Influence of the Shave-off Scan Speed on the Cross-Sectional Shape

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The shave-off scan method is a unique secondary ion mass spectrometry (SIMS) section processing technique using a gallium focused ion beam (Ga FIB). The sample had a distinctive cross-sectional shape after shave-off scanning, and the slope of the cross section related to the incident angle of the beam that affects sputtering yield. This study investigates sputtering yield as a function of shave-off scan speeds based on cross-sectional shapes examined using a scanning electron microscope (SEM). To conduct a more detailed comparison of the cross sections, the SEM images were converted to an \(X, Y\) coordinate system using an in-house program. The sputtering yield decreased as scan speed decreased and incident angle increased. This result opposed the results obtained using conventional raster scanning, and beam profiles could be predicted from the cross-sectional shapes. [DOI: 10.1380/ejssnt.2018.214]

Keywords: Ion-solid interactions; Sputtering; Shave-off method; Scan speed; Cross-sectional shape

I. INTRODUCTION

The shave-off scan mode is a unique section-processing technique that reduces the shape effects to achieve precise depth profiling. This method entirely sputters sample edges to a very slow vertical (\(Y\)-axis) sweep, which differs from the horizontal (\(X\)-axis) sweep of a focused ion beam (FIB). In the shave-off processing, the primary ion beam always keeps on the edge of the sample during the scanning. Therefore, a section of the sputtered sample is almost parallel to the axis in the direction of the FIB [1].

The sputtering yield is closely associated with various factors, such as mass, energy and incident angle of the bombarding ions. In particular, the incident angle of the primary ion beam affects sputtering yield, as well as secondary ion yield and depth resolution. As reported by Santamore et al. [2], the sputtering yield changed as a function of scan speed in raster scan mode. It is due to the angle between the ion beam and the sample surface. They achieved results that the slower scan the larger the angle, and the larger the angle the larger the sputtering yield.

Previous work concerning the scan speed of shave-off scan has been carried out that the speed influenced depth resolutions. The faster the speed scan has the lower depth resolution and smaller the influence of long tails of FIB changes [3]. In this paper, we have calculated sputtering yields by investigation of the cross-sectional shape as a function of scan speed. The cross-sectional shape under the shave-off method is important relates to the incident angle. Because in case of the shave-off method, the cross section could be kept its shape after every one horizontal sweep without re-deposition. Thus the incident angle is decided by the slope of cross-sectional shape which made by a single sweep as shown in Fig. 1.

II. EXPERIMENTAL

All experiments in this study were performed using a focused ion beam scanning electron microscopes (FIB-SEM, SH NanoTechnology Inc, SMI-3050 SE) operating at \(4 \times 10^{-6}\) Pa residual gas pressure. A gallium liquid-metal ion source was used to generate an ion beam with 30 keV accelerated energy. The nominal beam size was 40 nm for probe current of 260 pA.

A. Sample preparation

Tungsten samples were prepared using a two-step FIB-milling process. First, the tip of a tungsten wire (99.95%, Nilaco) with a diameter of 40 \(\mu\)m was diced into cuboid shapes with a 3.5 \(\mu\)m (\(X\)-axis) and over a 6.7 \(\mu\)m (\(Y\)-axis). The sample was then rotated 90°, and the bottom part was cut to adjust sample thickness (30 \(\mu\)m, \(Z\)-axis). For a detail explanation of this process was written in our previous paper [4].

B. Scan speed

In the digital scan mode of the FIB, the beam was controlled by moving pixels arranged on the \(X\)- and \(Y\)-axes. This allowed scan speed to be adjusted by changing the

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TABLE I. The conditions of shave-off scan mode.

|                      | (a) Standard | (b) 2 times faster | (c) 2 times slower |
|----------------------|--------------|-------------------|-------------------|
| X interval length    | 10           | 10                | 10                |
| Y interval length    | 1.68         | 3.35              | 0.84              |
| Dwell time           | 2000         | 2000              | 2000              |
| Horizontal speed (X) | 5            | 5                 | 5                 |
| Vertical speed (Y)   | 1.68 $\times 10^{-3}$ | 3.35 $\times 10^{-3}$ | 0.84 $\times 10^{-3}$ |
| Total scanning time  | $4 \times 10^3$ | $2 \times 10^3$    | $4 \times 10^3$    |
| Scanning area (X x Y)| 5.0 x 6.7    | 5.0 x 6.7         | 5.0 x 3.35        |

C. Error factor

The experiment was conducted three times at each scan speed, and the shave-off method gave comparable and repeatable results. The angle of incidence was measured with the trace of the beam as $90^\circ$. The traces made by the FIB were left on the sample. The error factor of the angle from the width of the mark is about 0.007$^\circ$.

