Specimen Preparation Technique for a Microstructure Analysis Using the Focused Ion Beam Process

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ABSTRACT In recent years the FIB technique has been widely used for specimen preparation in TEM observation and AES analysis, increasing demand for micro-analysis of semiconductor devices. Compared to the conventional process consisting of mechanical polishing and Ar ion milling, the FIB technique has several advantages: rapid process, preventing damage caused by mechanical polishing, and cutting out microstructure parts. However, there are various problems such as detection of large amounts of Ga from the ion source and formation of a damaged layer on the surface. We have developed FIB specimen preparation techniques to obtain valuable data for TEM and AES in consideration of these features of FIB processing.

1. INTRODUCTION

As electro-optical semiconductor devices become smaller, a microstructure analysis technique is required to analyze defects and control the production process. Transmission electron microscopy (TEM) and auger electron spectroscopy (AES) are analytical techniques used to evaluate microstructures.

Recently, the focused ion beam (FIB) technique has been used as a method of fabricating a specimen for TEM observation, besides the conventional ion milling method. This technique is indispensable for analyzing semiconductor devices, because cross-sectional TEM observation at any specified location is possible using the microsampling method[1], which enables specific parts to be cut out from a bulk sample. FIB is also an effective tool for fabricating samples for AES analysis[2]. This paper presents a FIB specimen preparation technique for TEM observation and AES analysis.

2. FIB PREPARATION FOR TEM OBSERVATION

2.1 FIB Process Damage and Problem of Sample Form

The FIB process, in which a convergent gallium ion beam is irradiated onto a sample and a specific part is made thinner, has the problem that damage caused is greater compared to the conventional ion milling method. There are two types of process-damage in compound semiconductors such as GaAs and InP, the first is the formation of amorphous layers that contain implanted gallium atoms, the second is the precipitation of particles. In particular, in TEM observation, precipitation produces a macula contrast, which greatly influences the image observed. The level of damage varies depending on the material, for example, for InP it is remarkably high. It also depends on acceleration voltage, incident angle, and temperature when fabricating. Damaged layers are created on the process surface. For these reasons, it seems that the ion milling method using a low acceleration voltage and a low incident angle at a low temperature is effective for reducing the damaged layer.

In conventional FIB preparations, a metal plate from 30μm to 50μm in thickness for TEM observation is used to fix samples. A sample is polished to the proper size by hand or picked up using a microsampling method, then it is mounted on a cross-section of Cu plate, and only the part required for observation is thinly fabricated up to a few hundred nanometers in thickness. To remove the damaged layer produced during the FIB process, it is necessary to irradiate an argon ion beam at a low angle from both sides of the sample surface. However, in the general process, the feature of the sample surface is concave, hence the Cu plate and the unprocessed part of the sample become walls and prevent the argon ion beam from reaching the processed surface (Figure 1). Furthermore, there is the problem that materials, which were sputtered from the walls, adhere again and contaminate the processed surface. Consequently, it is impossible to remove the damaged layer by the conventional sample preparation method.

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2.2 FIB Sample Preparation Method with Little Damage

2.2.1 Cu Half Plate with Ultra-thin Aluminum Foil and Microsampling Method

We developed a new specimen preparation technique using the microsampling method and a Cu half plate with ultra-thin aluminum foil to overcome the problem of specimen form, and we made it possible to fabricate a FIB sample with little damage using cold ion milling. The FIB process conditions are shown below.

FIB: Hitachi FB2000A
Acceleration voltage: 30 kV
Ion beam: Ga^+

To irradiate an Ar ion beam onto the processed surface and prevent redeposition, it is necessary to make a thin Cu half plate and reduce concavity and convexity near the processed surface. To fabricate the thin stand, an ultra-thin aluminum foil of 2.5μm in thickness is attached to the hole of the Cu half plate using epoxy resin. The part of the sample of the same thickness as the aluminum foil was picked up by the microsampling method, and both the left and right sides of the sample were fixed on the cross-section of the aluminum foil using tungsten deposition. Not only the observed area but also the aluminum foil were processed to be thinner using the FIB method. Using this method, the sample became flat, and surroundings of the fabricated part were opened, therefore, an Ar ion beam irradiated at a low angle could reach the processed surface from all directions (Figure 2-1, -2, -3).

