Compositions of Cross Sections Created with a Gallium Focused Ion Beam

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(Received October 18 1998; accepted January 6 1999)

The surface composition of the cross sections created with a gallium focused ion beam was compared with that of raster-scanned surface. *In situ* Auger measurements were performed to clarify three main factors (implantation of bombarding species, redepositon of substrate material and preferential sputtering) of composition alteration due to ion bombardment. For all of these factors, results showed that the shave-off cross-sectioning is more favorable than the raster scan to reduce compositional alteration.

1. Introduction

Among charged particle beams a gallium focused ion beam (Ga-FIB) has characteristics of small diameter, high density and momentum transportation, which enable unique application to micromachining as well as high spatial resolution SIMS. In such applications physical and chemical damage on the sample by the ion irradiation is of key issue to evaluate usefulness of each application. Physical damage introduced by the irradiation limits the usage of, e.g., the products machined by Ga-FIB or reliability of the TEM image obtained from Ga-FIB-prepared specimen. Chemical damage, i.e., compositional alteration by ion implantation, preferential sputtering or redeposition of sputtered species, changes properties of the device material and distorts analytical results.

Recently Ishitani *et al.* [1] reported simulation results of Ga-FIB bombardment damages on multilayer samples. They pointed out that less physical and chemical damages are expected when incidence of ions is more glancing. In the "shave-off" mode [2] of Ga-FIB scanning, ion incidence is kept extremely glancing condition. During ion bombardment, generally, three kinds of phenomena are major cause of composition alteration: implantation of bombarding species, redepositon of sputtered material, and preferential sputtering. The purpose of this study is to clarify experimentally the alteration of surface composition caused by the Ga-FIB bombardment.

2. Experimental

All the experiments in this study were performed using an ion and electron dual focused beam apparatus developed by our group [3]. The apparatus was designed for three-dimensional Auger microanalysis using a combination of Ga-FIB cross-sectioning and cross-sectional Auger mapping. Therefore, *in situ* Auger measurements can be performed immediately after the cross-sectioning. The minimum spot diameter of the Ga-FIB and the electron beam were 0.1 μm (25 keV, 60 pA) and 1 μm (4 keV, 1 nA), respectively. A cylindrical mirror analyzer was used for electron energy analysis with a resolution of 1.2 %.

For examination of Ga implantation effect, Si(110) single crystal was used. Auger spectra were obtained from a heavily raster-scanned surface and a cross section. The heavily raster-scanned surface was obtained by raster-scanning the Ga-FIB of 20 keV energy and 400 pA current over the area of 50 µm × 50 µm with 45 ° incidence for 45 minutes. The cross section was obtained by shave-off scanning the Ga-FIB of the same condition. For examination of redeposition and preferential sputtering, a few µm particles of Cu-Ni alloy (nominal composition was $Ni_{0.3}Cu_{0.7}$) supported on an In plate were used. Auger spectra were obtained from a raster-scanned surface and a cross section.

Auger spectra were acquired in an $E \cdot N(E)$ form with an electron beam of 5 keV energy and 15-21 nA current. After the experiments, spectra were first smoothed four times by Savitzky-Golay method (15 points). Spectra in a form of $N(E)$ were obtained by dividing $E \cdot N(E)$ by E. In quantitative analysis, peak-to-peak intensities in $dN(E)/dE$ spectra derived from $N(E)$ using 15 points smoothing-differentiation were used. Published relative sensitivity factors [4] were used for quantification.

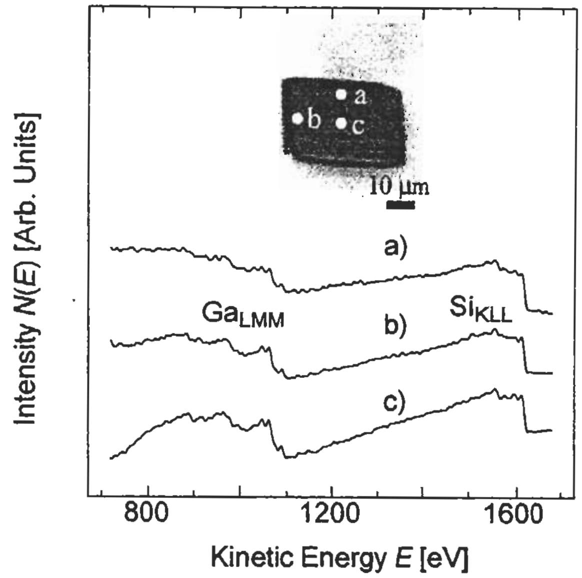

Figure 1: Auger spectra taken from points **a**, **b** and **c** within a raster etched crater on a Si(110) surface.

