Use of Low Energy Ions for Removal of Damaged Layer of Cross-sectioned Specimen Prepared by Focused Ion Beam

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(Received November 1, 2000; accepted November 21, 2000)

A focused ion beam (FIB) system attached to a compact floating type low energy ion gun (FLIG) has been developed to remove an ion induced damaged layer of a specimen prepared with an FIB for cross-sectional observation under a transmission electron microscope (TEM). The thickness of the damaged layer induced on a Si (111) surface with 25 kV Ga^+ ions was assessed with TEM to be ~24 nm. This damaged layer was reduced to ~3.5 nm by finishing with low energy Ar^+ ions of 200 eV.

Sputter-etching with low energy ions of ~100 eV has been strongly required for depth profiling of such item as shallow dopant semiconductors. For this we have developed a compact floating type low energy ion gun (FLIG) which produces 100 ~ 500 eV ions with high current intensity of ~ μA and high stability, $\Delta I/I < 0.01$ for 12 hours [1]. In present works, we applied this FLIG for reducing the damaged layer on a cross-sectional specimen prepared by a focused ion beam (FIB).

An FIB technique has come into wide use as an effective tool for preparing cross-sectional specimens for observation under a transmission electron microscope (TEM) [2]. An advantage of using the FIB technique to fabricate TEM specimens is that preparation can be restricted to a specific selected area of sub-micron size for such analysis of a failed part in a semiconductor device. However, it is reported that bombardment by a high energy FIB induces the formation of a thick damaged layer on the specimen [3]. To reduce the thickness of the damaged layer, the use of incident ions of a lower energy has been often attempted [4, 5]. Practically, sputter etching with lower energy ions requires a longer processing time because of the lower sputter rate. Therefore, development of a low energy ion gun that provides high ion current intensity is essential to reduce the thickness of the damaged layer by ensuring a high ion etching rate.

This paper presents an FIB system attached to an FLIG system, which has been developed to remove an FIB-induced damaged layer from a specimen. Using this present system, the cutting and finishing processes of TEM specimen fabrication can be made without exposure to the air. The one of advantages offered by the use of this system compared with chemical etching is related to the suppression of impurities resulting from the chemical reaction. This system has enabled the thickness of damaged layers on Si surfaces to be reduced by one order of magnitude, approaching the preparation of a damage-free specimen for TEM observation.

A schematic of the FIB-FLIG apparatus is shown Fig. 1 (a). The FIB column consists of a Ga liquid metal ion source and two electrostatic lens systems. The details of the FIB system have already been described elsewhere [6].

The FLIG system consists of a permanent magnet aided electron impact type ionization cell, an extractor, and a cylindrical retarding immersion lens system. The length of the ion gun is as short as 24 cm. An Alnico-8 permanent magnet with an inner diameter of 12 mm, an outer diameter of 26 mm, and a height of 24 mm was mounted in the ionization cell, increasing the ionization efficiency by two orders of magnitude more than same ion gun without a permanent magnet. The magnetic field of the central axis is ~0.2 T, two times greater than that of the previous device [7-9]. A rhenium-filament is used as the electron source to generate reactive ions (e.g., oxygen ions) for reactive ion etching. In this study, however, we simply

