

# Increasing sample preparation throughput





Micromanipulator fitted with a glass needle for ex-situ lift-out

### Introduction

Sample preparation is a necessary prerequisite for transmission electron microscopy (TEM). The advent of focused ion beam (FIB) workstations for the preparation of electron transparent lamellae has revolutionized TEM specimen preparation. An advanced analytical tool combined with a high-

precision nanomanipulation system gives you the benefit of sample preparation techniques that are faster, more accurate, more reproducible and more reliable.

The modern method for the preparation of a TEM specimens is to use a FIB to cut a thin lamella out of the bulk material. The transfer or "lift-out" of such a lamella from the bulk material to the TEM grid requires a high-precision manipulation system and a reliable process so that the lamella is not lost. This lift-out process can either take place ex-situ under a light microscope or in-situ in the FIB.

### Ex-situ lift-out

For the ex-situ procedure, specially prepared glass needles are used. Adhesive forces ensure that the lamella remains attached to the needle and the C-film on the TEM grid. This process requires a lot of operator experience and is not very reliable. The success rate is reported to be in the range of 90% by experienced operators.

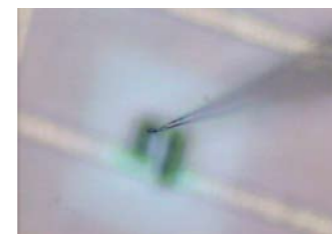
### In-situ lift-out

For the in-situ process, electron or ion beam induced material deposition (EBID) is used to attach the TEM lamella to a tungsten needle and finally to the TEM grid.

### The current in-situ lift-out situation

Conventional procedures for FIB prepared TEM lamellae involve the following steps:

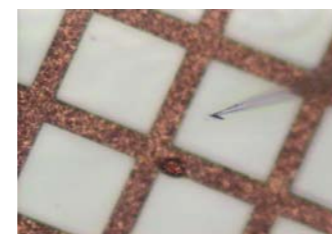
- Deposit a protective layer over the area of interest using electron or ion beam induced deposition (EBID)
- Mill both sides and pre-cut the membrane using the ion beam
- Attach the manipulator tip to the pre-cut specimen using EBID
- Free the specimen from the bulk sample using the ion beam
- Lift out the specimen with the manipulator and transport it to the TEM grid



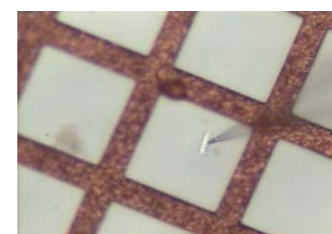
Ex-situ lift-out of the lamella



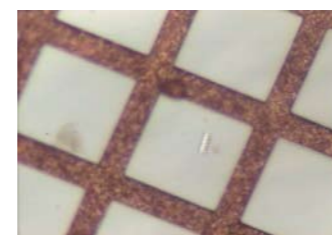
Adhesive forces hold the lamella to the glass needle



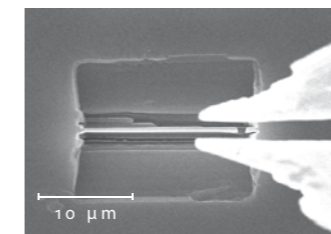
Transfer of the lamella to the TEM grid



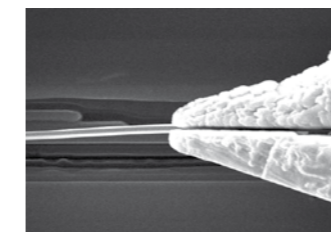
Placing the lamella on the C-film



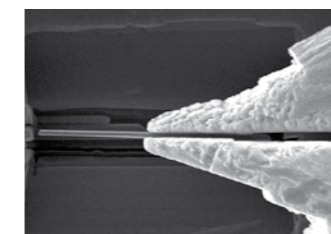
Extracted lamella on the TEM grid



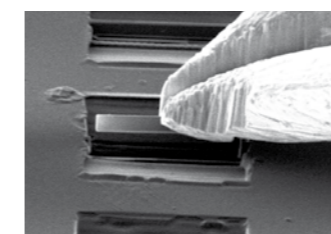
Grip the specimen using the microgripper



