The Versatile Application for In-situ Lift-out TEM Sample Preparation by Micromanipulator and Nanomotor

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Abstract

The device features have shrunk to sub-micron/nano-meter range, and the process technology has been getting more complicated, so TEM has become a necessary tool for PFA imaging and element analysis. Conventional FIB ex-situ lift-out is the most common technique for precise sample preparation. But this method has some limitations: samples cannot be reprocessed for further analysis; the carbon film supported grid affects the EDS analysis for carbon elements. A new installation will be introduced in this article, which is set up in FIB chamber for in-situ lift-out application. It not only overcomes the above problems, but also covers a wide application of TEM sample preparation.

Introduction

With the increasing complexity of process technology, device features are down to the deep sub-micron range. Transmission electron microscopy (TEM) has become the most important tool for detailed physical failure analysis and material analysis. The Focus ion beam (FIB) is also a necessity for failure analysis and TEM sample preparation. Ex-situ lift-out TEM sample preparation is to thin the sample by FIB and then transfers TEM lamella to carbon film supported grid by glass needle. It is the most popular method used to prepare TEM samples, but there are two problems with this technique: 1) TEM lamella on carbon film supported grid cannot be used for carbon signal analysis due to the carbon film which will affect the EDS analysis results, 2) it is also difficult to re-thin a TEM sample that has lain on a carbon film for further analysis (EELS, HRTEM). So an in-situ lift-out technique was developed to overcome these drawbacks.

Several commercial in-situ lift-out tools [1,2] are usually used for cross sectional TEM sample preparation. It lifts-out the sample and then attaches the sample to a half-cut copper grid inside the FIB chamber. For planar sample preparation and re-thinning of the TEM sample that has been put on a carbon film, the user must unload the copper grid with sample from the chamber and then reload it into the FIB chamber at a perpendicular angle for final milling. This increases the preparation time and risk of sample damage.

In this article, a novel in-situ lift-out tool will be introduced. It consists of a micromanipulator and a nanomotor, which can be easily installed into a FIB and does not have the drawback of the commercial tool mentioned above. Several applications of sample preparation will also be discussed such as lifting-out lamella from carbon film supported grid for further sample re-thinning to get element mapping by GIF, planar TEM sample preparation, and preparing samples for 3D TEM observation.

Tool Setup

This lift-out toll was set up in a FEI DB235, and it includes two key components: a Kleindiek MM3A micromanipulator and a Kleindieck rotational nanomotor (Fig. 1). The MM3A has two rotation axes and one linear axis. A tungsten needle can be inserted into the end of linear axis for lift-out. A TEM grid end-effector connects to nanomotor and can clamp a half-cut TEM copper grid to attach the lamella (Fig. 2). The nanomotor can rotate 360°. It is easy to orient the lamella to any direction with respect to the ion beam for further FIB milling so different kinds of TEM samples can be prepared by this tool without breaking vacuum. In addition, this tool is very compact. It is very easy to install and remove, and the

Figure 1: A MM3A manipulator and a nanomotor are installed on an adaptor plate, which can be set up and removed easily.
Sample Preparation Procedure

**Re-thin a TEM Lamella Lain on a Carbon Film**

It is very difficult to re-thin a lamella lain on a carbon film for further analysis like taking high resolution image or EELS analysis by a conventional ex-situ lift-out method. There is a paper that discusses a method to do this kind of preparation [3], but it is risky and not easy.

By using this novel tool, one can do this work easily. The preparation steps are described below:

- **Attach the lamella lain on a carbon film supported grid to FIB stage.**
- **Find the sample and use low ion beam current to pre-cut carbon film a little bit, then insert tungsten needle beside the sample (Fig. 4a).**
- **Stick the sample corner to tungsten (W) tip by using FIB induced platinum (Pt) deposition.**
- **Cut the carbon film around the sample so the W tip with lamella is free from the original copper grid.**
- **Lift-out the TEM lamella (Fig 4b).**
- **Move the FIB stage to center the half-cut Cu grid that is clamped by TEM grid end-effector (Fig. 4c).**
- **Insert the W tip with TEM lamella beside the half-cut Cu grid. In this step, the grid is perpendicular to the ion beam.**
- **FIB Pt deposition for lamella bonding to Cu grid.**
- **Cut the sample edge to separate W tip from lamella (Fig. 4d).**
- **Rotate Cu grid by nanomotor to parallel the sample to the ion beam for further milling (Fig. 4e). This is a very unique function of this tool; which makes the sample prepared without breaking vacuum.**
- **FIB re-thins the both sides of lamella (Fig. 4f).**
- **Figures 4g and 4h show the TEM image and further GIF analysis.**
- **If a STEM detector is installed, STEM image is possible to be observed. This work has been going on in our Lab.**

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**Figure 2:** A TEM grid end-effector (micro tweezers) clamps a half-cut copper grid and inserts into nanomotor.