2.2.2 Cooling Ion Milling

After sample preparation using the FIB method, the sample was milled by cooling ion milling (Figure 2-4). The milling conditions are shown below .

Ion milling: GATAN600N
Acceleration voltage: 2 kV
Incident angle: 15 degrees
Ion beam: Ar^+
Ion milling time: 5 min.
Specimen temperature: 77 K

2.3 TEM Observation and Evaluation of Damaged Layers

2.3.1 TEM Observation

To confirm the effects of removing damaged layers by cooling ion milling, an observation was made using a high-resolution transmission electron microscope (HRTEM). The sample before ion milling was observed for purposes of comparison. The observation condition is shown below.

TEM: Hitachi H9000UHR
Acceleration voltage: 300 kV
Sample: InGaAsP/InP MQW

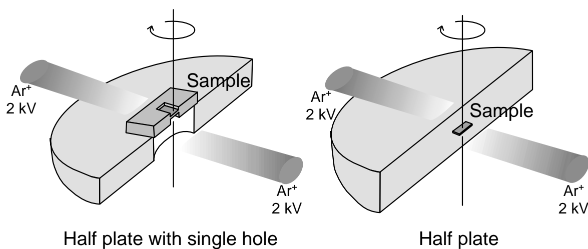
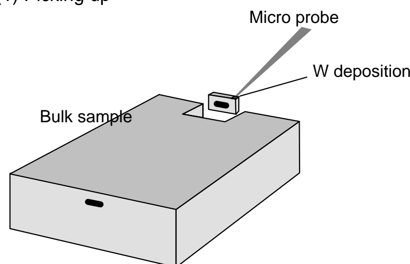
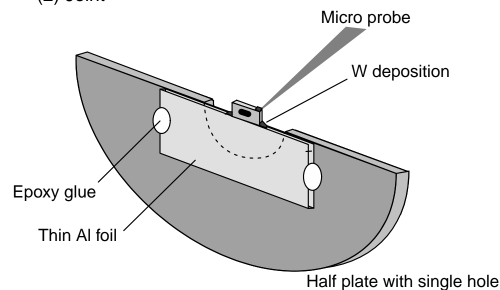
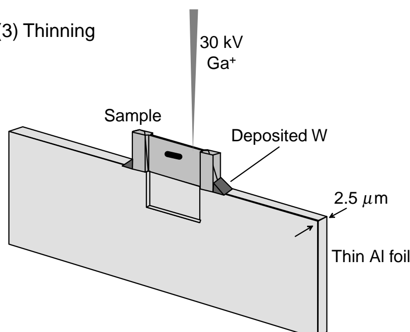


Figure 1 FIB-TEM Sample.







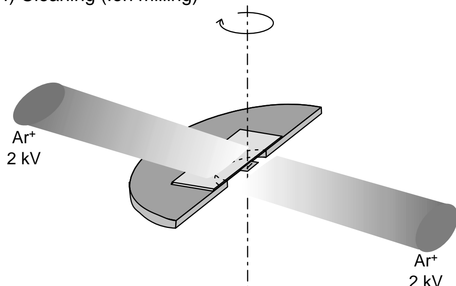


Figure 2 FIB sample preparation method for TEM observation with little damage.

