

# Automating FIB Sample Preparation to Improve STEM Throughput

Daisuke TSURUMI\*, Tomohiro DOI, Kazuki TORII, Rie MARUYAMA, and Shinji MATSUKAWA

In the development and production of compound semiconductor devices, atomic-scale structural analysis is required to evaluate their performance and characteristics. For the analysis, scanning transmission electron microscopy (STEM) is indispensable. However, it is necessary to control the sample thickness to around 100 nm using a focused ion beam (FIB) milling system, and the required number of samples could not be prepared quickly even by highly skilled engineers. In order to solve this problem, we have improved the processing capacity by automating the FIB milling process. Since a wide variety of devices are being developed and manufactured in our company, it is necessary to prepare various kinds of samples according to the analysis objectives of each device. Therefore, we identified the automation conditions for each sample, and by combining the established automation functions we constructed a system that can deal with a large number of samples expeditiously. The higher throughput enables us to repeat the device fabrication and evaluation in the shorter turnaround time, accelerating the development of devices and improvement of the quality.

Keywords: compound semiconductor device, structural analysis, FIB automation function, STEM, throughput

## 1. Introduction

In the large-capacity and high-speed communication networks, such as trunk lines, metro optical communication systems, smartphones, and wireless communications for base stations of the modern society, the compound semiconductor devices, such as laser diodes, photodiodes, and high electron mobility transistors, play important roles.<sup>(1)</sup> Since the network is expected to continue to rapidly increase in capacity due to the popularization of the Internet of Things (IoT) technology and the increase of cloud services etc., it is necessary to accelerate the research and development of the devices and improve the quality of the products in the manufacturing. For this purpose, iterations of device fabrication and evaluation should be conducted as quickly as possible.

Compound semiconductor devices have become more complicated and highly miniaturized, and structures having different functions must be integrated in one chip at high densities.<sup>(2)</sup> Therefore, a structural analysis technique for the evaluation process is crucially important. As an analysis method, scanning transmission electron microscopy (STEM),\*<sup>1</sup> which provides atomic-level spatial resolution, is one of the most useful techniques. However, sample preparation for STEM is very time consuming and the required number of samples cannot be prepared quickly. This is because it is necessary to lift out the analysis target from inside the device and uniformly thin the sample to around 100 nm thickness using a focused ion beam (FIB) milling system\*<sup>2</sup> so that the electron beam can transmit the sample. These processes take such a long time that required number of samples cannot be prepared quickly.

In order to eliminate this bottleneck and enlarge the processing capacity for STEM, we attempted to automate the FIB sample preparation process. In this paper, we report the use of the FIB automation function to improve STEM throughput. This enabled us to iterate the fabrication and

evaluation expeditiously, accelerate the research and development, and improve the manufacturing quality of the devices.

## 2. FIB Automation Function

We attempted to utilize the FIB automation functions of the NX2000 FIB-Scanning Electron Microscopy (SEM) system,\*<sup>3</sup> released by Hitachi High-Technologies Corporation.

As shown in Fig. 1, STEM sample preparation is composed of three steps: 1) milling trenches around the sample, 2) lift out, and 3) thinning.<sup>(3)</sup> While a wide variety of devices are developed and manufactured, it is necessary to prepare samples according to the specific analysis objectives of each device. Therefore, we identified the automation conditions for each sample preparation process, and by combining the established automation functions as appropriate, we constructed a system that can deal with a large quantity of samples expeditiously.

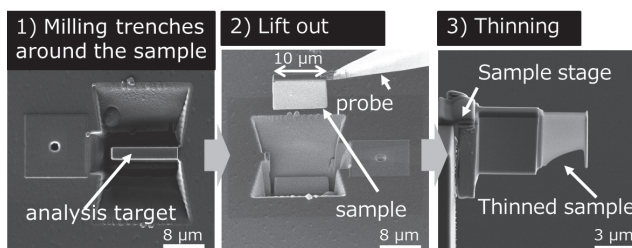


Fig. 1. STEM sample preparation steps

### 3. Utilization of FIB Automation Function

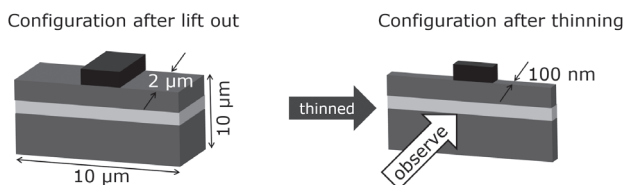
In this chapter, we describe the utilization of the FIB automation function for each step: 1) milling trenches around the sample, 2) lift out, and 3) thinning.

