Paper

Analysis of the Shape of Cross Sections Developed under Shave-off Condition Sputtering

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Shave-off method has been proven its efficacy for highly precise depth profiling in secondary ion mass spectrometry (SIMS) analysis. The unique technique, shave-off method has distinctive cross-sectional shape after scanning compared with raster scan method. We investigated the cross sectional shape of three different height tungsten samples using focused ion beam scanning electron microscopes (FIB-SEM) and transmission electron microscope (TEM). Though it is a simple cross-sectional shape, the analysis results enable the investigation of an angle between primary ion beam and sample surface, and sputtering yield.

1. Introduction

Secondary ion mass spectrometry (SIMS) is one of a mature surface analysis technique with a vast range of application. Our group has developed “shave-off method” which is a unique section processing technique for achieving highly precise SIMS depth profiling with reduction of shape effects. The procedure of conventional raster scan and shave-off scan is illustrated in Fig. 1. Unlike conventional raster scan mode of focused ion beam (FIB) which scans over an area rapidly and repeatedly in both vertical and horizontal sweeps (Fig. 1(a)), shave-off scan mode is that the primary ion beam always shaves the edge of the sample with the fast horizontal sweep and very slow vertical sweep (Fig. 1(b)) [1]. Therefore, section of the sample is shaved off flatly and almost parallel to the raster and the axis in direction of FIB, and shave-off scan has low sputtering re-deposition and restriction of primary ion implant influence. In the previous study, our group have developed dual FIB ToF-SIMS for accurate three-dimensional (3D) analysis and the shave-off method was determined that it can suppress surface damage than conventional method [2]. However, this 3D SIMS analysis method took a long process times due to use double systems composed by shave-off sectioning beam and analysis beam. Thus we suggested new 3D shave-off analysis method introducing the lens system which magnified image of the Z-axis detecting length simultaneously X and Y axis scanning in order to resolve mentioned demerits. For developing the method, firstly we need to understand the relation between primary ion beam and sample surface during the scanning. The shave-off method has a distinctive cross sectional shape compared with raster scan because of the unique scan mode. In this paper, we investigate the cross-sectional shape for obtaining information about primary ion beam and sputtering yield.

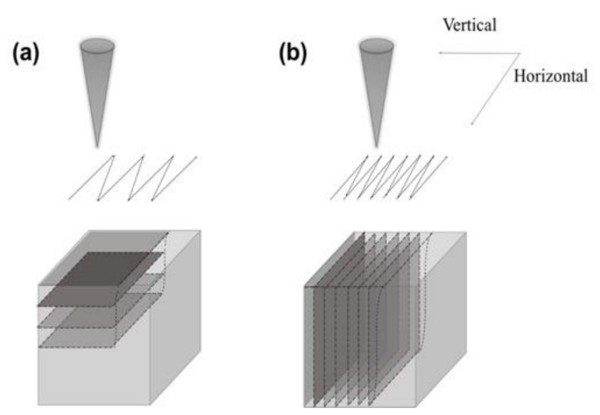

Fig. 1. The procedure of scanning. (a) Conventional mode, (b) Shave-off mode.

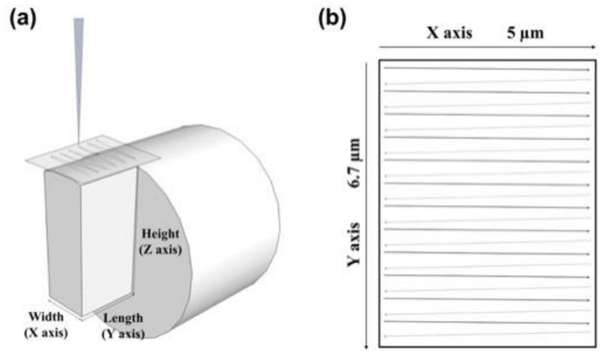

Fig. 2. (a) Schematic diagram of the shave-off scanning. (b) Scheme of the FIB scan.