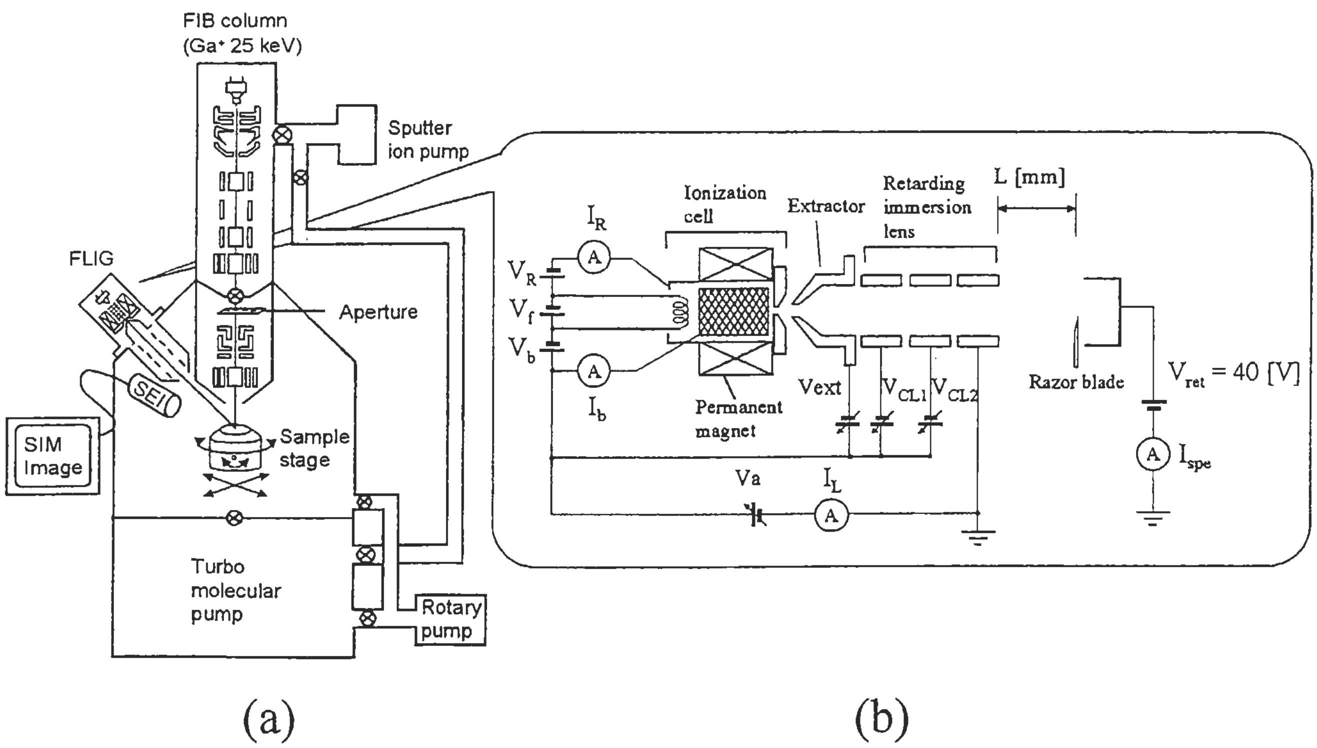

Figure 1. Schematics of (a) FIB-FLIG apparatus for TEM specimen fabrication and (b) developed compact FLIG

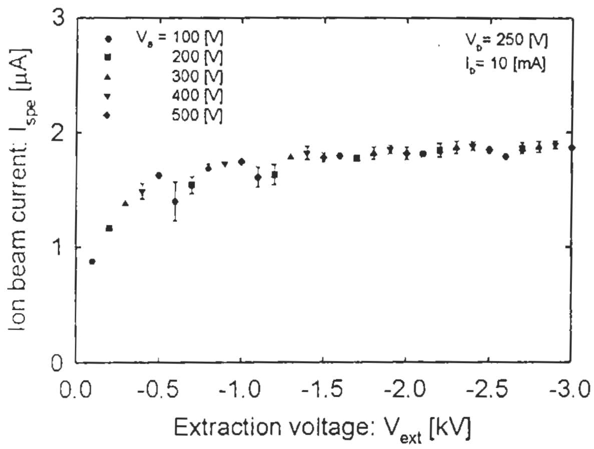

Figure 2. Ion beam current versus extraction voltage, V_{ext}, for acceleration voltages ranging from 100 to 500 V

used Ar for practical assessment of performance. As a means of obtaining a high beam current density at a low acceleration voltage, the ions generated in the cell are first extracted by an electrostatic potential ranging from −0.1 to −3 kV, then decelerated through the cylindrical immersion lenses. The entire system is so compact that it can easily be attached to conventional surface analytical instruments through an ICF-70 conflat flange. The present FLIG enables a high current intensity of μA even in a low energy region down to 100 eV.

Figure 2 shows a typical performance of ion beam current I_{spe}, vs extraction voltage, V_{ext}, for different acceleration voltages, V_a, ranging from 100 to 500 V. The gas species was Ar (99.99%) and the gas pressure in the ionization cell was 5×10^{-2} Pa, which leads to a the specimen chamber pressure of approximately 1×10^{-4} Pa. For all measurements, the electron bombardment voltage, V_b, was fixed at 250 V and the bombarding current, I_b, for ionization at 10 mA. To remove the effects of secondary electrons in the measurement of the beam