#### 3-1 Automation of milling trenches around the sample

For STEM sample preparation, trenches around the sample must be milled in order to lift out an about  $10 \times 10 \mu\text{m}$  section of the analysis target. For this process, we needed to establish the milling conditions for each sample. This is because not only various materials such as semiconductor materials (GaN, GaAs, InP, etc.) but also oxide films, metals, and resins are used and milling rates differ depending on the material. Furthermore, in addition to cross-section observation, plan-view observation as shown in Fig. 2<sup>(4)</sup> is required for spatially identifying the position and distribution of crystal defects. As a consequence, it is necessary to modify the size of the sample to be lifted out to about  $10 \times 10 \times 2 \mu\text{m}$  for cross-sectional observation and about  $10 \times 10 \times 10 \mu\text{m}$  for plan-view observation. We determined the processing times and patterns taking into consideration the difference in processing rates depending on the material and the observation orientation. Furthermore, we selected the position of the drift correction mark\*<sup>4</sup> that can be reliably recognized even if the device surface is markedly uneven. In this way, 12 types of base milling conditions were determined.

Thus, we succeeded in automating trench milling around the sample by applying the appropriate milling conditions according to the device and the specific analysis objectives.

#### a) Thinning process for cross-section observation



#### b) Thinning process for plan-view observation

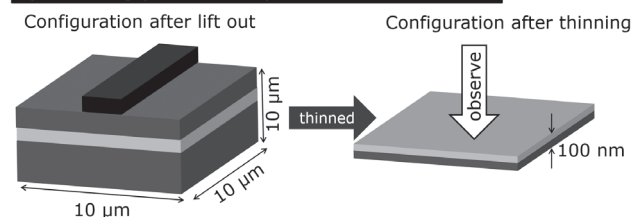


Fig. 2. Configurations of STEM samples for a) cross-sectional and b) plan-view observation

#### 3-2 Automation of lift out

Lift out is a process of picking the target sample up and attaching it to the STEM sample stage. Usually, the attitude of the sample lifted out from the device is as shown in Fig. 3 (a). However, devices usually have structures such as electrodes on the top of the semiconductor substrate, and the presence of these structures causes a difference in the

FIB milling rate during thinning the sample. As a result, artifacts (lines seen in the vertical direction indicated by arrows) occur on the milled surface of the semiconductor substrate, and then that part of the sample becomes thinner than the targeted thickness or sometimes disappears while the other part remains thicker during the thinning process. As a countermeasure, the sample can be thinned uniformly by irradiating FIB from the uniform substrate side as shown in Fig. 3 (b).<sup>(5)</sup> In this method, the attitude of the sample attached to the probe has to be controlled and the sample rotated upside down. Then, the sample is fixed to the sample stage, and thinned by FIB irradiated from the substrate side of the structure. Thus, the sample is uniformly thinned. When we tried to automate this process, there were challenges specific to attitude control such as the method of attaching the target sample to the probe and the method of image recognition of the rotated sample. We have optimized the conditions applicable to our devices and have eliminated these problems, and automation of the lift out process with the attitude controlled as shown in Fig. 3 (b) was achieved.

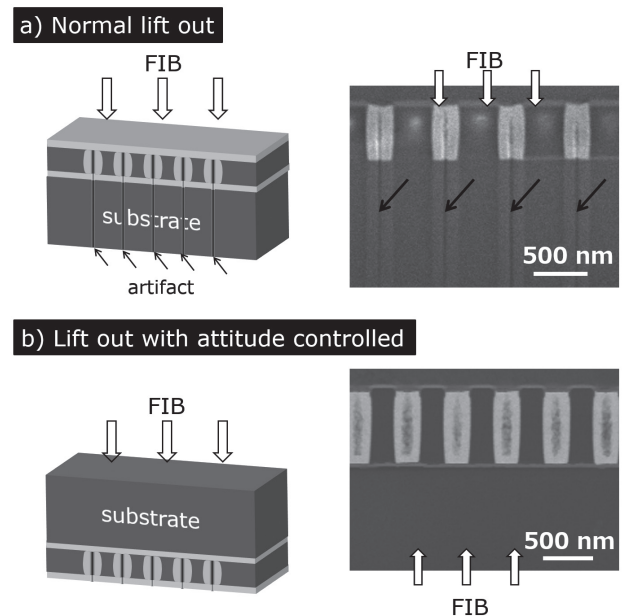


Fig. 3. a) The artifact caused by FIB milling during thinning the sample with the normal attitude and b) the effect of the attitude control on the artifact

#### 3-3 Automation of thinning

During STEM observation, the scattering of transmitted electrons is suppressed by reducing the sample thickness, thus making high-resolution observation possible. Therefore, it is necessary to reduce the thickness to 100 nm or less for atomic resolution observation.

In order to prepare a sample less than 100 nm thick, the irradiating current needs to be lowered gradually according to the sample thickness: a large irradiation current is used for milling to a sample thickness of about  $1 \mu\text{m}$ , and a small current is used for processing the last

100 nm. On the other hand, thick samples (about 1  $\mu\text{m}$ ) through which the electron beam can be transmitted are preferable when the purpose is to investigate defects such as dislocations. This is because the thicker the sample, the wider the observation range and the easier it is to find defects. Thus, it is necessary to control the sample thickness according to the analysis purpose.

