and characterized in real time. Carrier devices prepared via MEMS technology allow for *in situ* S/TEM heating (or electrical) characterization of materials (Allard et al., 2009; Young et al., 2010). These carrier devices contain electron transparent films or open regions surrounded by metal lines, which provide a source for Joule heating. Lift out for positioning on MEMS devices performed inside the FIB (e.g., *in situ* lift out (INLO)) requires multiple manipulation and FIB milling steps to ensure that a suitable region of interest is electron transparent without FIB imaging or milling the MEMS support device (Duchamp et al., 2014). As FIB imaging and ion beam deposition methods are necessary during INLO, the specimen is always lifted out "thick" and then final FIB polished *after* manipulation to a grid carrier. Conversely, EXLO of site specific and electron transparent FIB prepared specimens lends itself nicely for the precise placement of specimens onto these delicate MEMS devices (Bassim et al., 2014; Harlow et al., 2014) and examples will be described below.

Site Specific Plasma FIB Specimen Preparation

In the past few years, much interest has turned to development of different FIB sources for applications with different ions and varying ion beam currents (Smith et al., 2014). Giannuzzi & Smith (2011), and later Delobbe et al. (2014) used the Xe^+ plasma focused ion beam (PFIB) to create electron transparent S/TEM specimens. The higher mass Xe^+ ions are stopped closer to the specimen surface, and therefore, creates shallower ion implantation surface damage compared with Ga^+ ions at similar energy (Giannuzzi & Smith, 2011; Kelly et al., 2013). For applications in specimen preparation, the available beam currents in the PFIB enables site specific large area milling by inert ions not feasible with Ga^+ FIB (Tesch et al., 2008; Jiruse et al., 2012). Thus, the PFIB can create large area site specific specimens, which can be lifted out and manipulated via EXLO as described in detail below (Chan et al., 2014; Giannuzzi & Smith, 2014).

Design of a New Specimen Support Grid

Despite the well known advantages of EXLO, which include speed, ease of use, and reproducibility, recent generations of

FIB users are unfamiliar with EXLO due to perceived disadvantages (Giannuzzi & Stevie, 2005). In particular, the thin film supporting the electron transparent specimen can hinder some S/TEM imaging and analytical work such as electron energy loss spectroscopy, energy filtered TEM, electron holography, high resolution S/TEM, or energy dispersive spectrometry. The thin support film also makes it very difficult to plasma clean or re-thin the specimen (e.g., if the specimen is dirty or too thick) without also removing the support film, thereby losing the specimen.

There have been a few attempts of manipulating an EXLO specimen to a carrier without a support film. In one case, the carrier used was a 3 mm square mesh grid cut in half (Rossie et al., 2001). This required the use of an adhesive to attach the specimen to the square mesh support bars of the grid. Once the specimen was secured to the grid, it could be further FIB processed. In another example, INLO of an EXLO specimen was performed and then further thinned (Lee & Lee, 2005). In yet another attempt, a thick EXLO specimen was manipulated via adhesion forces to a beveled half grid (Patterson et al., 2002). After lift out, the carrier was returned to the FIB and the specimen was fixed in place using FIB deposition. A portion of the grid obscuring the line of sight for analysis was FIB milled away before FIB thinning the specimen. Removal of the blocking grid material required 20–30 min of additional FIB time before specimen FIB thinning could begin. In this case, the adhesion forces were strong enough to hold the specimen in place during transfer to the FIB. In yet another example, an EXLO specimen was manipulated directly to the surface of the grid bars of a 3 mm copper mesh grid with small 10 μm sized holes (Langford & Petford Long, 2001). In this geometry, a broad ion beam could mill only the top side of the specimen not obscured by the grid bars. The specimen was then flipped over using a micromanipulator tip so the underside of the specimen could then be ion milled. Once again, the adhesion forces were strong enough to hold the specimen in place during ion milling and TEM analysis. A grid sliced in half and then FIB processed to accept specimens has also been used to support EXLO manipulated samples (Lue et al., 2007). This technique also enabled subsequent FIB milling and manipulation without a thin support film. Ion beam deposited Pt was used to facilitate the manipulation process as well as secure the specimen to the grid. This process required several minutes of FIB time to ready the grids before EXLO.

To take advantage of EXLO speed and ease of use, and to make use of the strong adhesion forces known to hold and support a specimen on a smooth and clean grid surface, a new grid carrier with functional flexibility was designed (Giannuzzi, 2012*a*, 2012*b*, 2013, 2014*a*). The new patented EXpressLO™ carrier and method (Giannuzzi, 2014*a*, 2014*b*) does not require a support film and allows an EXLO specimen to be manipulated to a slotted half grid via adhesion forces, such that the specimen can be directly analyzed by S/TEM or other methods, or further processed by either FIB milling, plasma cleaning, or broad beam ion milling (Giannuzzi, 2012*a*, 2012*b*, 2013; Bassim et al., 2014).

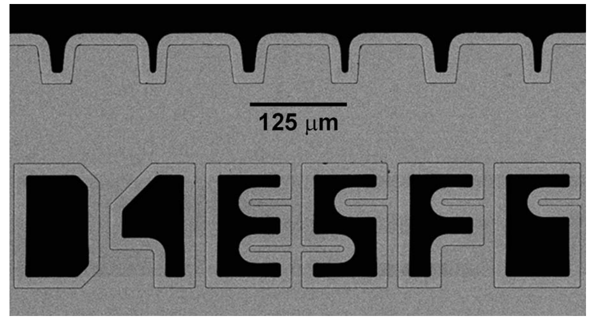

Figure 5. An SEM image of the patented EXpressLO™ slotted grid carrier (Giannuzzi, 2014*a*).

This new grid design is immediately ready for EXLO usage and does not require time consuming initial FIB preparation. Figure 5 shows an example of the slotted specimen support area. Each grid carrier is designed to accommodate several specimens of different size lift out lengths. The specimen support region is recessed below the outer edge of the grid carrier plane to protect the specimen. The smooth surfaces of the support region contribute to the adhesion forces acting on the specimen. The sidewalls of the support area could also be used to help wedge the specimen in place. To enhance adhesion and improve its hydrophilic surface, the grid should be plasma cleaned before use (O'Kane & Mittal, 1974). An adhesive could be used in this support area to hold the specimen in place (Rossie et al., 2001; Giannuzzi, 2014*b*), but is generally not needed. An electron transparent specimen centered across the slot has unobscured line of sight for S/TEM analysis (Giannuzzi, 2013). A thick FIB lift out specimen can be manipulated to the grid and then further FIB milled or processed (Giannuzzi, 2012*a*, 2012*b*, 2013). Specific examples will be given below.

