Effects of Charging and Crystallinity on Sputter-Etching Rate of Al_2O_3 Single Crystal

S. Araki, H. Tohma, and SERD Project Group of Surface Analysis Society of Japan

NISSAN ARC, LTD., 1, Natsushima, Yokosuka 237-0061, Japan

(Received May 8, 2002; accepted June 25, 2002)

In this study, we examined Ar^+ ion sputter etching rate ratio $R_{Al_2O_3}/R_{SiO_2}$ under different surface charging conditions and in different incident azimuthal directions of Ar^+ ion. When the surface charging of specimen was neutralized by irradiation with low-energy electrons, $R_{Al_2O_3}$ became larger than that of specimens without any electron irradiation at same ion current density. When the specimen was bombarded by Ar^+ ion from the azimuthal direction parallel to the a-axis on the surface of Al_2O_3 single crystal, $R_{Al_2O_3}/R_{SiO_2}$ 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 Ar^+ ion on $R_{Al_2O_3}/R_{SiO_2}$ 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 (R_C/R_{SiO_2}) of the etching rate for various compounds, R_C, to the rate for thermally oxidized SiO_2 film, R_{SiO_2} [1].

It has been reported that R_{GaAs} is directly proportional to R_{SiO_2} and R_{GaAs}/R_{SiO_2} is independent of the ion current density. However, several compounds such as Al_2O_3 show different behavior, with $R_{Al_2O_3}/R_{SiO_2}$ tending to decrease in the region of high ion current density. It is thought that this phenomenon is closely related to surface charging.

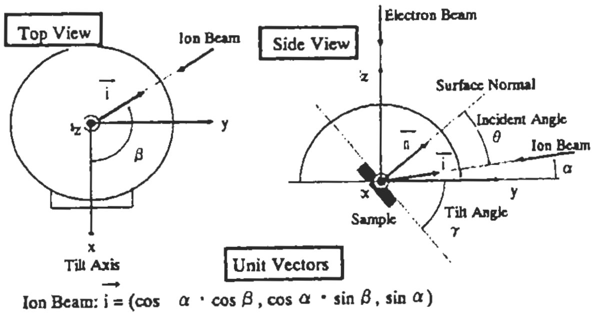
In this research, we determined $R_{Al_2O_3}/R_{SiO_2}$ under different surface charging conditions and in different sputter directions. Based on the results, we discuss the effects of surface charging and the incident Ar^+ ion azimuthal direction on $R_{Al_2O_3}/R_{SiO_2}$.

2. Experimental Procedure

Sapphire single crystal [α- Al_2O_3 (0001)] and thermally oxidized SiO_2 films were prepared for these experiments. The incident azimuthal direction of ion etching was parallel to the a-axis for Al_2O_3 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.



Ion Beam: $\vec{i} = (\cos\alpha \cdot \cos\beta, \cos\alpha \cdot \sin\beta, \sin\alpha)$

Surface Normal: $\vec{n} = (0, \sin\gamma, \cos\gamma)$

Incident Angle: $\theta = \cos^{-1}(\vec{i} \cdot \vec{n})$

$= \cos^{-1}(\cos\alpha \cdot \sin\beta \cdot \sin\gamma + \sin\alpha \cdot \cos\gamma)$

Fig. 1 Ion gun geometry.

Table 1 Experimental conditions

Instrument	Quantum2000 (PHI)	SAM680 (PHI)
Ion gun parameters	α=45 deg, β=0 deg	α=13 deg, β=90 deg
Ion incident angle θ (deg)	45 deg (tilt angle=0 deg)	32 deg (t.a.=45), 62 deg (t.a.=15)
Ion current density with Faraday cup at 3 kV (raster size=2 mm×2 mm)	62~67 (A/cm^2)	36~50 (A/cm^2)
Low energy electron neutralizer	emission current=20 μA	-

3. Results and Discussion

We first determined $R_{Al_2O_3}/R_{SiO_2}$ under different surface charging conditions. Figure 2 shows $R_{Al_2O_3}$ as a function of R_{SiO_2} with an increasing ion current density. In the region of a lower current density, the data confirm that $R_{Al_2O_3}$ was proportional to R_{SiO_2}. However, in the region of a higher ion current density, $R_{Al_2O_3}$ deviated from the proportional relationship to R_{SiO_2} and $R_{Al_2O_3}/R_{SiO_2}$ decreased (●). On the other hand, when the specimen was neutralized by irradiating it with low-energy electrons, $R_{Al_2O_3}/R_{SiO_2}$ (▲) became larger at the same ion current density compared with the ratio for specimens without any irradiation.