III. RESULTS AND DISCUSSION

A. Cross-sectional shape

Figure 2 shows the SEM images of the cross-sectional shapes after shave-off scanning at three scan speeds. Each of the cross sections was different in shape and removed the depth of the sample. Standard scan speed (a) and two-times faster scan speed (b) resulted in the bottom of the samples being 4 $\mu$m and 14 $\mu$m, respectively. In contrast, a two-times slower scan speed (c) entirely removed the 30 $\mu$m of the sample depth by the primary ion beam. This is due to the low number of sputtered atoms per scan area at fast scan speed. Thus, the cross-sectional shape had a shallower depth at the faster scan speeds. This result indicates a relationship between scan speed and sputtered atoms in the shave-off scan mode.

The cross-sectional SEM images into a graph using a program made in-house based on the Visual Basic software for comparison. In our previous report [4], the bottom part of the cross-sectional shape was affected by the primary ion beam and other factors. Therefore, only part of the 10 $\mu$m depth was converted from the sample surface to a graph of the cross-sectional shape. The graph of the cross-sectional shape is shown in Fig. 3.

In Fig. 3, which shows the surface position of the sample at 0 $\mu$m of the Z-axis. Each final scan position differed due to the differences in Y-interval length, thus, graphs for each of the highest slope positions were compared. This revealed that the slower scan speed increased the slope of the cross sections. Therefore, the slope of the cross-sectional shape was a function of scan speed and referred to the angle of incidence between the primary ion beam and the sample. These results will be discussed further in Sec. III C.

B. Beam profile

The erosion rate was affected by incidence ion flux, sputtering yield, and number density of the sample.
Therefore, we could assume the primary beam profile from the cross-sectional shape. Figure 4(a) is a derivative graph of Fig. 3. To compare graphs, the peak of the derivative graph moves as 0 μm as shown in Fig. 4(b). The peak of the graph shows that increased with the slower the scan speed. The height difference between the standard and the two-times-faster scan speeds is equal to the difference between the standard and the two-times-slower scan speeds. This result indicates that the sputtering yield changes according to the scan speed. If the sputtering yields were the same at each scan speed, the peak of the two-times-faster scan speed should have been twice as high as the standard scan speed, and the peak of the two-times-slower scan speed should have half of the peak of the standard. However, the highest peak in the experiment has the value of only 1.13 times higher and 0.85 times lower than standard, and the value difference is almost the same. It is concluded that the fact that the faster the scan speed, the higher the sputtering yield.

Figure 4(c) presents normalized curves at the maximum height of Fig. 4(b). In the experiments, although the shave-off scan speeds were different from each other, the condition of the primary ion beam was always the same at 260 pA. Thus all three derivation graphs coincide as shown in Fig. 4(c).

According to a report by Vladov et al. [5], the effective footprint of a beam which causes damage to the surface is larger than the physical size of the beam, and they defined the effective footprint as an apparent beam size. From the results shown in Fig. 4(c), we can assume the apparent beam size which has effectively influenced on the sample. A full width at half maximum (FWHM) was measured to be 0.15 μm, and this was larger than the nominal beam diameter (40 nm, physical size).