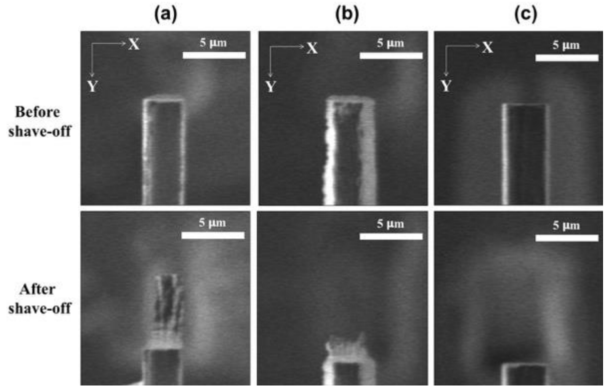

Fig. 3. Before (upper) and after (lower) SEM images of samples with height (a) 30 μm, (b) 20 μm, (c) 10 μm.

2. Experimental

A tip of tungsten wire (99.95%, Nilaco) with a diameter of 40 μm was diced into rectangular cuboid using FIB. To verify a cross section change with sample height, we fabricated three tungsten samples with different heights which has 30, 20 and 10 μm (Z axis), respectively, with 3.5 μm in width and over 6.7 μm in length as shown in Fig. 2(a). In this study, all of experiments were carried out using focused ion beam scanning electron microscopes (FIB-SEM, SII NanoTechnology Inc, SMI-3050 SE) with a gallium liquid metal ion source (LMIS). The cross-section and size of samples were measured using FIB-SEM and transmission electron microscope (TEM, JEOL, JEM-1010, operated at 100 kV). The primary ions (Ga^+) of this apparatus were operated 30 keV accelerated energy with 260 pA beam current. Nominal beam diameter was 40 nm. The FIB scanning area for experiments was 5.0 μm (X axis) × 6.7 μm (Y axis) (Fig. 2(b)). The total scanning time was 4000 s from upper to lower part of the scanning area. In the shave-off scanning, the sweep speed of beam was 5 μm/s in the horizontal direction (X axis) and 0.0017 μm/s in the vertical direction (Y axis). The number of shave-off scanning rasters was 4000 in 6.7 μm with interval length of 1.68 nm. In the raster scan the number of scanning raster was 134 in 6.7 μm with interval length of 50 nm. 1.34 s frame scan was repeated 2985 times.

3. Results and discussion

Figure 3 shows top view SEM images of the samples before and after shave-off scan. It can be observed that the sample having height of 30 μm was not completely etched (Fig. 3(a)), the 20 μm height sample remained about 2 μm from the bottom of scanning area (Fig. 3(b)). However, the sample having height of 10 μm was wholly shaved off (Fig. 3(c)) under the shave-off condition. In order to obtain a precise analysis of cross-sectional image after shave-off scan, we observed the y-z plane of the sample (Fig. 2(a)) using TEM. The cross-sectional images which are expected to have a distinctive shape are shown in Fig. 4. For quantitative analysis, we converted only a part of cross-sectional shape in the TEM images to x and y coordinate system. The composite graph of each cross sectional shape is shown for comparison in Fig. 5. The cross-sectional shape of the three different samples is considerably similar to each other. The graphs from the surface (0 μm) of the sample to 13 μm of z-axis show smooth and similar results, whereas a part of lower than 13 μm was not similar and smooth. We expected that the unusual shapes of lower 13 μm is to be the results from mixed three factors by; (1) primary beam ions (Ga^+), (2) re-sputtered Ga^+ ions from the 0 μm to 13 μm of z-axis, and (3) sputtered neutral tungsten atoms from the sample. In addition, the shave-off method was acquired a depth profile by Y direction with slow scanning. So normally the z axis of sample was considered lower than 10 μm, the average angle between primary ion beam

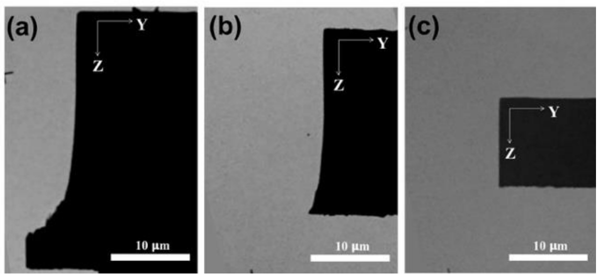

Fig. 4. Transmission electron microscopy (TEM) images of sample profile with height (a) 30 μm, (b) 20 μm, (c) 10 μm.

