

High-Accuracy Sample Preparation for Three Dimensional Atom Probe Tomography Using Orthogonal Column Layout FIB-SEM and its STEM function

Miki Tsuchiya¹, Yoshihisa Orai¹, Takahiro Sato¹, Xin Man² and Junichi Katane¹, Tsuyoshi Onishi³

¹. Hitachi High-Technologies corp., Science System Product Div., Hitachinaka-shi, Japan

². Hitachi High-Tech Science corp., Beam Technology Systems Design Group 1, Oyama-cho, Japan

³. Hitachi High-Technologies corp., Science System Sales & Marketing Div., Tokyo, Japan

Atom probe tomography (APT) analyses chemical composition in three-dimensional distribution with near atomic-scale resolution. Recently, the APT method can be widely applied from high-conductivity materials to semiconductors and insulators. Reason of this wide-spreading of the APT method is because the use of FIB (Focused Ion Beam)-SEM (Scanning Electron Microscope) contributes to site-specific sample preparation and fine needle tip sharpening which diameter is less than 100 nm [1]. However, it becomes more difficult to determine the end-point of FIB fabrications with only SEM observation due to the decrease in information such as contrast and signal from a smaller structure surface. Thus, the authors developed the high-accuracy and high-quality sample preparation technique for APT using SEM and STEM (Scanning Transmission Electron Microscope) signals which detected by orthogonal column layout FIB-SEM.

The sample we applied the new technique is a commercial 22 nm finFET. The orthogonal column layout FIB-SEM is used for APT sample preparation [2][3]. One of the features of the FIB-SEM is an optimized arrangement of all detectors including EDX (Energy Dispersive X-ray spectroscopy), EBSD (Electron Backscatter diffraction) and STEM. As shown in figure 1, the beam irradiation direction of the FIB-SEM and each detector are arranged to the beam coincident point. The high-speed STEM detector is placed underneath the sample so that the APT sample milling process is observable with STEM and SEM images during FIB processing simultaneously. To ensure the making of an APT sample including the target, STEM and SEM monitoring is done from two directions, 0° and 90°.

The nanomesh® is newly developed for the APT method as shown in figure 2, which has 5 Si square pillars. Figure 3 shows the schematic image of sample preparation workflow and SEM or STEM images of each process. The lift-out sample is fixed on the square pillar by filling the gap between the sample and the pillar with tungsten deposition (a) (b). When the sample is mounted on the pillar, the grid is rotated to 45° so that the section of the lift-out sample is observable from two directions. Then, the grid is tilted at 45° and trimmed from the right and left of the sample to be a square pillar of 400 nm in width with a flat surface (c) (d). After the sample trimmed until around 400 nm width, the grid is back to 0°. The part surrounding the target of the sample is then annularly milled using a donut-shaped fabrication pattern to obtain a conical tip with an end diameter of around 100 nm. STEM images taken by switching between two positions alternately are used to monitor the target position inside the sample during FIB processing simultaneously (e). Finally, low accelerating voltage FIB is used to remove both the metal gate and the damage layer induced by 30 kV FIB [Figure 4].

The APT sample is transferred to LEAP400XSi to obtain the three-dimensional chemical composition. The APT analysis result of a Si fin and gate dielectric layers is shown in Figure 5. Three-dimensional chemical distribution around the Si fin is observed. This result proves that the sample prepared with STEM monitoring includes the target site.

APT sample preparation containing a specific structure in the tip's apex has been realized by both SEM images of orthogonally arranged FIB-SEM and STEM image information obtained in real time. Using STEM

monitoring from two directions during FIB processing ensures the end point detection in APT sample preparations. This technique can provide high-accuracy APT sample preparations and high-throughput APT analyses.

References:

- [1] M.K.Miller *et al*, Microsc.Microanal., **13** (2007), p. 428.
- [2] X.Man *et al*, Microsc.Microanal., **20. Suppl 3** (2014), p. 354.
- [3] M.Tsuchiya *et al*, Proceedings of the 73rd Annual meeting of JSM2017 **52**, (2017) p. 88.