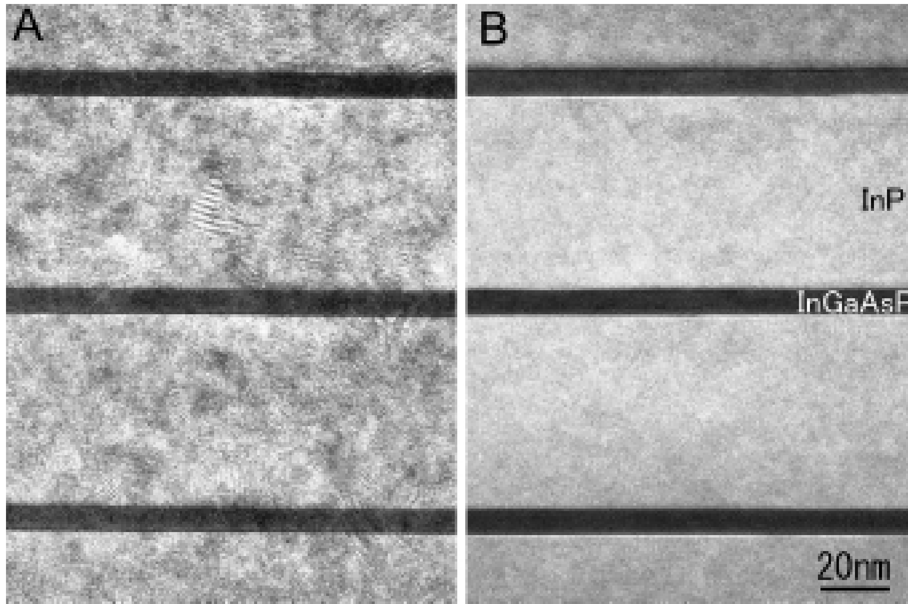


Photo 1 Dark field image of InGaAsP/InP MQW.

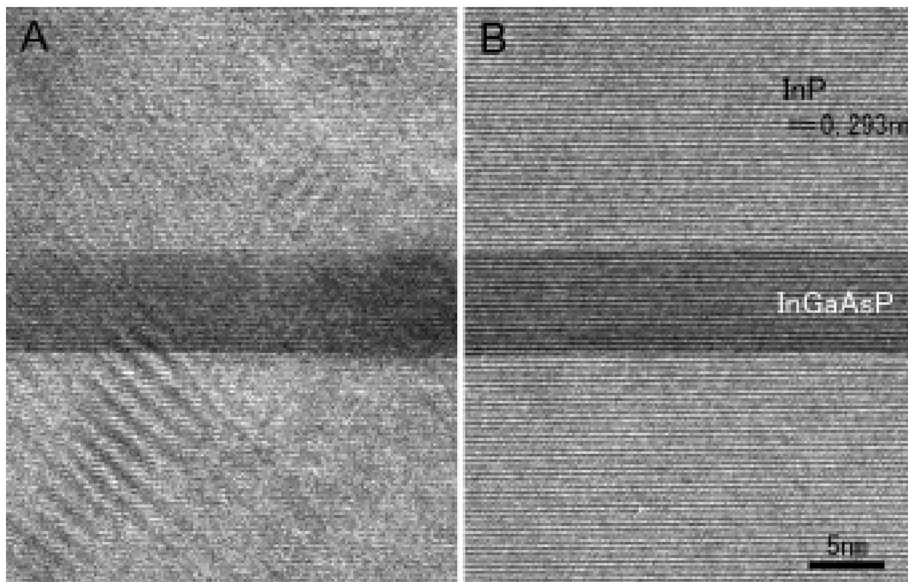


Photo 2 Lattice image of InGaAsP/InP MQW.

Photos 1 and 2 show cross-sectional TEM images (dark-field image and lattice image) of InGaAsP/InP MQW. In sample A before removing the damaged layers, observation was made of contrast and Moiré fringe, which appear when crystals with a different orientation or spacing of lattice planes overlap. In addition, the lattice cannot be observed clearly due to the thin damaged layers caused by gallium implantation. The influence of damage to the sample caused only by FIB processing is remarkable. In contrast, in sample B after removing the damaged layers by ion milling, the lattice is observed clearly without contrast and Moiré fringe. These investigations demonstrate that the damaged layers on the processed surface are removed to obtain valuable data for TEM observation by cooling ion milling.