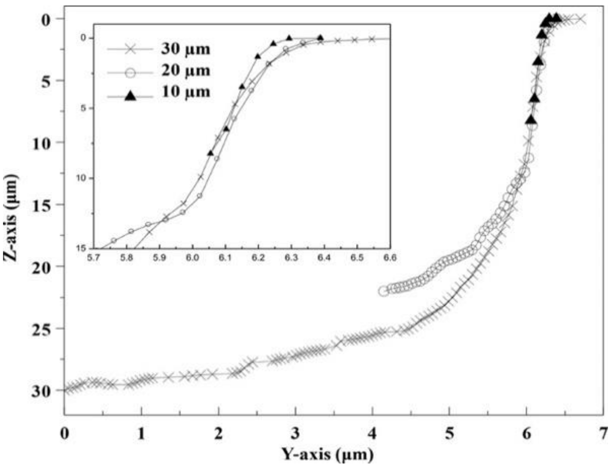

Fig. 5. A composite graph of the cross-sectional shape.

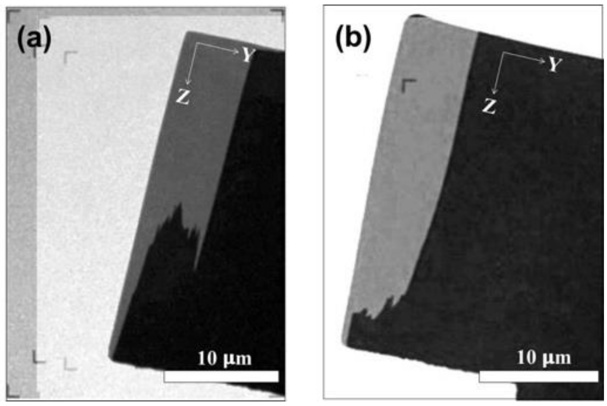

Fig. 6. A composite TEM image of before and after scanning. (a) raster scan, (b) shave-off scan mode.

and cross-sectional sample surface is calculated about 3° from the results. The angle is a considerably significant factor relative to incident angle which affects sputtering yield, secondary ion yield, and depth resolution in SIMS analysis. This value is also anticipated to be related with angular distribution of secondary ions with incident angle.

Moreover, we can compare the sputtering yields of raster scan and shave-off scan from observing sputtered out area. Figure 6 shows composite TEM images from before and after scanning. A part of the black in the figures is cross sectional shape after scanning and gray part is cross-sectional shape of the sample before scanning. The experiment was conducted in same beam condition, same scan area (5.0 × 6.7 μm^2) and scanning time (4000 s) with tungsten sample (W, height; 30 μm). The profiles of raster scan mode (Fig. 6(a)) and shave scan mode (Fig. 6(b)) were clearly different. We could expect comparison of sputtering yields from the sputtered out area calculation. The sputtered out area was calculated using ImageJ program, and the area was 100.6 μm^2 (raster scan mode) and 130.7 μm^2 (shave-off scan mode), respectively. It could be calculated 2.22×10^{13} atoms and 2.88×10^{13} atoms, the results implied that the shave-off scan mode has 1.3 times higher sputtering yield compared with raster scan mode at the same beam condition. This ratio almost agreed with our previously reported results (shave-off scan sputtering yield: 3.6 atoms/ion, raster scan sputtering yield: 2.79 atoms/ion) [3].

4. Conclusion

We investigated a cross-sectional shape of three different heights of the samples after shave-off scan using FIB-SEM and TEM. Although heights of the samples were different, the cross-sectional shape was similar to each other below a certain height. When the heights of the samples were under 13 μm, the average angle between primary ion beam and sample cross section was calculated about 3° after shave-off scan. In addition, we compared the sputtering yield of raster scan mode and shave-off scan from calculation of the sputtered-out area. Shave-off scan mode had 1.3 times higher sputtering yield than raster scan mode at the same beam condition, this result corresponds to our previous simulation results. This novel approach for the analysis of shave-off cross-sectional shape will have important functions for development of three-dimensional shave-off SIMS by providing information about the relationship between primary ion beam and sample surface.

5. References

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