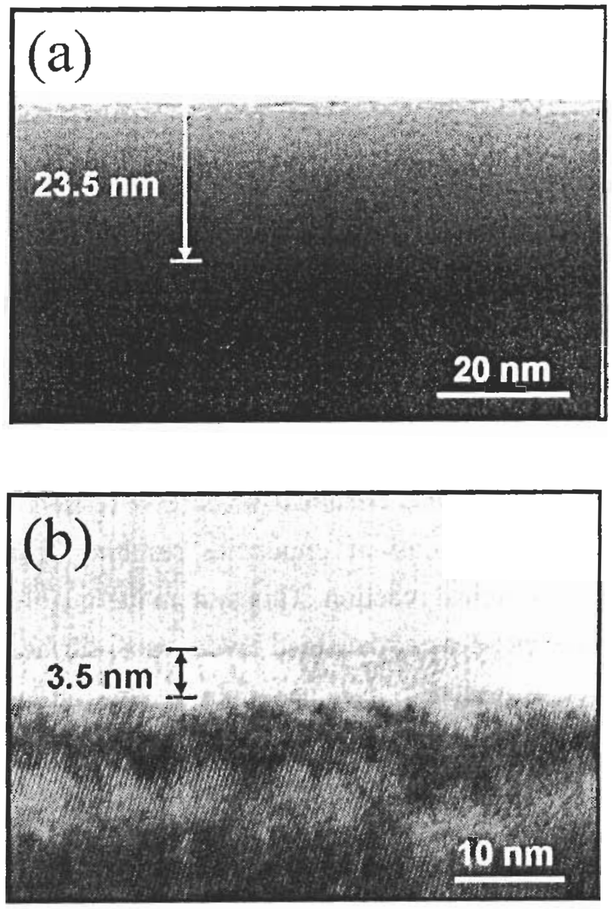

Figure 3. TEM images of (a) damaged layer on Si surface induced by Ga^+ ions at 25 kV and (b) 200 eV Ar^+ ions after 3 hours of finishing at an incident angle of 30 degrees

current, the specimen stage was biased at +40 V. For the acceleration voltage, V_a, of 100 V, the bias voltage was set to 0 V to prevent the electrostatic field around the specimen stage from disturbing the ion beam. The maximum ion beam current at V_a = 100 ~ 500 V was found to be 1.81 ~ 1.91 μA at the optimized V_{ext}. Under these conditions, the load current, I_L, remained at ~2.2 μA. This result clearly indicates that, even when the acceleration voltage goes down to 100 V, the present FLIG enables extraction of the ions generated in the ionization cell onto the specimen surface at a high efficiency, I_{spe}/I_L, of ~82%.

Applicability to preparation of the

cross-sectioning TEM specimen has been assessed by observing the damaged layer under high resolution TEM. First, a Si (111) wafer was twisted, then cleaved to a tiny section of less than 0.5 mm^3. This tip of the cleaved Si was thinly shaved by FIB irradiation with 25 keV Ga^+ ions of 230 pA for TEM observation. TEM observation was performed with a 200-kV TEM, JEM-200CX. The thickness of the damaged layer observed from Si <110> direction was over 24 nm. This is shown in the TEM micrograph shown in figure 3 (a). This photograph was taken at a direct magnification of 2×10^5. The thickness of the damaged layer was evaluated on the lamella; the average value of the portion was ~ 20. The photograph shows a typical damaged layer with thickness. The specimen was next finished by bombardment with 200 eV Ar^+ ions at 15 $\mu A/cm^2$. The incident angle of the Ar^+ ions was set at 30 degrees. The dose of Ar^+ ions was 1×10^{18} cm^{-2}. After the finishing, the thickness of the damaged layer was reduced to ~3.5 nm, as seen in the TEM 4×10^5 direct magnification micrograph shown in figure 3 (b). The etching rate in the damaged layer was ~ 12 nm/h when 200 eV Ar^+ ions were used. Therefore, we considered that the layer damaged by FIB to be completely removed, and that the damaged layer in figure.3 (b) was newly formed by a 200 eV Ar^+ ion bombardment with possible contaminated layer on it by expose to air. Al-Bayati et al. reported on the thicknesses of damaged layers of Si (100) induced by Ar^+ ion bombardment measured by the medium energy ion scattering (MEIS) method [4], stating that for an Ar^+ ion energy of 210 eV at a 1×10^{16} cm^{-2} dose with normal incidence, the thickness of the damaged layer was ~3.3 nm. The present results agree with their result quite well. It is expected that the thickness of the damaged layer can be reduced even more by optimizing the incident angle and other species of using primary ions.

In conclusion, an FIB system with FLIG attached has been developed that enables removal of an ion induced damaged layer from specimens prepared for cross-sectional observation under TEM. The present system reduced the damaged layer by approximately one order of magnitude after finishing with 200 eV Ar^+ ions. Of the finished specimen, a 3.5 nm thick damaged layer remained because the surface suffered further oxidation by being exposed to the atmosphere before it was placed in a TEM for observations. This type of oxide layer formation on silicon is considered to be ~1 nm [10]. Therefore, we deduced that the thickness of the original damaged layer was less than the 3.5 nm observed under TEM.