2.3.2 Evaluation of Damaged Layers

We evaluated the damaged layers. Two types of sample were prepared. In the first sample, damaged layers were formed by FIB fabrication. In the second sample, damaged layers were formed, and then removed by cooling ion milling. Figure 3 shows the sample preparation techniques. Part of bulk sample was fabricated by FIB and the damaged layers were formed. The damaged layer of the second sample was then removed by cooling ion milling. Processed surfaces of both samples were coated with a metal layer to prevent re-damage due to FIB fabrication. The samples were picked up using the micro-sampling method, and fabricated using the method shown in Figure 2. Cross-sectional TEM images of these samples are shown in Photo 3. In the FIB fabricated sample (photo-

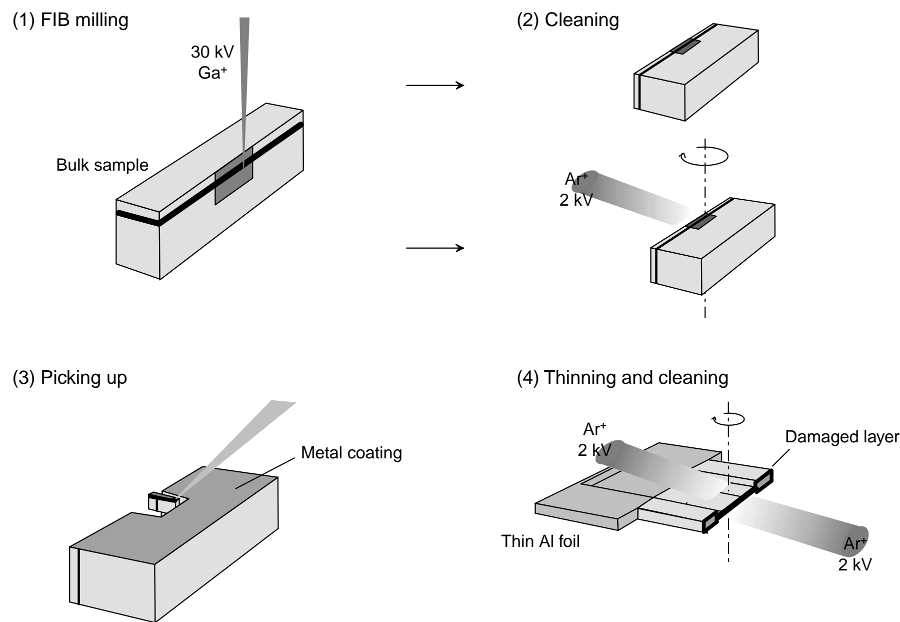
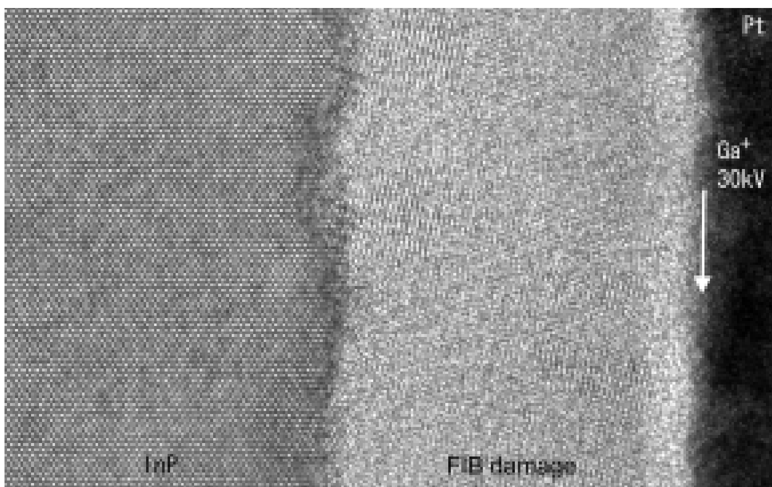


Figure 3 Damaged layer evaluation sample preparation method.