This new grid carrier design also allows fast and easy manipulation of thick specimens into a backside orientation (Giannuzzi, 2012*b*). This orientation is important in, e.g., integrated circuits where FIB milling curtaining artifacts of patterned metal lines or other layers can cause thickness changes in the substrate (Schwarz et al., 2003). As the specimen is rigidly affixed to a probe when lifted out inside the FIB, manipulating a specimen into a backside orientation requires two or more time consuming rotational steps of either the grid itself and/or the specimen and probe with respect to the grid via multiple deposition and mill release steps (Schwarz et al., 2003; Gazda et al., 2010). As shown below, using EXLO with this new grid design, a specimen can be directly positioned into the backside orientation in just minutes using the manipulator probe to either lean it to the appropriate side or flip it to the correct orientation.

Vacuum Grippers and Manipulators

Vacuum micropipettes with beveled or angled surfaces designed for gripping and manipulating have been used in cell physiology for decades (Brown & Flaming, 1986). Vacuum grippers have been exploited in the microrobotics industry for precise placement of micrometer sized objects (Zesch et al., 1997). The micropipette tip can be bent, tilted, or rotated to any angle of approach and/or the hole opening itself can be angled or beveled with respect to the micropipette's long axis to optimize its position with respect to the target surface for either the "pick" or the "place" step of the manipulation process (Brown & Flaming, 1986; Zesch et al., 1997; Petrovic et al., 2002). The optimum tip size and opening should scale with the object and be ~25–50% of the object size (Zesch et al., 1997). In addition, a glass micropipette may be sputter coated with a metal or a metal micropipette may be used to minimize the electrostatic adhesion force between the micropipette and the specimen (Petrovic et al., 2002). The vacuum gripper may have force sensing incorporated to monitor the pick and place steps (Petrovic et al., 2002). The micropipette stage motion and platform may be moved either manually or automatically via computer control. The micropipette motion and manipulation is generally monitored using an optical microscope system and may also have (i) a graphical user interface with incorporation of image recognition of the manipulation process using one or more cameras with multiple light sources for illuminating the region of interest from different angles, (ii) computerized micropositioning and movable microscope stage system, and (iii) manual or computer-controlled vacuum pressure (Codourey et al., 1997; Zesch et al., 1997; Petrovic et al., 2002; Chen et al., 2005).

Adhesion forces dominate once the small object is attached to the vacuum gripper. If the adhesion force between the object and micropipette is strong enough, the vacuum may be switched off during this hold phase (Zesch et al., 1997). Releasing strategies for placing the objects include pushing the object off the manipulator with a second tip or tool, stopping or reversing the vacuum pressure (e.g., with air) to help blow or push the object off the tip, and/or directly placing or scraping the specimen onto its target (e.g., as in conventional EXLO techniques) (Zesch et al., 1997; Petrovic et al., 2002). Use of a vacuum microgripper for applications in EXLO will be given below.

Introduction Summary

In this paper, methods of conventional EXLO are used to manipulate electron transparent specimens on MEMS carrier devices designed for *in situ* S/TEM analysis. A jig and new grid design without a support film is described for EXLO. Large EXLO specimens are manipulated from plasma FIB prepared specimens. Finally, applications of EXLO and manipulation of FIB specimens using a vacuum micropipette method are also described.

Materials and Methods

Unless specifically mentioned, all Ga^+ FIB specimens were prepared using either an FEI Quanta 3D, DB235, or FEI, Helios 660 FIB/SEM instrument (FEI Company, Hillsboro, OR USA). All EXLO specimens were ~15–20 μm long and FIB milled free as described in the preferred steps in the introduction above. The thickness of each specimen varied with application of interest and will be mentioned when critical to the discussion below. Each Ga^+ FIB specimen took ~20 min or less to prepare. Xe^+ PFIB specimens were prepared using either an Oregon Physics (Oregon Physics, LLC, Hillsboro, OR USA) Hyperion PFIB Column or a Tescan FERA3 FIB/SEM (Tescan USA, Warrendale, PA, USA). The PFIB Si specimens were ~40–50 μm in length and 1–2 μm in thickness and each specimen was prepared in only ~15–20 min. Thus, in the same amount of time, the PFIB produced specimens having more than twice the length of the Ga^+ FIB produced specimens. All lift outs were performed on an EXpressLO™ EXLO station in ambient conditions (i.e., outside of the FIB chamber). An FEI Tecnai F20 operating at 200 keV was used for the TEM analysis.

Results and Discussion

EXLO for Precise Specimen Placement on MEMS Devices

EXLO and manipulation to MEMS carrier devices are nearly identical in technique as conventional EXLO to thin film coated TEM 3 mm grids. For thin film coated TEM grids, care must be taken to avoid poking the probe through the film, resulting in possible loss of the specimen. Likewise,

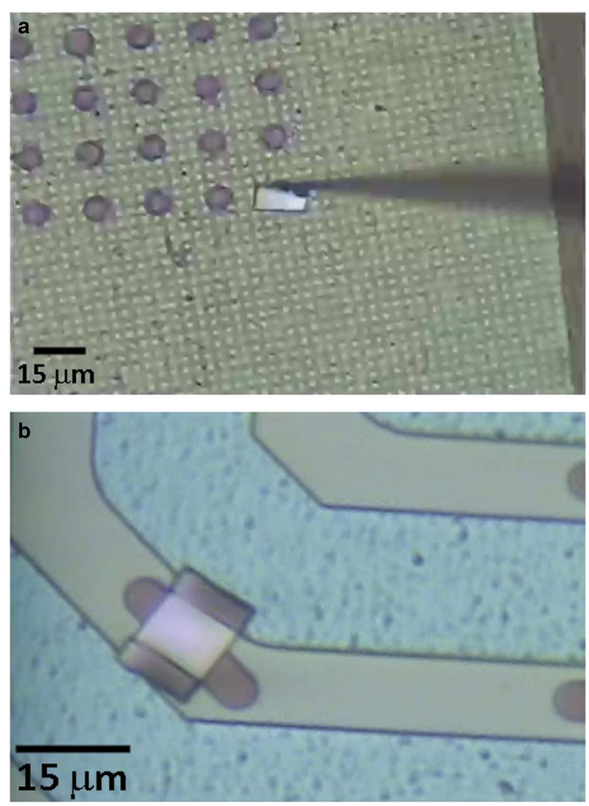

Figure 6. Electron transparent *ex situ* lift out specimens manipulated to (**a**) Protochips, Inc. MEMS carrier device and (**b**) DENSsolutions MEMS carrier device.

