Using the *in situ* lift-out technique to prepare TEM specimens on a single-beam FIB instrument

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**Abstract.** Transmission electron microscope (TEM) specimens are today routinely prepared using focussed ion beam (FIB) instruments. Specifically, the lift-out method has become an increasingly popular technique and involves removing thin cross-sections from site-specific locations and transferring them to a TEM grid. This lift-out process can either be performed *ex situ* or *in situ*. The latter is mainly carried out on combined dual-beam FIB and scanning electron microscope (SEM) systems whereas conventional single-beam instruments often are limited to the traditional *ex situ* method. It is nevertheless desirable to enhance the capabilities of existing single-beam instruments to allow for *in situ* lift-out preparation to be performed since this technique offers a number of advantages over the older *ex situ* method. A single-beam FIB instrument was therefore modified to incorporate an *in situ* micromanipulator fitted with a tungsten needle, which can be attached to a cut-out FIB section using ion beam induced platinum deposition. This article addresses the issues of using an ion beam to monitor the *in situ* manipulation process as well as approaches that can be used to create stronger platinum welds between two objects, and finally, views on how to limit the extent of ion beam damage to the specimen surface.

1. Introduction

Preparation of transmission electron microscope (TEM) specimens using focussed ion beam (FIB) instruments offers the possibility of producing site-specific specimens from a wide range of different materials [1, 2, 3]. The lift-out method is one FIB technique which recently has become increasingly popular, mainly due to the relatively short times required to produce high-quality TEM specimens. Principally, this method can be divided into *ex situ* and *in situ* lift-out. Both involve the use of a fine needle attached to a micromanipulator to pick up a cut-out cross-section from a bulk sample in order to transfer it to a TEM grid.

The *ex situ* technique was the first procedure to be developed. It utilises an optical microscope and a glass needle to perform the lift-out. Consequently, the method suffers from a number of drawbacks which are related to the use of mere electrostatic forces to attract and keep the TEM section attached to the lift-out needle whilst transferring it from bulk sample to grid. These forces are often too weak and unpredictable to constitute a reliable means of performing the lift-out. Moreover, the use of an optical microscope limits the image resolution and depth of field which adds to the difficulty of exercising this method.

The other process, called the *in situ* technique, is performed inside the FIB instrument by using a built-in micromanipulator. The major deficiencies of the *ex situ* technique are eliminated since the...
A manipulator needle is welded onto the specimen using ion beam induced platinum (or tungsten) deposition. A similar welding process is also performed to attach the section to a TEM grid. This increases both the physical strength of the connections as well as the overall reliability of the lift-out concept.

However, this relatively new method tends to be more commonly used on modern dual-beam systems which are combined FIB and scanning electron microscope (SEM) instruments offering the possibility of simultaneous electron beam imaging and ion beam milling [4, 5]. Such versatility is useful when performing in situ manipulation since the process needs to be closely monitored, and should ideally be done so without damaging the surface.

Modifying existing single-beam instruments to enhance their capabilities is nevertheless a desirable prospect as it would allow older FIB systems to take advantage of the benefits associated with the in situ technique. An FEI FIB 200 single-beam workstation using a 30 keV gallium liquid metal ion source (LMIS) and a platinum gas injection system (GIS) was therefore modified and fitted with a Kleindiek piezo-electric micromanipulator to allow specimen lift-out to be performed in situ.

This article will address some important aspects on how the lift-out procedure can be performed to ensure a high degree of success when transferring sections from bulk sample to TEM grid. The emphasis is on the use of single-beam instruments although the methods which are discussed are also applicable to dual-beam systems.

2. Method

The main concern when performing in situ lift-out is how to safely manoeuvre the needle into position, both when picking up a section and when transferring it to the TEM grid. This will be described, together with a way to produce secure welds by using ion beam induced platinum deposition. There are also concerns regarding the use of single-beam instruments with respect to surface damage since the ion beam offers the only source of imaging. Simple measures on how to limit the extent of damage will therefore be discussed in brief.

2.1. In situ manipulation

The process of manoeuvring the lift-out needle into position is supervised in real time through continuous ion beam induced secondary electron (ISE) imaging. The beam is focussed on the top surface of the object of interest, i.e. the cross-section or the TEM grid, which is maintained at eucentric height. The tip of the manipulator needle is typically located a few millimetres above the object and will appear out of focus. This difference in focus is consequently used as a guide to the relative difference in height between needle and object.

The needle can be moved relatively quickly down towards the object until it is almost perfectly in focus. At some point, however, when the needle is close to the object surface, it becomes difficult to qualitatively judge the difference in focus. Fortunately, a local change in image contrast appears shortly before contact is established and can be used to assist final movements. This local contrast change can be seen as ‘shadowing’ taking place at the object surface as the needle approaches. The position of the needle can thus be tuned according to the ‘shadow’ that forms before contact.

The ‘shadow’ is related to a local decrease in the amount of ISEs that are detected due to the obstruction caused by the manipulator needle. This effect has proven highly useful since the ‘shadow’ tends to move and those movements can be correlated with the difference in height between needle and object. The point where contact will be made can therefore be predicted by monitoring this ‘shadow’.