Figure 3 shows the stylus profiler 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 $R_{Al_2O_3}/R_{SiO_2}$.

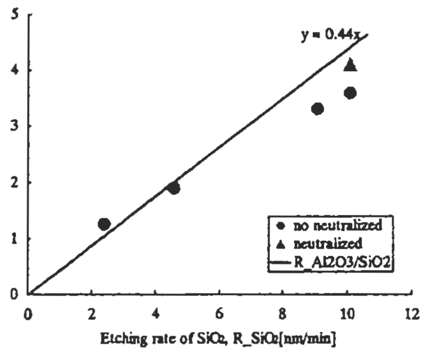


Fig. 2 Etching rate of Al_2O_3 as a function of the rate of SiO_2; incident ion species was Ar^+ and incident angle was 45 deg. Ion energy was 3keV.

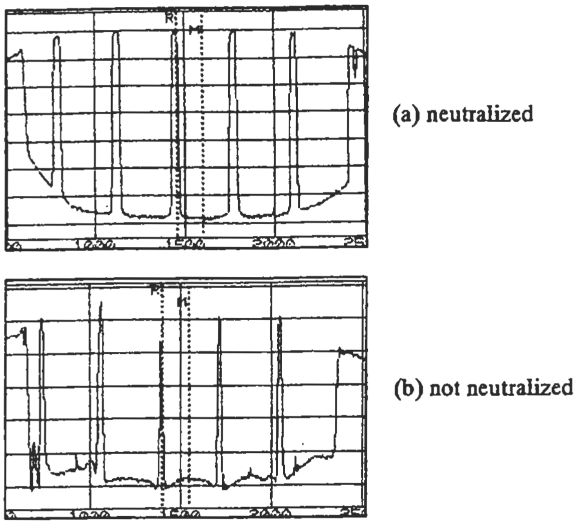


Fig. 3 Stylus profiler images of Al_2O_3 were obtained after sputtering. (a) The specimen was neutralized; (b) the specimen was not neutralized.

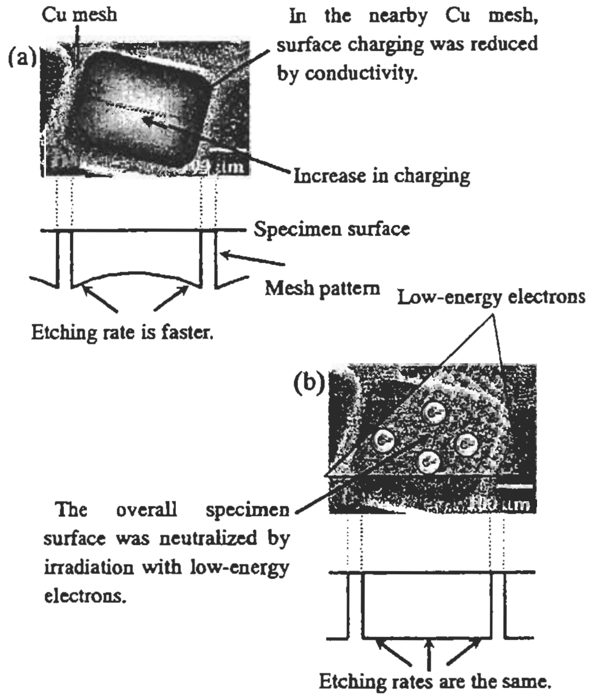
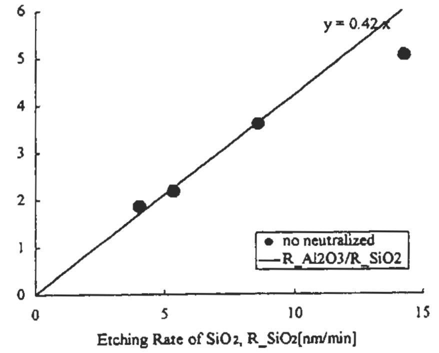
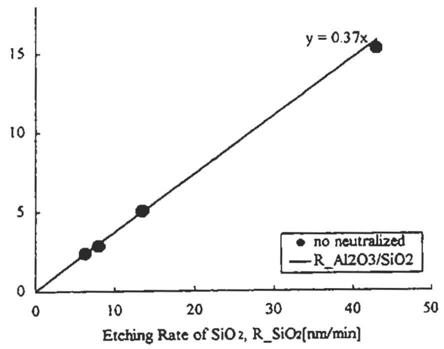