**Figure 3:** Control cable connects to a chamber port.

tungsten needle replacement is also easy and quick. Figure 3 shows that only one electric cable is needed for tool control.
Planar TEM Sample Preparation

The traditional technique for planar TEM sample preparation is time-consuming. This tool can be used for this application easily and quickly.

The preparation steps are described as below:

- Deposit a platinum protective layer on the specific area.
- Mill the front and back edge of the area at zero tilt. So the bottom of the triangular bulk is free from the substrate (Fig. 5a).
- Cut the right sidewall of the triangular bulk.
- Stick the sample corner to W tip by using FIB Pt deposition.
- Cut the left sidewall of the bulk so the W tip with the bulk specimen is free from the substrate completely. Then lift-out the sample (Fig. 5b).
- Move the FIB stage to center the half-cut TEM Cu grid.
- The following steps are the same as the procedure for re-thinning a lamella lain on carbon film. Stick the sample to half-cut Cu grid and cut the sample edge to separate the W tip from the sample (Fig. 5c).
- Rotate the Cu grid 90 degree.
- FIB mills both sides of the bulk sample (Fig. 5d).
- Figure 5e shows the planar TEM image.
Reprocess a TEM Lamella from Carbon Film for 3D Observation

A defect is not always easy to be precisely located to make a cross-section TEM. So, the sample needs to make plane view TEM observation first to locate the exact defect position, and then make a precise cross-section. Sometimes the physical information of a defect is not provided sufficiently by TEM observation in only one direction. The 3D information of the defect (plane view and cross-section, X and Y direction cross-section) becomes necessary to judge the failure root cause of the problem [4].

The following procedure will describe the sample preparation method using the in-situ lift-out tool for 3D observation:

1. Get an X direction cross-section TEM image (Fig. 6a).
2. Attach the carbon film supported copper grid to FIB stage.
3. Pre-cut carbon film a little bit and insert W needle beside the sample.
4. Stick the sample corner to W tip by using FIB Pt deposition.
5. Cut the carbon film around the sample.
6. Lift-out the TEM lamella (Fig. 6b).
7. Center the TEM half-cut grid.
8. Insert W tip with TEM lamella beside half-cut Cu grid. The grid is always parallel to the ion beam in this method.
9. Pt deposition for lamella bonding to Cu grid.
10. Cut the end of W tip to separate W tip from lamella.
11. Pt deposition on the sample to protect the target (Fig. 6c).
12. FIB mills both sides to approach the target (Fig. 6d).
13. Figure 6e, 6f show a further Y direction cross-section TEM image of sample in Fig. 6a.

The X direction cross-section image shows a defect under poly, but it is hard to judge when and how the defect formed depending on the less information. If this is the only sample, then there will be no chance to make another direction TEM sample to collect more data. This tool easily makes it possible to prepare Y direction cross-section sample from the original X direction cross-section sample. Figure 6e, 6f
are the TEM image taken from another direction, which clearly shows the substrate damage is due to spacer etching.

Figure 6b: Cut the carbon film around the sample then lift-out the lamella.

Figure 6c: Stick the sample to half-cut Cu grid, then cut the sample corner to separate the W tip from the sample.

Figure 6d: FIB mills both sides to approach the target in Y direction.

Figure 6e, 6f: Further Y direction cross-section TEM image of sample in Fig. 6a shows the substrate damage is due to L spacer etching.

are the TEM image taken from another direction, which clearly shows the substrate damage is due to spacer etching.

**Conclusions**

A novel in-situ lift-out installation consists of a micromanipulator and a nanomotor with TEM grid end-effector was set up in a dual-beam FIB for TEM sample preparation. This tool was used to demonstrate several kinds of sample preparation successfully for TEM analysis demands. All preparation procedures are carried out without breaking vacuum. It also has some advantages: 1) it is compact and easy to be installed and removed, 2) tungsten needle can be replaced easily and quickly, 3) it is cheap, 4) it can be set up to existing FIB. This tool has already been widely used for difficult case handling in our lab.

**References**

[1] AutoProbe, Omniprobe Inc., Dallas, TX