Figure 1 illustrates the in situ lift-out process through a sequence of images. The sequence starts with manoeuvring the lift-out needle into position, to the side of a pre-milled section, which is only attached to the bulk sample at one point. The pre-milling process is similar to the one used for ex situ lift-out. The procedure is therefore shown from the point where it starts to differ from ex situ lift-out.
Figure 1. Lift-out sequence showing three consecutive steps; (a) the needle has been manoeuvred into position using difference in focus and the ‘shadow’ (arrow 1) which forms due to a local decrease in the amount of ISEs reaching the detector (arrow 2); (b) the needle has been welded to the section (arrow 1) and a cut has been made to detach the section from the bulk sample (arrow 2); (c) the sample stage has been lowered to move the section away from the bulk sample.

2.2. Creating platinum welds

Physical welds are created by depositing a layer of platinum (or tungsten) onto two connected objects. The strength of the weld can be related to the area of deposited platinum that is in contact with both of these two objects. Use of blunt lift-out needles (see figure 1) therefore has the advantage of offering a larger contact area between the two objects which results in more secure welds. Another benefit of this is the significant reduction in specimen preparation time that is achieved when the time consuming process of re-sharpening the needle between each lift-out can be avoided.

However, it is also important that the surface where deposition is to be carried out is reasonably flat. It has been found that this has a major influence on the quality of the weld. The deposited layer can otherwise fail to attach properly to both of the joined objects. This is illustrated in figure 2 which shows the importance of attaching the lift-out needle in such a way that a flat welding surface is obtained.

Figure 2. Schematic illustration showing the importance of having a reasonably flat welding surface where (a) shows a manipulator needle attached to the top of an object resulting in a principally weak weld since the platinum tends to deposit primarily on the upper part. A stronger weld is obtained in (b) since the positioning of the needle results in a flatter welding surface.
It can be seen in the first image (figure 2a), where the needle is placed on top of the object surface, that the deposited platinum layer forms primarily on the top part and then continues downwards in a stepwise manner. This is a typical phenomenon which has been frequently observed. The lower part will consequently have a very limited surface area in contact with the platinum layer, resulting in a weak weld. It should be noted, however, that needles can be successfully welded onto the top surface (as in figure 2a) if the tip is sufficiently sharp. This would nonetheless require the tips to be re-sharpened by FIB milling or electropolishing between every lift-out operation.

Welding specimens onto TEM grids requires a similar approach. The key is to avoid a connection where one part is situated higher up than the other. That could otherwise create a problem similar to figure 2a where the material is primarily deposited on one side. Similarly, joining two objects having sloping sides can sometimes leave a gap between them. Such structures can be welded together by first ‘filling’ the gap with platinum before a second layer is deposited across the two objects. The purpose of the first weld is simply to create a flatter surface onto which the second major weld can be deposited.

2.3. Minimising surface damage

It is well known that ion beam induced imaging causes surface damage in form of ion implantation and surface amorphisation [6]. The extent of this can, however, be limited by implementing some protective measures. Firstly, covering bulk samples with protective coatings ensures that some degree of imaging can be carried out without damaging the original surface. Secondly, cross-sections which are significantly thicker than the final thickness can be cut out and transferred to a grid. This enables damaged layers to be removed during the final stages of ion beam polishing, when the section is thinned to electron transparency. And finally, imaging and polishing should be exclusively performed perpendicular to the foil thickness during final thinning since this decreases the degree of surface damage [7]. Imaging the section from the sides is only carried out when its thickness exceeds the depth of beam induced surface damage.

3. Conclusions

The main weakness of single-beam instruments is their inability to image an object without causing surface damage. The damage caused by ion bombardment can, however, be reduced by protecting bulk samples with surface coatings and by avoiding direct imaging of the sides of TEM cross-sections during the last stages of thinning. Another limitation is the lack of simultaneous imaging and milling which can serve as a valuable monitoring tool during critical operations. Nevertheless, single-beam instruments can be used to rapidly and consistently produce in situ lift-out specimens with minimum surface damage if the appropriate techniques are used.

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References

[1] Mayer J, Giannuzzi L A, Kamino T and Michael J 2007 MRS Bul. 32 400–7
[2] Giannuzzi L A and Stevie F A 1999 Micron 30 197–204
[3] Li J, Malis T and Dionne S 2006 Mat. Char. 57 64–70
[4] Langford R M and Clinton C 2004 Micron 35 607–11
[5] Sivel V G M, van den Brand J, Wang W R, Mohdadi H, Tichelaar F D, Alkemade P F A and Zandbergen H W 2004 J. Microsc. 214 237–45
[6] Volkert C A and Minor A M 2007 MRS Bul. 32 389–99
[7] Prenitzer B I, Urbanik-Shannon C A, Giannuzzi L A, Brown S R, Irwin R B, Shofner T L and Stevie F A 2003 Microsc. Microanal. 9 216–36