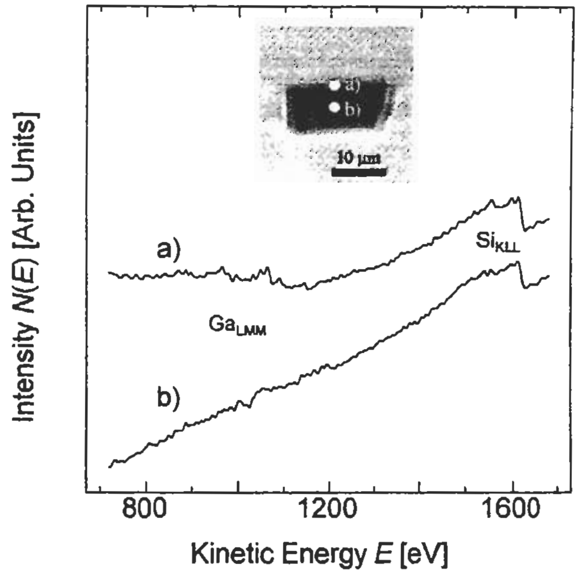

Figure 2: Auger spectra taken from points **a** (top-edge of the cross section) and **b** (center of the cross section) within a shave-off cross section in a Si(110).

3. Results and Discussion

3.1 Gallium implantation

Figure 1 shows Auger spectra obtained at different points in a crater bottom of the heavily raster-scanned Si(110). In all of three spectra intense Ga_{LMM} peaks as well as Si_{KLL} were observed. From peak-to-peak intensities in $dN(E)/dE$ spectra (not shown) implanted Ga atomic concentration relative to Si were calculated as (**a**) 0.12, (**b**) 0.18 and (**c**) 0.18.

Onset photo of Fig.2 shows the image of the cross section created by the shave-off scan of Ga-FIB viewed from the direction of the electron beam. Auger spectra were taken at points **a** (top edge of the cross section) and **b** (center of the cross section). From point **a**, Ga_{LMM} peaks were still observed, though much smaller than those in Fig.1. On the other hand no apparent peaks were observed in the spectrum from point **b**. Atomic concentrations of Ga relative to Si at points **a** and **b** were 0.19 and less than 0.05, respectively. During

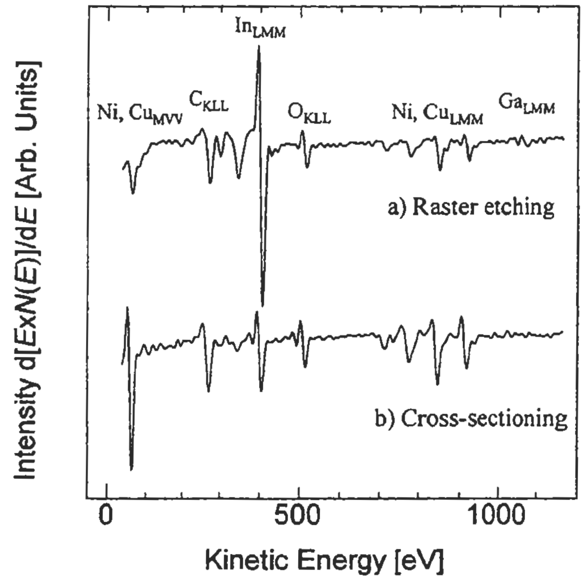

Figure 3: Auger spectra from a) raster etched surface and b) shave-off cross section.

the shave-off cross-sectioning incidence angle of Ga ions with respect to the cross section plane is estimated to be less than 3 ° [5,6]. These results shows, therefore, in such an extremely glancing condition implantation of bombarding species is reduced to a very low level. Higher concentration at the top edge of the cross section is due to higher incidence angle of Ga ions in a beam circumference.

3.2 Redeposition of sputtered material

Figure 3 shows Auger spectra obtained from surface of the raster-scanned particle (**a**) and the cross-section created by Ga-FIB shave-off scan (**b**). Very intense peaks of $\mathrm{In_{LMM}}$ in (**a**) were originated from redeposited In on the raster-scanned surface. Strongly suppressed MVV peaks of Ni and Cu indicated that the surface was almost covered with In. $\mathrm{Ga_{LMM}}$ peaks were also detected. From the cross section $\mathrm{In_{LMM}}$ peaks were still observed but with much less intensity. Redeposition was reduced by a factor of 3 or more.