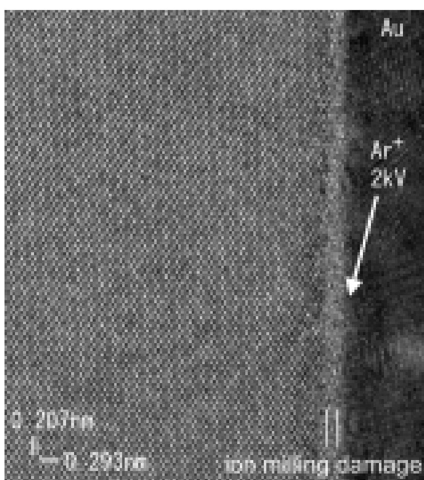


Photo 3 Cross-sectional TEM image of damaged layer.

graph above), the amorphous-like nano-crystalline damaged layer of more than 20 nm thick is formed on the processed surface. Contrary to this, it should be noted that the thickness of the amorphous damaged layer is approximately 1.5 nm using cooling ion milling (photograph below). It is understood that most of the damaged layers formed by FIB processing are removed by cooling ion milling.

2.3.3 Summary

We developed specimen preparation FIB techniques using cooling ion milling to reduce damaged layers. In the conventional FIB technique, damaged layers could not be removed because the specimen's shape prevents the argon ion beam from reaching the processed surface. This problem was solved using a new specimen preparation technique with Cu half plate, ultra-thin aluminum, and micro-sampling method, which allows most of the damaged layers to be removed. High-resolution TEM images were obtained. As a result of an evaluation of thickness of damaged layers, it is clear that a damaged layer of more than 20 nm in thickness is formed on the processed surface. This indicates that, if a thin film sample of 100 nm in

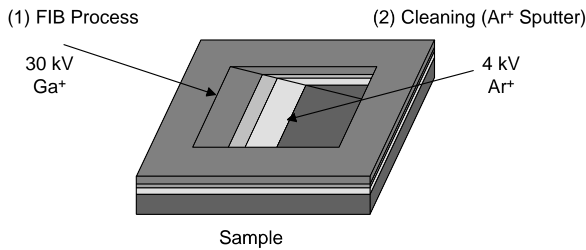


Figure 4 FIB sample preparation for AES analysis.

thickness is fabricated using a conventional FIB sample preparation technique, about half of the thin film is a damaged layer.

Although only an InP system device was studied in this paper, it is expected that similar effects are achieved with other materials.

3. FIB PROCESS TECHNIQUE FOR AES ANALYSIS

Sputter depth profiling using AES is an effective technique when multilayer films consisting of layers each with a thickness of several tens of nanometers are evaluated. Since sputter depth profiling outputs one-dimensional data only, however, it is impossible to execute a precise analysis of complicated samples, such as particles in multilayer films, which require two- or three-dimensional analysis.

Although there have been direct analysis techniques of a cross-section exposed by mechanical polishing or crater edge, it is difficult to expose specified sections using these techniques. The FIB technique then makes it possible to fabricate and expose the specified cross-section to be observed. In addition, several cross-sections from one sample can be fabricated. More information can be obtained from the exposed section by combining qualitative analysis with line analysis and elemental maps using AES.

3.1 FIB Process

If the thickness of one layer is several tens of nanometers, it is difficult to analyze clearly the cross-sections exposed vertically by the FIB process because the AES spatial resolution is about 15 nm. The thickness of layers are increased by fabricating diagonally with FIB as shown in Figure 4. For instance, when a Ga ion beam is irradiated from the vertical direction of 80 degrees, the exposed section is increased about six times, and can be analyzed using AES.

Because the sample stage in the FIB chamber could be inclined only up to 60 degrees, a sample was fixed to the sample stand with a slope of 45 degrees so that the sample could be inclined by more than 60 degrees.

The surface layer is cut down through the flare of the Ga ion beam due to current density distribution in the ion beam. To avoid this problem, we coated Pt-Pd or carbon layer on the surface of the sample before the FIB process.

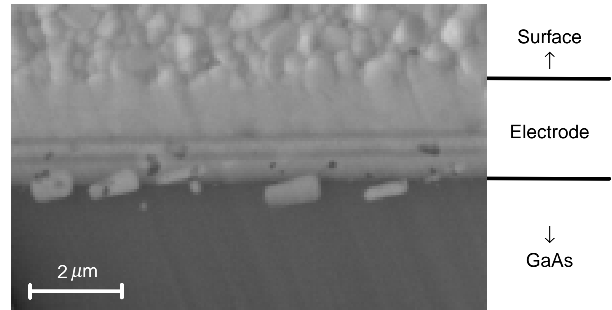


Photo 4 Cross-sectional SEM images of electrode after FIB processing and ion sputtering.