We applied the automation function to this thinning process, and it was found that a sample with a thickness of 120 nm or more can be consistently produced. Therefore, this automation function is used for preparing samples over 120 nm thick, and for further thinning, a skilled engineer operates the process using SEM images and finely adjust the milling process.

#### 4. Application Example of Automation Function

The automation function was applied to each of the three processes in STEM sample preparation. By combining the three types of automation functions according to a wide variety of devices and analysis purposes and performing automatic processing continuously, we constructed a system that can deal with a large number of samples and achieved 5 times the throughput of the conventional STEM analysis. As a result, STEM analysis can be utilized for prompt failure analysis, structure analysis of prototype devices, and evaluation of in-plane positional variation of semiconductor wafers, among other things that were previously difficult to accomplish.

As an application example of the automation function, Fig. 4 shows the relationship between measured length obtained by STEM images and the electrical characteristics of a device. This is the result of STEM analysis on 16 samples using the automation function. The graph shows that there is a correlation between the length and the current value. This result indicates that it is necessary to control the accuracy of the structure within 1 nm in this device, and we can improve the manufacturing process based on this feedback.

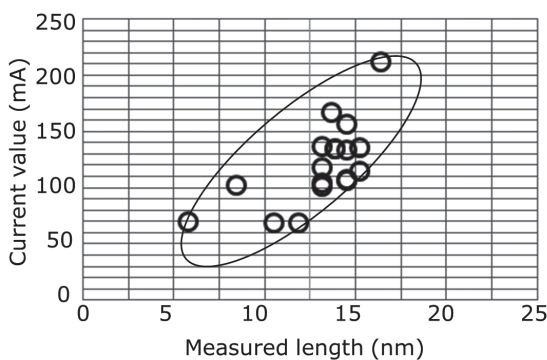


Fig. 4. Relationship between the measured length obtained by STEM analysis using the automation function and current values of a device

## 5. Conclusion

STEM analysis is indispensable in the development and mass production of compound semiconductor devices. The FIB process was automated to eliminate the STEM sample preparation bottleneck. We have enlarged processing capacity by introducing the automated functions of the FIB milling system. We constructed a system that can deal with a large quantity of samples expeditiously by combining the automation functions according to a wide variety of devices and specific analysis objectives, and performing automatic processing continuously. Thus we have achieved a throughput 5 times the amount of the conventional STEM analysis. As a result, STEM analysis can be utilized for prompt failure analysis, structure analysis of prototype devices, and evaluation of in-plane positional variation of semiconductor wafers, among other things that were previously difficult to accomplish. We will continue our efforts to accelerate the development of semiconductor devices and further improve the quality.

#### Technical Terms

- \*1 Scanning transmission electron microscopy (STEM): A high-resolution microscopy that can observe atomic image directly. Electron beam is irradiated to thin sample, and images are constructed by detecting transmitted electrons through the sample.
- \*2 Focused ion beam (FIB) milling system: A system for milling by irradiating a Ga ion beam on a sample. It is possible to cut out the cross section of the desired position in nano-meter level.
- \*3 FIB-SEM system: A system that has both FIB and scanning electron microscopy (SEM) functions in one facility. During or immediately after milling with FIB, the milled surface can be observed by the SEM. In STEM sample preparation process, operations can be performed precisely while observing the milling status.
- \*4 Drift correction mark: When the processing time by FIB becomes long, the milling position may shift due to the drift of the sample stage with time. As a countermeasure, a unique mark is milled on the sample, and the processing position deviation due to the drift is corrected based on the mark position.

#### References

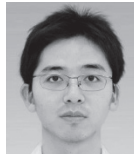
- (1) Hideki Hayashi, "Development of Compound Semiconductor Devices - In Search of Immense Possibilities-," SEI TECHNICAL REVIEW, Number 72, pp. 4-16 (Apr. 2011)
- (2) H. Yagi et al., "InP-Based Monolithically Integrated Photonic Devices for Digital Coherent Transmission," IEEE J. of Sel. Topics in Quantum Electron., Vol. 24, No. 1 (Feb. 2018)
- (3) T. Ohnishi, et al., "A New Focused-Ion-Beam Microsampling Technique for TEM Observation of Site-specific Areas," Proceeding from the 25th International Symposium for Testing and Failure Analysis, Santa Clara, California, pp. 449-453 (Nov. 1999)
- (4) H. Saka et al., "Plan-view transmission electron microscopy observation of a crack tip in silicon," Philosophical Magazine Letters, Vol. 72, No. 4, pp. 251-255 (May 1995)
- (5) Jun Kato, "Evaluation of micro-devices using high-end dual beam," The TRC News, Vol. 121, pp. 12-15 (Oct. 2015)

---

**Contributors** The lead author is indicated by an asterisk (\*).

**D. TSURUMI\***

- Ph.D.  
Group Manager, Analysis Technology Research Center



**T. DOI**

- Analysis Technology Research Center



**K. TORII**

- Analysis Technology Research Center



**R. MARUYAMA**

- Assistant Manager, Analysis Technology Research Center



**S. MATSUKAWA**

- Group Manager, Analysis Technology Research Center

