doi:10.1017/S1431927618004798
Microsc. Microanal. 24 (Suppl 1), 2018

Preparation of Sharp Tip Samples Using a Tescan GAIA3 FIB-SEM System

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Very sharp tip samples are needed for a combination of atom probe tomography (APT) and TEM tomography. Most of the tip samples have been prepared so far in FEI DualBeam systems and Zeiss crossbeam systems. Here I report the tip sample preparation using a Tescan GAIA3 FIB-SEM system. General steps of the sample preparation in the Tescan GAIA3 system are like those in other FIB-SEM systems, including sample surface protection, lift-out, sample mounting onto an OMNI TEM grid, and tip forming. Some unique features of the GAIA3 system make the detailed procedures for the tip sample preparation different from other systems.

The Tescan GAIA3 FIB-SEM system at UCI (Figure.1) is equipped with an OmniProbe 400 nanomanipulator and a 5-line gas injection system (GIS). When the sample stage is horizontal without tilting, the 5-line GIS can be inserted only when the distance between the electron objective lens pole-piece and sample surface (Dos) is larger than 6mm, while the coincident point of electron beam and ion beam is 5mm below the pole-piece. Fortunately, when the sample stage is tilted towards the ion beam by 55 degrees, the GIS can be inserted when the Dos is 5 mm (Figure. 2). So, the limitation for GIS operation at zero stage tilting does not affect Pt deposition procedures, but the sample lift-out cannot be done when the sample stage is horizontal.

The OmniProbe 400 nanomanipulator is set up so that a TEM lamella can be lifted out when the sample is tilted by 55 degrees (Figure. 2). The GAIA3 system is equipped with a 35-degree pre-tilt STEM holder mounted at the edge of the sample stage (Figure. 3). A normal OMNI TEM grid can be installed easily into the STEM holder with a small Tescan tool. The GIS position was tuned carefully so that the GIS, OMNI probe and the pre-tilt STEM holder can be operated together when the stage is not tilted and the TEM grid is at Dos=5mm (Figure. 3). It is easy to mount a TEM lamella on the side surface of a TEM grid bar, however, there is a little challenge to mount a tomography sample securely onto the top of a grid bar. A strategy has been figured out to solve this issue (Figure. 4).

Final tip forming can be done by circle milling with varying diameters (Figures. 5-6), which is controlled by a Tescan software called Drawbeam. That software is very powerful to generate various patterns for ion etching. Different ion beam voltages and currents were used to sharpen the tip. The final tip sample (Figure. 7) was completed by using a low energy Ga ion beam (3keV).

The OMNI TEM grid can be mounted into the home-made adapter which may replace the removable sample tip of Hummingbird Scientific tomography holder for TEM imaging (Figure. 7). High quality HRTEM images have been obtained in the region of interest. This preliminary work has proved that the procedures are feasible to make a sharp tip sample for tomography. Further optimizing the final tip forming procedures and reducing amorphous layer on the tip are in progress.

The author would acknowledge the use of IMRI facilities for the FIB and TEM work. The test sample was provided by Matt Law’s group.