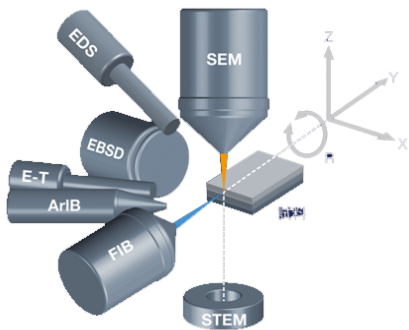


Figure 1. Schematic image of L-Shape FIB-SEM triple beam system.

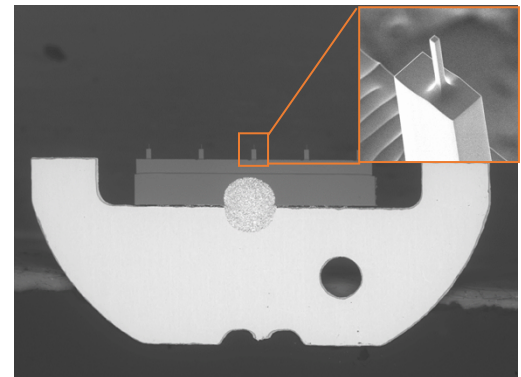


Figure 2. Newly developed nanomesh® for APT sample.

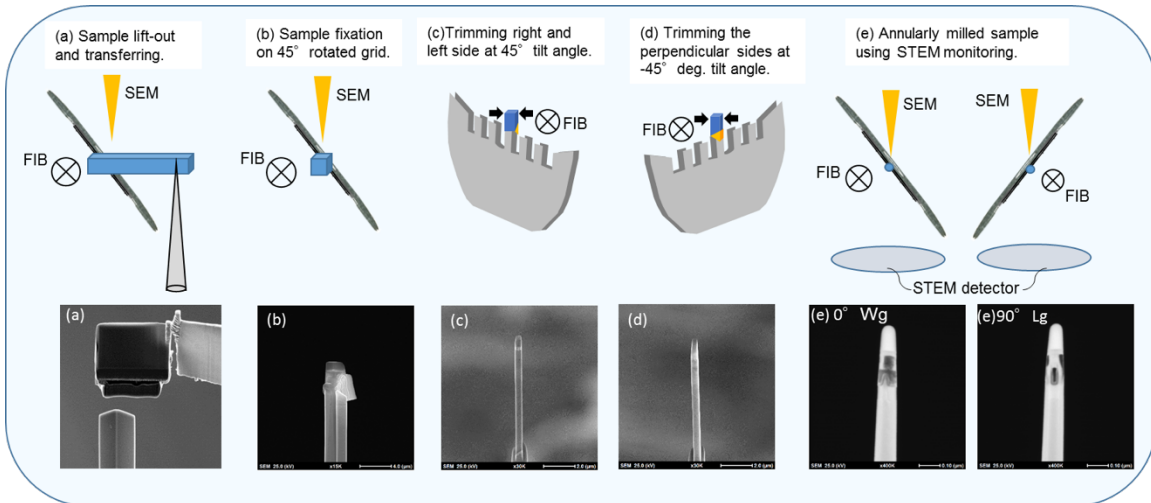


Figure 3. SEM image and STEM image in the thinning process

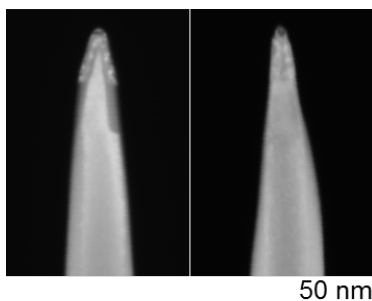


Figure 4. 25 kV STEM image of a p-type fin after final milling. (a) 0° direction and (b) 90° direction

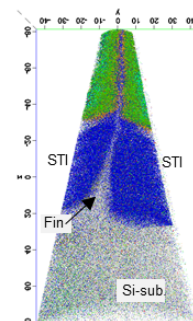


Figure 5. Atom probe tomography (APT) map of a p-type semiconductor fin