The FIB process conditions are shown below.

FIB: Hitachi FB2000A
Acceleration voltage: 30 kV
Ion beam: Ga^+
Diagonal processing: 80 degrees
Coating: Pt-Pd

3.2 Removing Damaged Layers

A damaged layer is formed on the sample surface after the FIB process as shown by TEM observation. Ga is detected on the cross-section in the AES analysis after the FIB process because the damaged layer is implanted Ga ion. Then the cross-section is sputter-cleaned using an Ar ion beam in the AES chamber to remove the damaged layer with Ga. Steps are formed because sputter rates depend on the material of each layer, thus SEM images become clear and specification of analysis area is facilitated. The sputtering conditions are shown below.

AES: PHI670
Acceleration voltage: 4 kV
Ion beam: Ar^+
Sputtering time: 3 min.

3.3 Results

The Au/Ge/Ni multilayer film for the electrode, which is deposited on a GaAs substrate, is processed by the procedure mentioned above, and is inclined in the AES chamber so that the cross-section is vertical for the electron beam. The AES analysis conditions are shown below.

AES: PHI670
Acceleration voltage: 20 kV
Sample current: 10 nA
Sample: AuGeNi electrode.

A SEM image of the electrode deposited on a GaAs substrate after FIB process is shown in Photo 4. This electrode with an AuGeNi layer is given an ohmic property by annealing. Intermetallic compounds are seen in metal/semiconductor interface and electrode. The rough area at the upper part of the SEM image is the sample surface. Photo 5 shows the SEM image and results of elemental mapping of Au, Ge, and Ni in part of AuGeNi/GaAs interface. Diffusion of Au and behavior of Ge and Ni in the

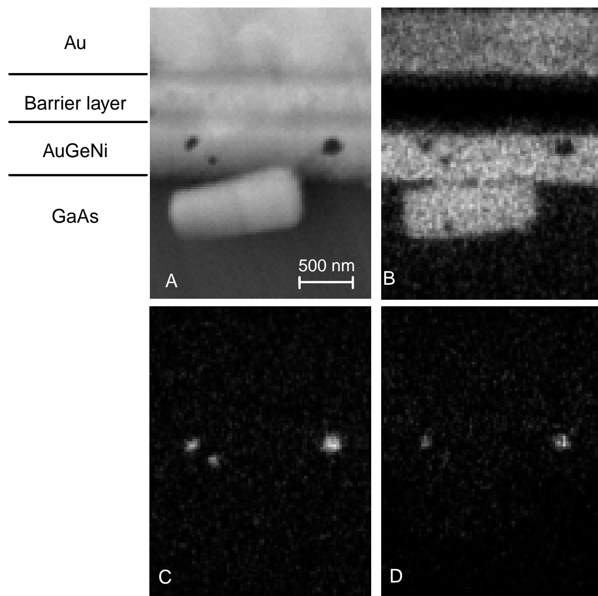


Photo 5 **A) SEM image of electrode/semiconductor interface. B) Au elemental map. C) Ni elemental map. D) Ge elemental map.**

metal/semiconductor interface could be observed clearly from the elemental mapping results. The three layers on AuGeNi layer are barrier layers to prevent diffusion of Au, Ge, and Ni, and the film thickness of the entire barrier layers is about 100 nm. These layers are seen to be about 600 nm layers in Photo 5. Therefore, it can be confirmed that the layers increased in size six times.

4. CONCLUSION

A method of FIB sample preparation for TEM observation and AES analysis was established. The method described in this article is expected to contribute to the development of devices and materials, which involve fine structures such as laser devices.

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2) K.Mogi, T.Iizuka and M.Suzuki: J. Surface Analysis, 7, 2000, 228 (in Japanese)