manipulation to MEMS devices requires similar care. Figure 6 shows electron transparent EXLO specimens manipulated to (a) a Protochips, Inc., MEMS carrier device (Protochips, Inc., Morrisville, NC USA) and (b) a DENSsolutions MEMS carrier device (DENSsolutions, Delft, The Netherlands). Conventional EXLO protocol was used and each specimen was easily centered on the window of interest while touching and adhering to the surrounding heating device via adhesion forces. Careful and delicate micromanipulation was easily performed without damaging either MEMS carrier device. Each specimen lift out and manipulation process took only 2–5 min to perform. Once the specimen is manipulated to the MEMS carrier, it can be put back into the FIB-SEM where electron beam assisted deposition can be used to secure the specimen to the carrier to prevent drift during heating (Harlow et al., 2014).

EXLO for Manipulation of Large PFIB Specimens

As mentioned above, a wide range of characterization techniques may be applied to site specific lift outs placed on suitable carriers. The Xe^+ PFIB can prepare much larger specimens than the Ga^+ FIB in the same amount of time or less. Figure 7a shows an SEM image of a ~40 μm long Xe^+ PFIB prepared EXLO specimen that has been completely FIB milled free. Figure 7b shows the same PFIB specimen after it was manipulated via EXLO to a formvar coated TEM grid. The lift out process and manipulation was performed in <2.5 min. This specimen can now be directly analyzed by any number of techniques.

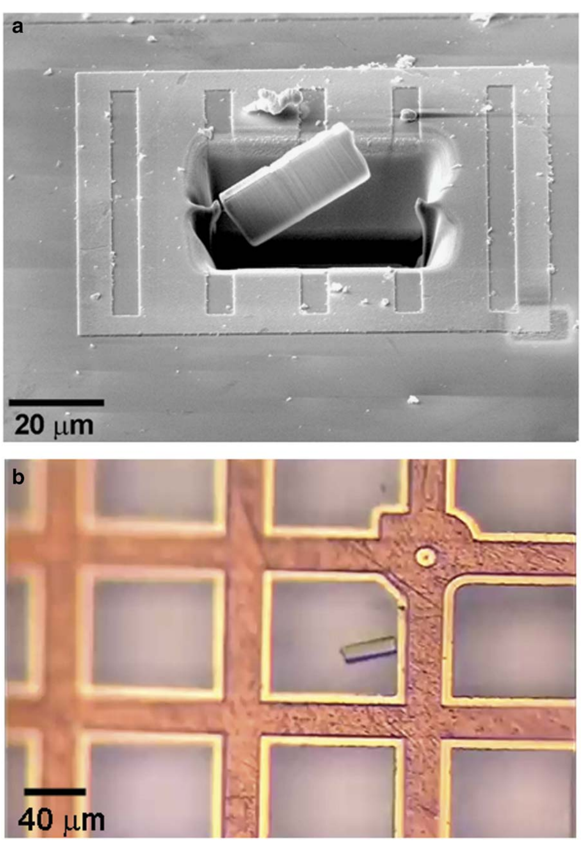

Figure 7. (**a**) Scanning electron microscope image of large (~40 μm) plasma focused ion beam EXLO prepared specimen and (**b**) after manipulation to a formvar coated copper transmission electron microscope grid.

EXLO to a New EXpressLO™ Slotted Grid

EXLO of Electron Transparent Specimen to EXpressLO™ Grid

Figure 8a shows an electron transparent specimen after lift out and manipulation to the patented EXpressLO™ slotted grid. The specimen clings to the grid support surface via the adhesion forces discussed above. This specimen was lifted out and manipulated to the grid in ~2.5 min. For manipulation to the slotted grid after lift out, it is actually beneficial to rotate the probe with the specimen attached such that the specimen is positioned on top of the probe. Conversely, for conventional EXLO, it is best to have the specimen positioned underneath or

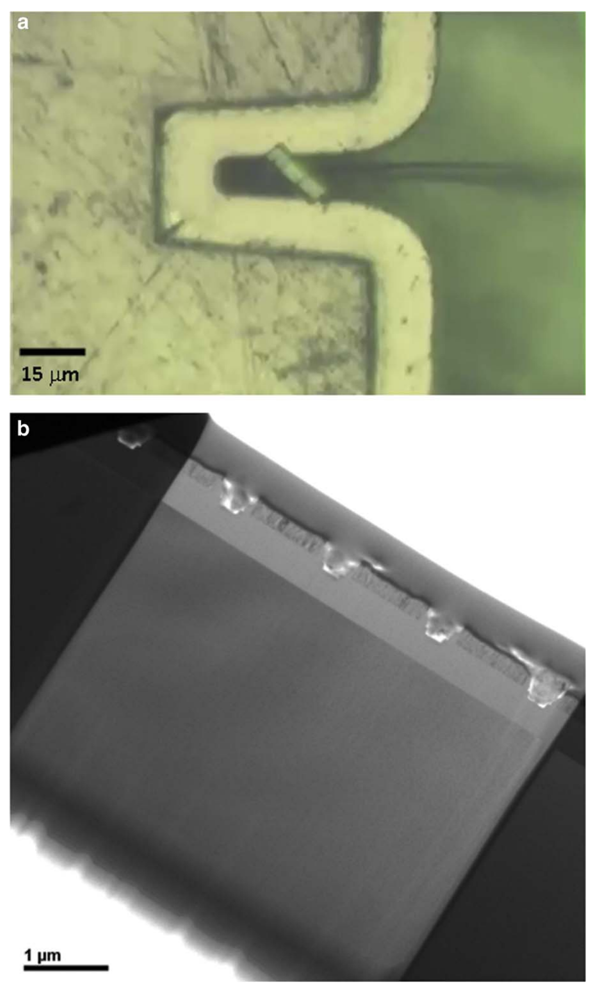

Figure 8. Use of the EXpressLO™ grid for electron transparent specimens (**a**) manipulation of an electron transparent specimen directly to the slotted grid and (**b**) corresponding transmission electron microscope image.

