Effects of Charging and Crystallinity on Sputter-Etching Rate of Al2O3 Single Crystal

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In this study, we examined Au+ ion sputter etching rate ratio RAl2O3/RSiO2 under different surface charging conditions and in different incident azimuthal directions of Au+ ions. When the surface charging of specimen was neutralized by irradiation with low-energy electrons, RA12O3 became larger than that of specimen without any electron irradiation at same ion current density. When the specimen was bombarded by Au+ ion from the azimuthal direction parallel to the a-axis on the surface of Al2O3 single crystal, RAl2O3/RSiO2 became higher than that of specimen bombarded from the azimuthal direction vertical to the a-axis. The effects of surface charging and incident azimuthal direction of Au+ ion on RAl2O3/RSiO2 are discussed respectively on the basis of the experimental results.

1. Introduction

In AES (Auger Electron Spectroscopy), XPS (X-ray Photoelectron Spectroscopy) and SIMS (Secondary Ion Mass Spectrometry) depth profiling measurements, the sputter-etching rate, R, is a very important factor in determining the sputter depth from the sputtering time. The Sputter Etching Rate Database (SERD) project has been undertaken by the Surface Analysis Society of Japan (SASJ) to accumulate data as the ratio (RC/RSiO2) of the etching rate for various compounds, RC, to the rate for thermally oxidized SiO2 film, RSiO2 [1].

It has been reported that RGaAs is directly proportional to RSiO2 and RGaAs/RSiO2 is independent of the ion current density. However, several compounds such as Al2O3 show different behavior, with RA12O3/RSiO2 tending to decrease in the region of high ion current density. It is thought that this phenomenon is closely related to surface charging.

In this research, we determined RAl2O3/RSiO2 under different surface charging conditions and in different sputter directions. Based on the results, we discuss the effects of surface charging and the incident Au+ ion azimuthal direction on RAl2O3/RSiO2.

2. Experimental Procedure

Sapphire single crystal [α-Al2O3 (0001)] and thermally oxidized SiO2 films were prepared for these experiments. The incident azimuthal direction of ion etching was parallel to the a-axis for Al2O3 single crystal, except when a different incident azimuthal direction was specified.

Sputter-etching rates were measured with the mesh replica method proposed by Suzuki et al. [2]. Cross-sectional profiles were obtained by using a stylus profiler (Sloan, Dektak3030). The average values obtained from more than four measurements were used in plotting the profiles.

Figure 1 shows the experimental ion gun geometry and table 1 shows the experimental conditions.
3. Results and Discussion

We first determined $\text{RAIO}_x/\text{RSIO}_2$ under different surface charging conditions. Figure 2 shows $\text{RAIO}_x$ as a function of $\text{RSIO}_2$, with an increasing ion current density. In the region of a lower current density, the data confirm that $\text{RAIO}_x$ was proportional to $\text{RSIO}_2$. However, in the region of a higher ion current density, $\text{RAIO}_x$ deviated from the proportional relationship to $\text{RSIO}_2$ and $\text{RAIO}_x/\text{RSIO}_2$ decreased (●). On the other hand, when the specimen was neutralized by irradiating it with low-energy electrons, $\text{RAIO}_x/\text{RSIO}_2$ (▲) became larger at the same ion current density compared with the ratio for specimens without any irradiation.

Figure 3 shows the stylus profilometer images obtained after sputtering, where (a) is the image for a neutralized specimen. It is seen that the bottom of the sputtered zone was flat. The image in (b) for a specimen that was not neutralized shows that the bottom of the sputtered zone was semi-cylindrical. These results indicate that surface charging is a contributing factor to the decrease in $\text{RAIO}_x/\text{RSIO}_2$.

![Stylus profilometer images](image)

**Fig. 3** Stylus profilometer images of $\text{AlO}_x$ were obtained after sputtering: (a) The specimen was neutralized; (b) the specimen was not neutralized.

**Caution:** In the nearby Cu mesh, surface charging was reduced by conductivity.

![Cu mesh effect](image)

**Fig. 4** SEM images and cross-sectional profiles of the specimen surface of an insulator following irradiation with $\text{Ar}^+$ ions.

**Fig. 4** shows SEM images and cross-sectional profiles of the specimen surface of an insulator irradiated with $\text{Ar}^+$ ions. In (a), the specimen was not neutralized. In the nearby Cu mesh, surface charging decreased because...
of conductivity, so the sputter-etching rate was faster than that at locations more distant from the Cu mesh. In (b), the overall specimen surface was neutralized, so the same etching rate was obtained throughout the sputter zone.

Figure 5 shows the etching rate for Al₂O₃ as a function of the rate for SiO₂; the incident ion species was Ar⁺ and the ion energy was 3keV. Wider scatter is observed for SERD data (●) than ARC data including specimen neutralized data (△). Part of the difference between the scatter of two data is attributed to surface charging. For instance, since specimen that was not neutralized shows that sputtered zone is not flat as shown in Fig. 4, it is thought that cross-sectional profiling measurement of error is larger.

Figure 7 shows the relative etching rates of Al₂O₃ to SiO₂ in different incident azimuthal directions, either parallel or vertical to the a-axis on the surface of Al₂O₃. The parallel direction was higher than that in the vertical direction. It is thought that this shows the planar channeling effect on Al₂O₃.

At higher ion energy levels, the channeling of ions in the "open" crystal directions causes the yield to differ. For instance, in Cu at 5 keV Ar⁺ ion bombardment, the sputtering yield of a (111) surface is nearly double that of a (110) surface [3].

In this experiment, ion energy was not very high. However, R₂O₃ differed depending on whether the sputter azimuthal direction was parallel or vertical to the a-axis. This result supports the above-mentioned observation that the lattice plane affects the etching rate of a crystal.
The etching rate was then determined for an Al₂O₃ film (Al₂O₃/SiO₂/Si-sub.) obtained by sputtering deposition.

Figure 8 shows \( \text{RAl}_2\text{O}_3(\text{film}) \) as a function of \( \text{RSiO}_2 \) with an increasing ion current density obtained by the mesh replica method. Figure 9 shows the XPS depth profile of \( \text{Al}_2\text{O}_3/\text{SiO}_2/\text{Si-sub} \) obtained with a Quantam-2000 instrument (PHI), and figure 10 shows an SEM image of this sample taken with an SAM680 instrument (PHD). Table 2 shows the etching rate ratio \( \text{RAl}_2\text{O}_3/\text{RSiO}_2 \) by the mesh replica method and XPS depth profiling, respectively.

By the mesh replica method, \( \text{RAl}_2\text{O}_3(\text{film})/\text{RSiO}_2 \) was 0.59, while \( \text{RAl}_2\text{O}_3(\text{sapphire})/\text{RSiO}_2 \) was 0.45. It is thought that the difference in density between the sapphire single crystal and the film obtained by sputtering deposition affected \( \text{RAl}_2\text{O}_3 \). While \( \text{RAl}_2\text{O}_3(\text{film})/\text{RSiO}_2 \) was 0.50 by depth profiling, this tendency agree with the thermally oxidized \( \text{SiO}_2 \) film in SERD report [1].

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4. Conclusions
1) Surface charging is a contributing factor to the decrease in the etching rate observed for an insulator such as Al₂O₃.
2) It is thought that the lattice plane affects \( \text{RAl}_2\text{O}_3 \).

References