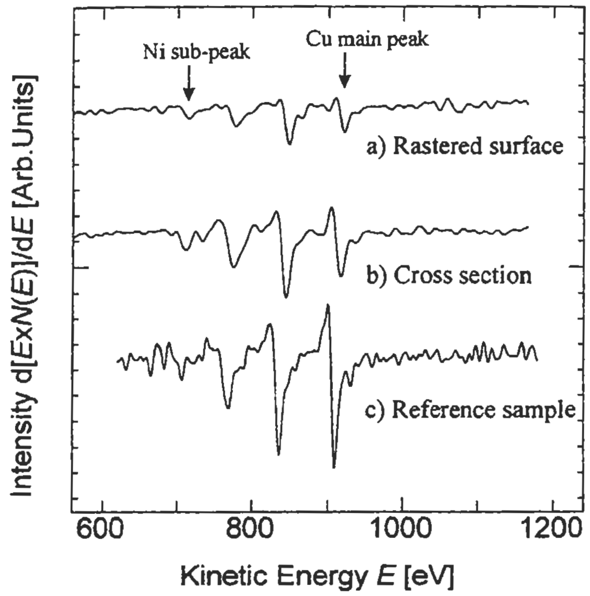

Figure 4: Auger spectra from a) raster etched surface and b) shave-off cross section and c) a reference sample consisting of a Cu sheet and a Ni sheet with area ratio of 7:3.

3.3 Preferential sputtering

Figure 4 shows Auger spectra from the raster-scanned surface (**a**), the cross section created by Ga-FIB shave-off scan (**b**) and the reference sample consisting of a Cu sheet and a Ni sheet with area ratio of 7:3 (**c**). Since the main peak of $\mathrm{Ni_{LMM}}$ is overlapped on a $\mathrm{Cu_{LMM}}$ subpeak, the intensity of $\mathrm{Ni_{LMM}}$ was estimated from the Ni subpeak and the intensity ratio obtained from pure Ni sheet. Five particles for the raster scan and three particles for cross-sectioning were analyzed. Figure 5 summarizes atomic concentration ratios of Ni to Cu. In the case of rastered surface, the ratio was about three times larger than the nominal ratio. This means that Cu was preferentially sputtered as well known [7]. As for the save-off cross-sectioning, the ratio was still larger than the nominal one. However, discrepancy was smaller than that in the raster scan. Since it was very difficult to measure the true concentration ratio before Ga-FIB bombardment, we could not evaluate the reliability of the nominal ratio for this kind of discussion. However, it can be said that the preferential sputtering effect in the shave-off cross-sectioning is smaller than in raster scan.

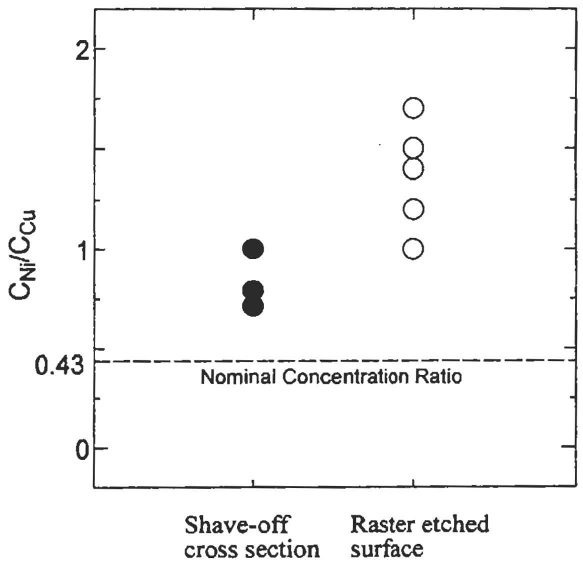

Figure 5: Atomic concentration ratios (C_{Ni}/C_{Cu}) obtained from shave-off cross sections and raster etched surfaces. The nominal ratio (0.43) before Ga-FIB bombardment is derived from sample specification data.

4. Conclusion

From the comparison of Auger spectra obtained from the raster-scanned surface and the shave-off cross section, we reached the following conclusions: 1) implanted Ga concentration in the cross section is around detection limit of Auger electron spectroscopy, typically a few atomic percent or less, 2) redeposition of sputtered substrate material is greatly reduced in the cross-sectioning, and 3) composition change due to preferential sputtering in binary alloy is also reduced.

These features of the shave-off cross-sectioning are quite favorable for the preparation of cross-sectional samples, while detailed study on the mechanisms of preferential sputtering is still required.

This study was supported by the Grant-in-Aid for Scientific Research #09450308, the Ministry of Education, Science, Sports and Culture, Japan.

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