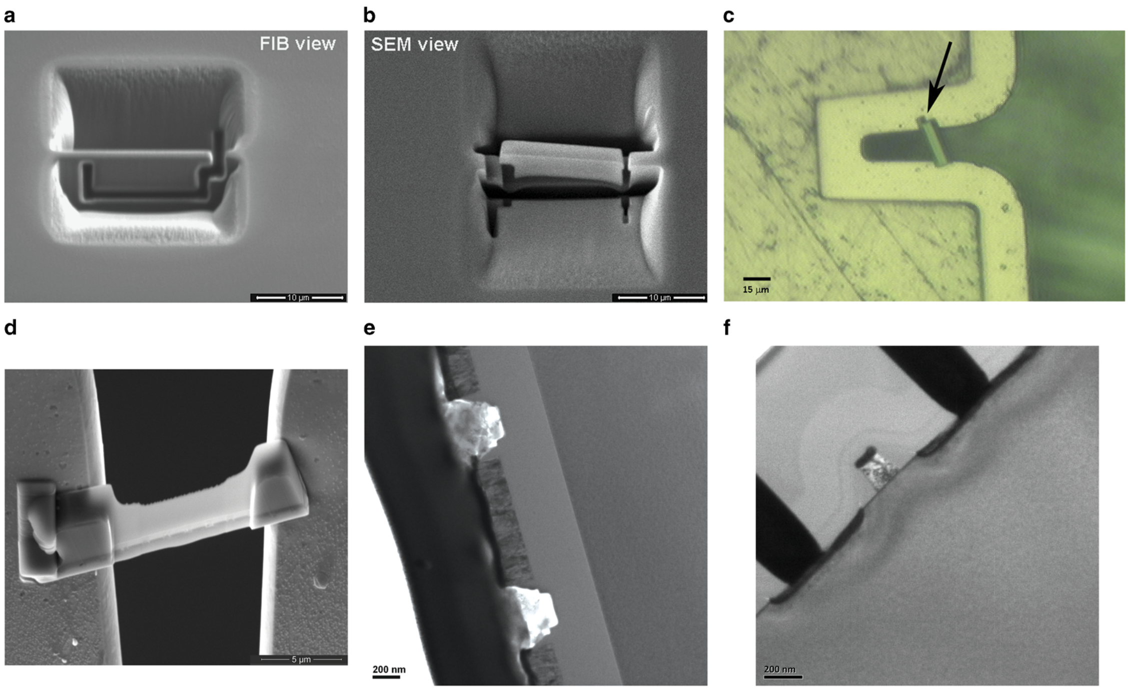

Figure 9. Process for backside manipulation and reducing FIB curtaining (**a**) perform asymmetric FIB undercut, polish sides and (**b**) FIB mill free, (**c**) perform *ex situ* lift out and manipulate into a backside orientation using the asymmetry (see arrow) to position correctly, (**d**) insert specimen and grid into FIB, secure specimen with FIB-deposited coating and FIB mill as usual to achieve desired thickness, including any low-energy FIB milling, (**e**) transmission electron microscope (TEM) image without curtaining artifacts (**f**) another TEM example without curtaining artifacts.

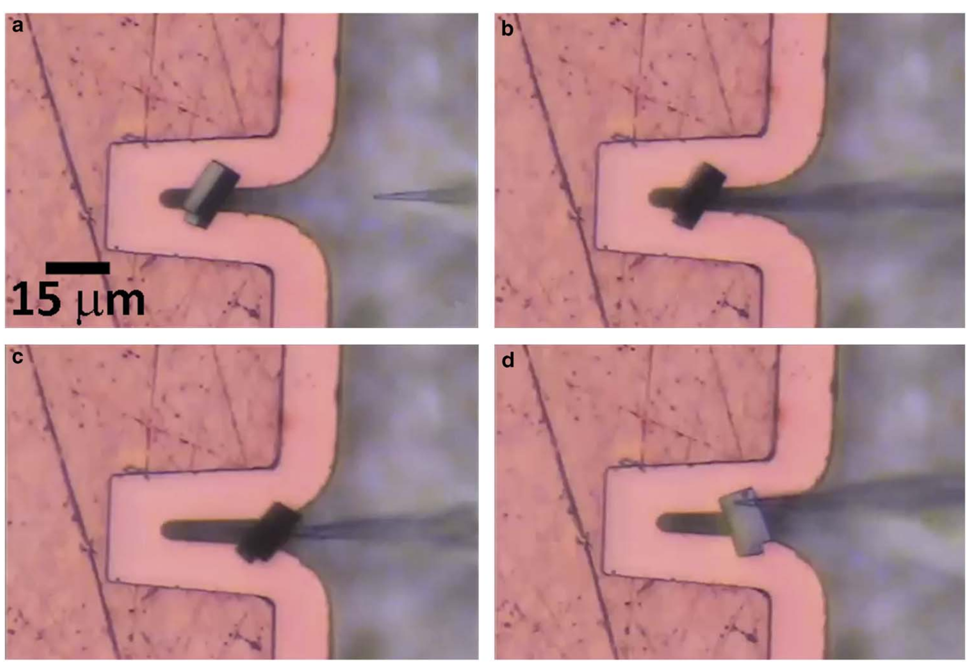

Figure 10. Flip a specimen into the backside orientation when (**a**) the specimen is not flat and its orientation is incorrect. (**b**) Pick the specimen with the probe and (**c**) flip over. (**d**) Gently tap the corner of the specimen to ensure it adheres to the grid.