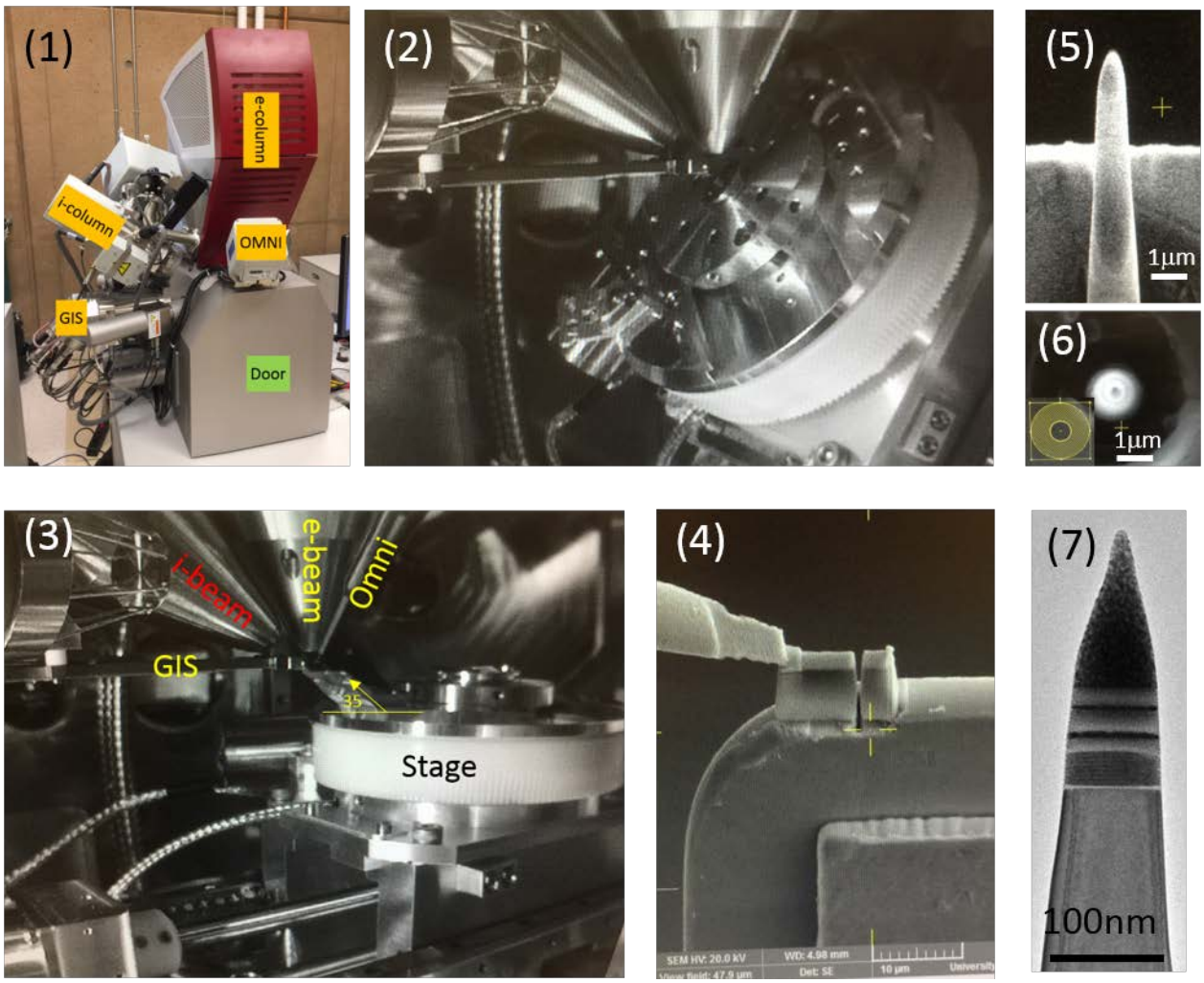

Figure 1. Tescan GAIA3 FIB-SEM system at UCI
Figure 2. Setup for ion beam induced Pt deposition with stage tilted at 55 degree and GIS inserted.
Figure 3. Setup for sample wielding on the top of an Omni TEM grid. The grid is installed in a STEM holder with a pre-tilt angle of 35 degrees relative to stage surface. GIS and OMNI probe are inserted.
Figure 4. SEM image showing the steps to transfer the sample onto the top of an Omni grid bar. The sample is cut free.
Figure 5. SEM image showing the side view of a tip after initial tip forming procedure
Figure 6. FIB image showing the top view of the same tip displayed in Figure 5. The inset is the tip forming pattern defined by an outside circle and an inner circle. The pattern can provide flexible control of the ion beam and enable the beam to move along the circles with different diameters.
Figure 7. TEM image of a test tip which is composed of 30 nm thin film on Si (100) substrate and protected by e-beam-induced Pt and i-beam-induced Pt deposition. The tip was sharpened with 30 kV Ga ion beam and finally cleaned by a 3kV Ga ion beam. A region of interest smaller than 100 nm in diameter was achieved. Atomic resolution HRTEM image of the thin film and Si was recorded. The amorphous layer in the Si surface is less than 2 nm.

https://doi.org/10.1017/S1431927618004798 Published online by Cambridge University Press