C. Sputtering yield

It is well known that the erosion depth and sputtering yield depend on the scan speed [2, 6–8]. From the observation of cross-sectional shape, we could assume that the sputtering yield was changed as a function of scan speed. The shave-off method always keeps the FIB at one edge of the sample during the scanning, and the cross section can be maintained its shape after each horizontal sweep without re-deposition. Thus we can use a surface erosion calculation which does not taken into consideration the re-deposition and reflection of sputtered atoms. The

\[
J = \frac{N \tan \theta}{S(\theta)}
\]

Here, \(J\) is the ion flux (ions/μm²/s), \(V\) is the beam scan speed (μm/s), \(N\) is the atomic density of the target (atoms/μm³), and \(S(\theta)\) is the sputtering yield (atoms/ion). The ion flux from the primary ion beam is \(2.32 \times 10^9\) ions/μm²/s, and the atomic density of W is \(6.3 \times 10^{10}\) atoms/μm³.

As mentioned above, the angle of incidence was associated with the cross-sectional shape using the shave-off method. Figure 5 presents the angles between the primary ion beam and cross-sectional shape. The angle of incidence (θ) was measured at each peak (0.25 μm, 0.10 μm, and 0.22 μm on the Y-axis) of Fig. 5 and the sputtering yield was calculated from Eq. (1). Table II presents the sputtering yields and the angles of incidence. Although it is a small difference, the angle of incidence decreased with faster scan speed.

For the raster scan method, a slower scan speed has a larger the angle of incidence and a larger the angle leads to increased sputtering yield [2]. However, this study shows that the sputtering yield decreases with decreasing scan speed. If the ratio of the sputtering yield of the standard condition is set to 1, the sputtering yield at a two times faster scan speed is about 1.71 times higher, and
TABLE II. The angle of incidence at steepest position on the cross-sectional shape and calculation of sputtering yield as function of scan speed.

| Vertical speed (V) | Angle of incidence (θ) | Sputtering Yield [S(θ)] | Ratio |
|-------------------|------------------------|-------------------------|-------|
| μm/s              | °                      | atoms/ion               |       |
| 1.68 × 10^{-3}    | 88.84                  | 2.27                    | 1     |
| 3.35 × 10^{-3}    | 88.65                  | 3.87                    | 1.71  |
| 0.84 × 10^{-3}    | 88.98                  | 1.28                    | 0.57  |

FIG. 6. Sputtering yield in dependence on the angle of incidence obtained from SRIM and SDTrimSP.

the two times slower scan speed has about 0.5 times the lower sputtering yield. This contrary result is explained by the unusual incident angle of the shave-off scan mode compared with the conventional raster scan mode. The shave-off scan mode always had an incident angle above 75° as shown in Fig. 5.

To examine the sputtering yield as a function of incident angle for 30 keV Ga ion irradiation of W, we calculated the sputtering yield using the SRIM 2013 and SDTrimSP V5.7 simulations as shown in Fig. 6. The simulation results show that the sputtering yield increases with increasing incident angles until 70° (SDTrimSP) and 75° (SRIM) for the two types of simulation, respectively. At angles larger than these angles, the sputtering yield decreases sharply due to the increasing the reflection of ions [9].

In the conventional raster scan method of the FIB system, the scanning is usually operated with an incident angle under 60°. Thus the angle between the ion beam and the surface is normally increased with decreasing scan speed, and the larger angle has the larger sputtering yield. However, using the shave-off method, the slope of the sidewall has an angle of more than over the 75° as shown in Fig. 5, and it becomes the incident angle of the primary ion beam. Thus, a slower the scan speed has a smaller the incident angle with decreasing sputtering yield. This result gives the relation between the angle of incidence, scan speed, and sputtering yield using the shave-off method with Ga-FIB.

IV. CONCLUSION

In the shave-off method, the slope of the cross section is closely related to the angle of incidence due to the unique sectioning technique. The slope of the cross section increases with decreasing scan speed. And, the sputtering yield decreases with decreasing scan speed and with increasing the angle of incidence. This result is opposite to the tendency of the raster scan method because the shave-off method usually has a larger the angle of incidence than the raster scan method. In this experiment, the sputtering yield decreased for the angles of incidence larger than 75°. This is due to the increase in the reflection of ions at the surface. Although the cross sections have different shapes as a function of scan speed, the normalized derivative graphs have been shown as one composite graph which has an apparent beam size of 150 nm. The relationship between the beam profile and the cross-sectional shapes can be estimated. Going forward, we envision that this study will enable us to calculate the scan speed and angle of incidence for getting the highest sputtering yield on our shave-off method.

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