hanging from the side of the probe so that it can be positioned or dropped onto the film. With the specimen on top of the probe, the probe may be lowered through the open slot, allowing the specimen to come to rest on the grid surface. If the specimen does not lie flat, the probe can be positioned to gently tap the top or corner of the specimen until it does lie flat on the grid support surfaces. The specimen is flat when the smooth FIB specimen surface shines brightly as imaged with the light optical microscope. Adhesion to the slotted grid surface may be checked by lightly touching the specimen corner with the probe ensuring that the specimen is indeed stuck to the grid. The electron transparent specimen on the grid was transferred to the TEM and imaged as shown in the low magnification image in Figure 8b. Note that curtaining artifacts are observed in the substrate, particularly evident by the variation of mass thickness contrast at the bottom of the specimen. Avoiding curtaining will be discussed below. Thus, this method provides a way for FIB specimens to be prepared and analyzed very quickly using EXLO, without the presence of a support film. In addition, re-thinning is always possible after manipulation to the slotted grid as described in the next section. These are important features realized in previous reports (Langford & Petford-Long, 2001; Rossie et al., 2001; Patterson et al., 2002; Lee & Lee, 2005; Lue et al., 2007).

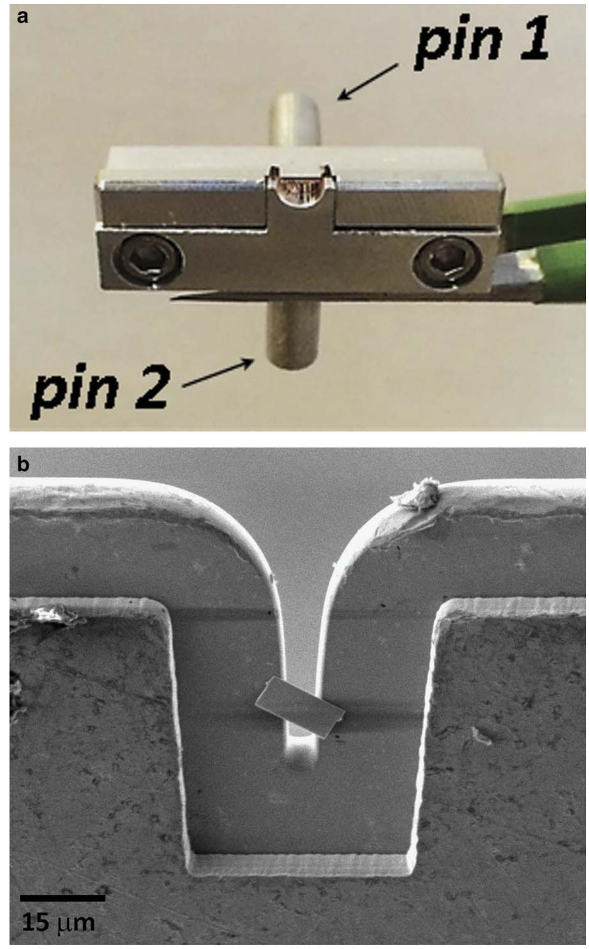

Figure 11. (**a**) Dual pin Pick&Place™ holder for EXpressLO™ manipulation (via pin 1) and additional FIB milling (via pin 2), (**b**) scanning electron microscope image of 1 μm thick specimen manipulated to the new slotted grid using the Pick&Place™ holder.

Manipulation for Backside FIB Milling Using the EXpressLO™ Grid and Method

This example is from the same sample type shown in Figure 8 so that a direct comparison can be made between top-down and backside milling. For manipulation into a backside orientation (i.e., substrate toward the FIB), it is recommended to perform an asymmetric FIB undercut as shown in Figure 9a such that the specimen orientation can be readily identified during manipulation under the optical microscope. Both specimen surfaces can be FIB polished as before and the last bit of material can be FIB milled free as shown in the SEM image in Figure 9b. Note that in this case, the specimen was FIB milled only to ~1,500 nm thick so that final FIB milling can be performed from the backside after lift out to avoid curtaining artifacts. EXLO was performed as usual and the probe was used to lean the specimen in the appropriate backside orientation using the asymmetry of the undercut as a visual guide (see arrow in Fig. 9c). The lift out and manipulation to the grid into a backside orientation took ~2.5 min. The grid supporting the specimen was inserted back into the FIB and the specimen was secured with a FIB deposited coating (see Fig. 9d). FIB milling was then performed as usual to achieve the desired thickness as per Figure 9d. Note that in this case, a FIB deposited protective coating was not used on the backside of the specimen as the Si substrate itself was used as a sacrificial layer during FIB milling. Note that any low-energy milling, broad beam ion milling, or plasma cleaning can be performed after this step. This specimen was then imaged in the TEM and Figure 9e shows no curtaining artifacts in the Si substrate. A second specimen of a different sample type was prepared in this backside manner and the corresponding TEM image is shown in Figure 9f with no evidence of curtaining and only the presence of diffraction contrast in the Si substrate.

As mentioned, the asymmetric undercuts help to identify the specimen orientation. If upon initial manipulation, the specimen position is deemed incorrect (or not flat and secure) as in Figure 10a, the manipulator probe can be used to flip it into the correct backside orientation as shown in Figures 10b–10d. The probe was used to grab the specimen as in Figure 10d and flip the specimen over as in Figure 10c. Note that the “dark” contrast from the specimen indicates that it is not flat on the grid. The probe was used to gently tap the corner of the specimen to ensure it adhered to the grid as in Figure 10d. The additional bright contrast observed in Figure 10d also indicates that the specimen is flat.

Design of New Pick&Place™ Holder for the EXpressLO™ Grid and Method

As discussed above, thick specimens may be manipulated to the new slotted grid (in any orientation) and returned to the FIB for final milling as usual. To facilitate the process between lift out, manipulation, and return to the FIB, a grid specimen holder was designed with dual pins at 90° to each other as shown in Figure 11. The first pin is used to secure the Pick&Place™ holder with slotted EXpressLO™ grid for sample manipulation in a planar orientation as in Figure 11a.