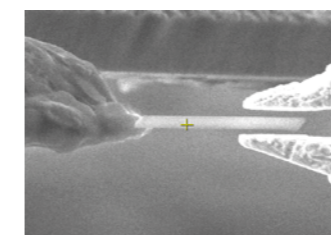
Free the specimen by moving it horizontally back and forth a few times



Specimen can also be freed using a cut from the ion beam



Lift out the specimen



Transport and attach the specimen to the TEM grid

- Attach the specimen to the TEM grid with EBID
- Free the specimen from the manipulator tip using the ion beam
- Reprocess the specimen with the ion beam to produce the TEM lamella
- Reshape and clean the manipulator tip for re-use by using the ion beam or vent the chamber to replace the manipulator tip



Micromanipulator fitted with a microgripper for in-situ lift-out

This process is more reliable than the ex-situ lift-out process, but it is considerably more expensive and requires lots of valuable time on the FIB. Time and cost factors call for a faster, simpler procedure without reducing the accuracy and reliability of the results.

### New methods

Increased sample preparation throughput is possible by introducing tools that drastically accelerate various steps in the conventional procedure. Instead of relying on time-consuming EBID and ion beam cutting, a high-precision microgripper can be mounted to the manipulator and used to grip, lift out, transport and release the milled specimen.

A fast, simple and reliable lift-out procedure using the MM3A-EM micromanipulator fitted with the MGS2-EM microgripper involves the following steps:

- Grip the specimen using the microgripper
- Free the specimen from the bulk sample by moving the microgripper horizontally back and forth a few times
- Lift out the specimen with the manipulator and transport it to the TEM grid
- Attach the specimen to the TEM grid without using EBID by simply focusing the ion beam in and out on the desired connection point
- Release the specimen

Due to the absence of EBID and ion beam cutting the microgripper is not contaminated or deformed in any way. Reshaping or cleaning is not required and the chamber does not need to be vented if the sample is removed via the load-lock.

### STEM analysis

In many FIB systems an electron column is available, allowing a secondary electron SEM image for the visual control of the prep-



Micromanipulator fitted with a rotational tip

aration process. Optionally, scanning transmission electron microscopy (STEM) detectors can be fitted, allowing the creation of STEM images either provide the final information required about the TEM specimen

or can be used to estimate the thickness of the lamella. Lamellae that are too thick can be immediately reprocessed in the FIB.

The process of ion beam thinning (in particular backside milling), positioning above the STEM detector and repositioning under the ion beam requires a flexible manipulation system with four degrees of freedom and a large working range.

A lift-out procedure using the MM3A-EM micromanipulator fitted with a ROTIP-EM rotational tip and the MGS2-EM microgripper is considerably faster than current methods:

- Grip the specimen using the microgripper
- Free the specimen from the bulk sample
- Lift out the specimen with the manipulator
- Rotate the specimen as desired for ion beam thinning
- Rotate the specimen to 90° and reposition for STEM analysis

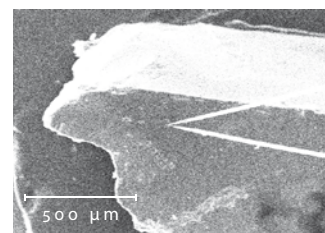
### Conclusions

The combination of a microgripper and a rotational tip represents the optimal nanomanipulation system for increasing sample preparation throughput. Using new tools a complete in-situ lift-out procedure can be carried out in under 20 minutes. Compared to conventional alternatives, this is a significant improvement in the speed and ease of TEM sample preparation.

In addition, a manipulator fitted with a rotational tip can be used for the flexible manipulation of TEM specimens inside FIB systems with multiple detector arrangements allowing the optimal positioning of the specimen with respect to the various detectors and beams.

### Further information

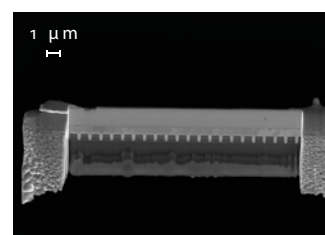
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After lift out of the specimen



Rotation of the specimen by 90°



The reprocessed lamella



STEM detector image of the lamella

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