Optimization of the incident angle and the use of low energy ions of other species are currently under examination, and a report is to be published shortly.

Acknowledgments

The support for T. M. by the Japan Society for the Promotion of Science (JSPS-RFTF96R1410) is gratefully acknowledged.

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Reviewer's comments

Reviewer: Tetsu Sekine (JEOL)

The damage induced on a TEM lamella due to FIB cutting is a problem that must be solved for higher resolution observation. The authors have shown an advanced approach for reducing the damage area. They have attached a specially designed low energy ion gun, which is enabling higher current density, to FIB system and shown that the damaged layer is greatly reduced by milling lamella surface with it after FIB cutting.

Comments:

C1) The authors named the ion gun "FLIG" as floating type. However, as far as we see in Figure 1 (b), it is not the so-called floating circuit. I wonder this naming might lead misunderstanding to readers.

A1) It was our serious mistake. We have corrected the schematic diagram for the floating type. Thank you very much for pointing this.

Questions:

Q1) Is there any possibility that higher current ion beam, even if it is low energy, gives damage to the lamella such as distortion, bending, crack and so on?

A1) So long as our experiments on Si and GaAs with current intensity ~1 μA are concerned we have not found such marked damages as distortion, bending, crack and so on. However, some experiment draw the attention to such a damaging, as formation of protrusions and/or island formation on a surface under bombardment with low energy ions of high current density. (See for instance; "Establish Accuracy in Ultra-Shallow Profiling", M. G. Dowsett, J. Bellingham, S. Al-Harti, B. Guzmàn, T. J. Ormsby, G. A. Cooke, and C. McConville *in Proceeding of The Second International Symposium on SIMS Related Techniques* (J.S.P.S. 141 Committee, Seikei Univ., Tokyo, 2000)

Q2) The thickness of damaged layer might be depending on the height of lamella wall, namely at the top, middle, and bottom. Which portion has been taken out and evaluated?

A2) The thickness of the damaged layer was evaluated at the top of the lamella, where the average value of the portion was ~ 20. The photographs show a typical damaged layer having an average thickness.

Q3) Related for Figure 3, milling with 200 eV Ar^+ ions for 3 hours corresponding to remove 24 nm thickness or much more than that?

A3) The etching rate in the damaged layer was ~ 12 nm/h when 200 eV Ar^+ ions were used. Therefore, we have judged that damaged layer caused by FIB processing was completely removed. Then the damaged layer shown in figure.3 (b) was formed anew by 200 eV Ar^+ ion bombardment with possible contaminated layer on it by expose to air.

Reviewer: Hirofumi Matsuhata
(Electrotechnical Laboratory)

In this paper, authors' group described the development of a new FIB machine attached with a low energy ion gun, and they investigated the effect of the low energy ion gun to remove the damage layer induced by the FIB. The results are reasonably good, and this paper is worth to publish in Journal of Surface Analysis. However, I recommend improving the manuscript considering the following point. There are some other methods to remove the damage layer, such as ordinary ion etching, chemical etching etc. We can use these etching techniques after FIB process, and the results are often not bad.

Questions:

Q1) What is the advantages of this new machine compared with the method of ordinary ion etching, or chemical etching after ordinary FIB process. Probably there are many advantages.

A1) The main advantage the present FLIG is to be used for shallow dopant depth profiling, which requires very high depth-resolution. At present to our knowledge, the FLIG is considered the most promising tool to for it. (ref. [1]) Using this new machine, the cutting and finishing processes of TEM specimen fabrication can be made without exposure to the air. The one of advantages of using this machine compared with chemical etching is the suppression of the introduction of impurities caused by the chemical reaction in the process.
Thank you very much for helpful suggestion. We have added the sentence in our paper. We also add the sentence in the beginning of the paper, describing the main purpose of the development of FLIG.

Q2) What is the advantages of the low energy ion process compared with ordinary ion etching inside a chamber of the FIB machine after an optimization of incident angle. Probably there are some.

A2) A few kV energy ions are widely used in the surface analysis. In this case, however, atomic mixing causes the damaged layer which is ~5 nm thick in GaAs/GaAsAl super lattice structure specimen even at the optimum angle of incidence (Kazuo Kajiwara, Doctorate Thesis, Faculty of Engineering, Osaka University, 1992.). The thickness of the damaged layer can be primarily reduced by lowering ion energy.