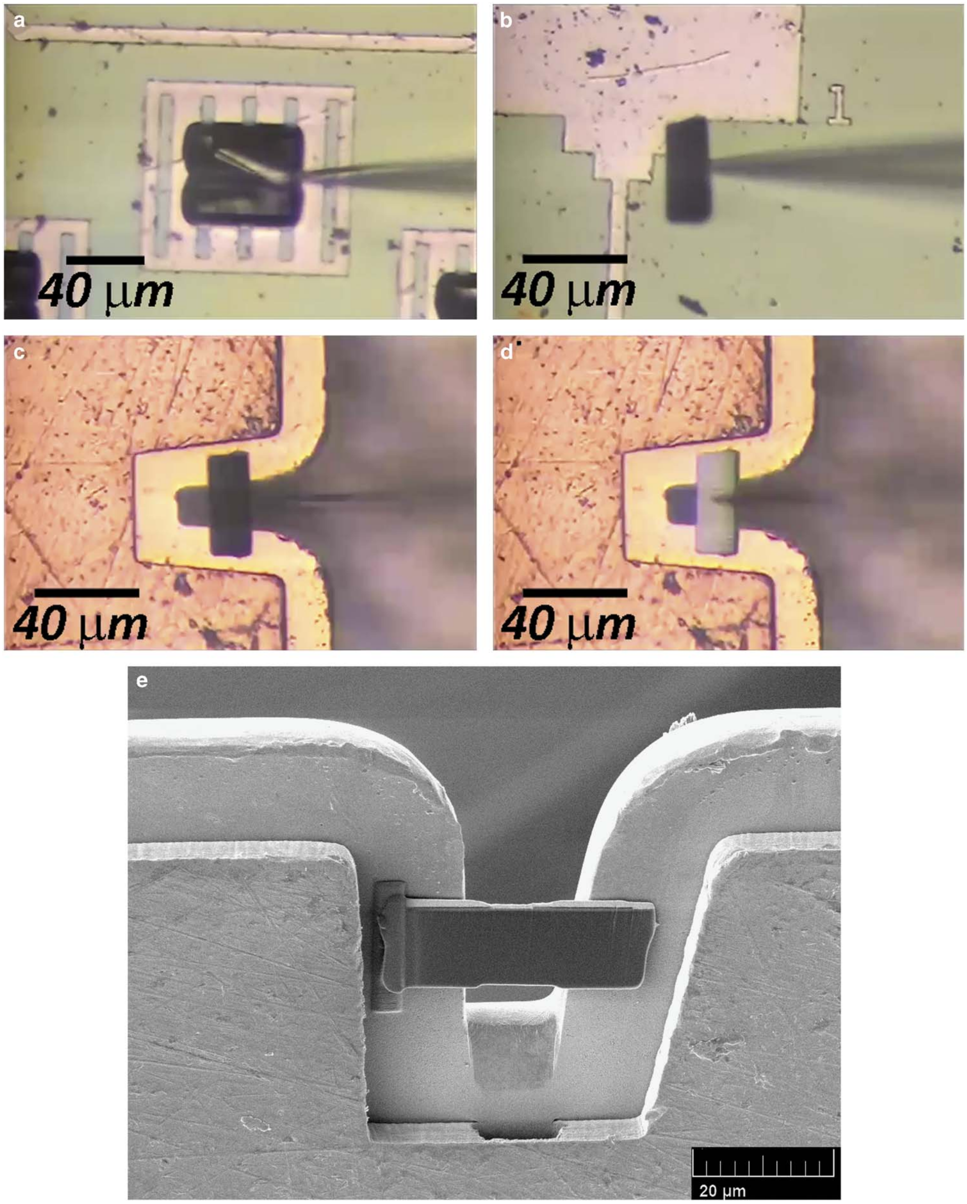

Figure 12. *ex situ* lift out of a PFIB prepared specimen to an EXpressLO™ grid. (**a**) The probe attempts to lift out a stuck specimen. (**b**) The probe picks up specimen that ricocheted away. (**c**) Manipulation of PFIB specimen to a slotted EXpressLO™ grid. (**d**) The probe gently touches top of specimen to ensure adhesion forces activate. (**e**) scanning electron microscope image of the specimen on the grid after secured with FIB deposited W.

After specimen manipulation to the grid, the holder may be moved to the FIB using the second pin to orient the grid in an upright orientation. In this manner, excess handling of the slotted EXpressLO™ grid containing the specimen is avoided. A 1 μm thick specimen manipulated to the new slotted grid facilitated by the Pick&Place™ holder is ready for additional FIB preparation as per Figure 11b.

Manipulation of PFIB Specimen to a Slotted EXpressLO™ Grid

As shown in Figure 7, large Xe^+ PFIB prepared specimens are just as easily lifted out via EXLO as Ga^+ FIB prepared specimens. The advantage is that a PFIB can prepare larger specimens faster than a Ga^+ FIB. EXLO success rates depend critically on proper FIB milling, and as stated above, specimens should be FIB milled free. As mentioned earlier, if the specimen is not FIB milled free, it can ricochet during probing. Figure 12a shows a glass probe pushing on a PFIB prepared specimen that is not properly FIB milled free. After several probing attempts, the specimen ricocheted out of the trench and landed on the surface of the wafer a few hundred micrometers away as shown in Figure 12b. This is fortuitous, as manipulation to a slotted EXpressLO™ grid is most easily performed with the specimen on *top* of the probe. (After lift out, the probe can be easily rotated to optimize specimen

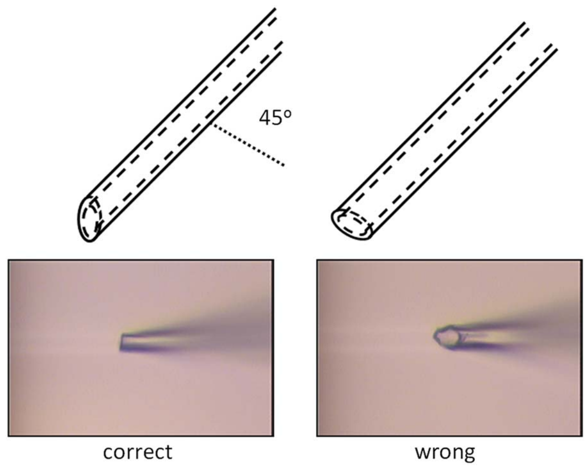

Figure 13. Correct and incorrect ("wrong") orientation of the vacuum probe tip for lift out and manipulation.

positioning before manipulation to an EXpressLO™ grid). The probe is positioned *under* the specimen for the pick and place to the EXpressLO™ grid by manipulating the probe down through the open slot, allowing the specimen to rest on the grid surface as in Figure 12c. The dark contrast of the specimen in Figure 12c indicates that it is not flat and the probe is used to gently touch the top of the specimen to ensure adhesion forces between the specimen and the grid take hold (see Fig. 12d). Lift out and manipulation of this "stuck" specimen was performed in <3 min (Chan et al., 2014). Note that manipulation to the EXpressLO™ grid is easiest and most successful when the specimen length is ≥3 × the slot width. An SEM image of the manipulated PFIB specimen after FIB deposited W applied is shown in Figure 12e. At this point, further FIB thinning using either PFIB (Delobbe et al., 2014) or Ga^+ FIB (Giannuzzi & Stevie, 2005) is routine and no different than any other usual final FIB polishing steps.

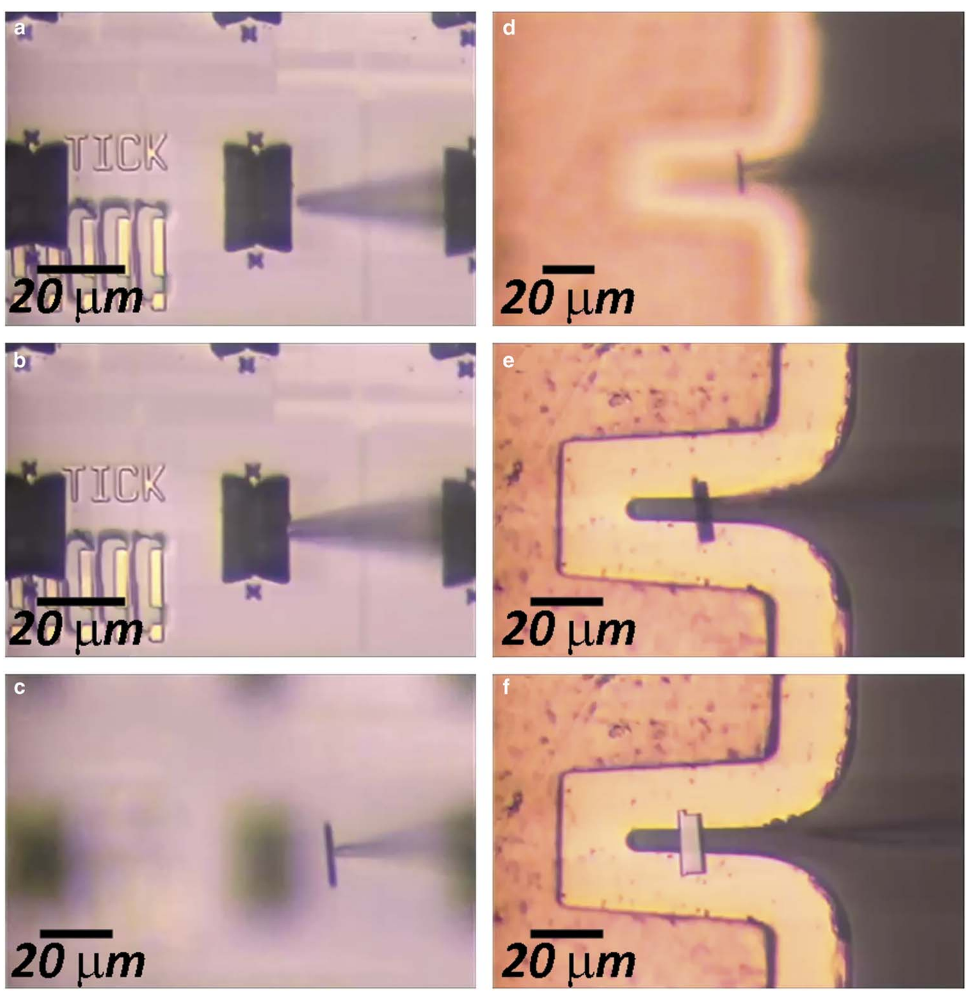

Figure 14. Vacuum lift out Pick&Place™ sequence of a FIB milled freed specimen to an EXpressLO™ grid. (**a**) The vacuum valve is opened. (**b**) The probe tip approaches the FIB milled free specimen. (**c**) The specimen is lifted out. (**d**) The vacuum valve is closed and the valve to atmosphere is opened. (**e**) The probe slides through the grid slot to position the specimen in a backside orientation. (**f**) The probe gently touches the specimen to ensure it is flat and secure on the EXpressLO™ grid and then moves away from the specimen.

Use of Vacuum Micropipette Pick&Place™ with EXpressLO™

As mentioned above, vacuum manipulation has been used for precise placement of micrometer sized objects. These same vacuum pipetting principles may be applied to lift out and manipulation of FIB prepared specimens. In this case, a hollow glass tube is pulled to a tip of about 1 μm in diameter. The end of the tip is beveled with a 45° angle via mechanical polishing or FIB milling to a long outer diameter of ~2–3 μm (i.e., 25–50% of the specimen depth). If mechanical polishing is used, the tip end should be ultrasonically cleaned in ethanol to remove any polishing debris before use. The end of the probe tip micropipette is fitted with a hose and T-valve to accommodate both vacuum and compressed air (or nitrogen). The probe is mounted at a 45° angle and attached and rotated such that the flat probe tip opening is parallel (or nearly parallel) to the face of the FIB milled free specimen. As shown in Figure 13, when the probe is rotated correctly, the optical microscope image of the probe will show a flat edge at the end of the tip perpendicular to the probe long axis with no evidence of the beveled surface observed through the transparent tip.

The vacuum lift out pick and place sequence of images are shown in Figure 14. Once the probe tip approaches within a few micrometers of the specimen, the vacuum valve is opened (Fig. 14a). The probe tip is centered and touched to the FIB milled free specimen surface enabling the specimen to cling to the probe tip via vacuum suction (Figs. 14b, 14c). The critical difference here compared with previous conventional lift out examples shown is that the specimen plane normal is coplanar with the probe axis direction (see Fig. 14c) as a direct result of the suction forces along the probe axis. This orientation is difficult to achieve using only adhesion forces. Note that the sample can be rotated to optimize the FIB mill freed specimen face with respect to the tip orientation. Once the probe tip approaches within a few micrometers of the grid surface, the vacuum valve is closed and the air valve is opened to atmosphere. At this point, just adhesion forces take over and the specimen can be released to the grid surface as usual (Fig. 14d). (Positive air or nitrogen flow may also be used to facilitate release of the specimen from the probe.) The probe is manipulated through the open grid slot such that the specimen falls to the grid surface into a backside orientation (Figs. 14e, 14f). The probe may be used as before to gently touch the specimen to ensure that it is flat and secure on the EXpressLO™ grid. The vacuum lift out pick and manipulation place process was performed in <2 min. At this point, the specimen may be put back into the FIB without directly handling the grid via the Pick&Place™ holder by securing pin 2 to the FIB stage. An FIB deposited layer can be added and then the specimen may be further thinned to electron transparency as per figure 9d.

Note that the advantage of vacuum manipulation is greater control, more accurate and reproducible specimen centering on the probe, and ease of centering and orientation of the specimen to the grid surface as the probe slides through the grid slot. As shown in the schematic images in Figure 15, the probe can be used to lean the specimen into a frontside (e.g., Fig. 15a) or backside orientation (e.g., Fig. 15b). After lift out, the probe can be tilted back from its set position of 45° to, e.g., 20° to prelean the specimen for manipulation for conventional frontside orientation as shown in Figure 15c. In addition, preleaning after lift out combined with grid rotation can also be used for backside manipulation as shown in Figure 15d.

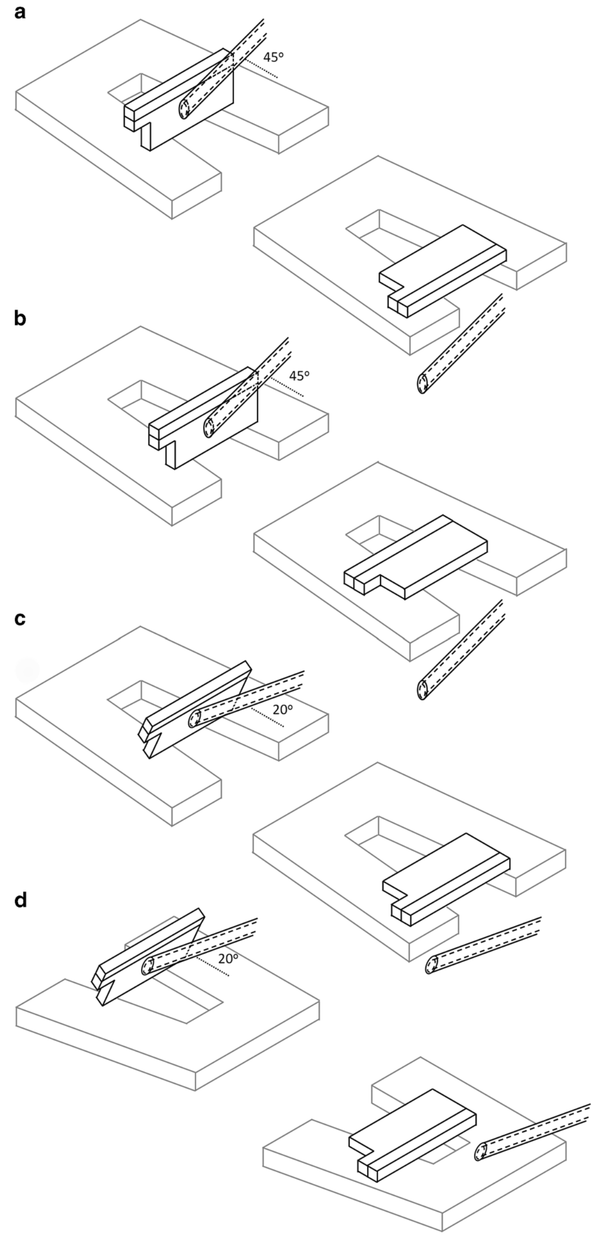

Figure 15. Schematic perspective diagram of the vacuum pick and place lift out for (**a**) conventional frontside manipulation or (**b**) backside manipulation. Facilitating the manipulation of the specimen by (**c**) preleaning the specimen *after* lift out for frontside manipulation and (**d**) combined with rotating the grid by 180° for backside manipulation.

Summary

The EXLO process and adhesion force theory have been detailed and reviewed, and new applications have been described. The adhesion force theory that governs the EXLO process shows that van der Waals forces dominate for conducting specimens. Electrostatic forces can be detrimental causing either the inability to lift out insulating specimens due to large repulsive forces, or the inability to release insulating specimens due to large attractive forces. Electrostatic forces can be negated by using a metal tip or applying a metal coating to the glass probe. EXLO is fast, easy, and reproducible, and is suitable for lift out of site specific electron transparent specimens to MEMS carrier devices designed for *in situ* S/TEM studies. A new slotted grid carrier was developed that negates the need for a thin film support. This new EXpressLO™ grid allows for manipulation of thin or thick specimens to the carrier. The specimens can be FIB processed after manipulation to the new grid. The new grid geometry and lift out process also enables easy manipulation of specimens into a backside orientation, which reduces curtaining during subsequent FIB milling. A new dual pin grid holder also facilitates lift out, manipulation, and further FIB milling of specimens without excessive grid handling. EXLO was used to lift out large PFIB prepared specimens and manipulated to carbon coated TEM grids and to EXpressLO slotted grids. Adhesion theory suggests that ultra large (>1 mm) specimens may be lifted out for site specific analysis. Vacuum manipulation methods similarly used in biological and nanorobotic pipetting techniques have been incorporated into EXLO with the new EXpressLO™ slotted grid. The vacuum lift out method provides easier centering of the probe with respect to the specimen for easy positioning of the probe through the open grid slot.

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