Fig. 4 SEM images and cross-sectional profiles of the specimen surface of an insulator following irradiation with Ar^+ ions.

Figure 4 shows SEM images and cross-sectional profiles of the specimen surface of an insulator irradiated with 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.



(a) 32 deg



(b) 62 deg

Fig.5 Etching rate of Al_2O_3 as a function of the rate of SiO_2; incident ion species was Ar^+ and incident angles was (a) 32 deg. and (b) 62 deg. Ion energy was 3keV.

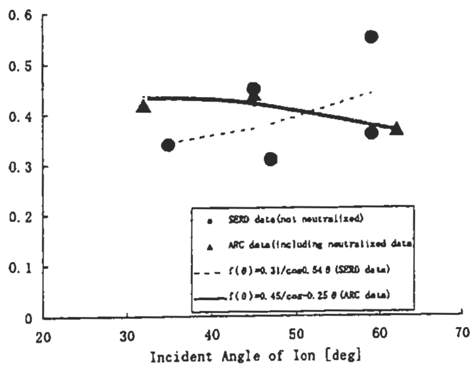


Fig. 6 Relative etching rates of Al_2O_3 to SiO_2 as a function of the incident angle; incident ion species was Ar^+ and ion energy was 3keV.

Figure 5 shows the etching rate for Al_2O_3 as a function of the rate for SiO_2, when the incident ion species was Ar^+ and the ion incident angles were 32 and 62 deg., respectively.

Figure 6 shows the incident angle dependence of the relative etching rates of Al_2O_3 to SiO_2, when 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 $R_{Al_2O_3}/R_{SiO_2}$ for different incident azimuthal directions, either parallel or vertical to the a-axis on the surface of Al_2O_3. $R_{Al_2O_3}$ in the parallel direction was higher than that in the vertical direction. It is thought that this shows the planar channeling effect on $R_{Al_2O_3}$.

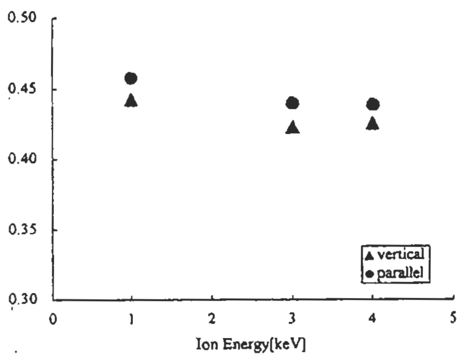


Fig. 7 Relative etching rates of Al_2O_3 to SiO_2 in different incident azimuthal directions, either parallel or vertical to the a-axis on the surface of Al_2O_3.

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, $R_{Al_2O_3}$ 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 Al_2O_3 film ($Al_2O_3/SiO_2/Si$-sub.) obtained by sputtering deposition.

Figure 8 shows $R_{Al_2O_3(film)}$ as a function of R_{SiO_2} with an increasing ion current density obtained by the mesh replica method. Figure 9 shows the XPS depth profile of $Al_2O_3/SiO_2/Si$-sub obtained with a Quantam-2000 instrument (PHI), and figure 10 shows an SEM image of this sample taken with an SAM680 instrument (PHI). Table 2 shows the etching rate ratio $R_{Al_2O_3}/R_{SiO_2}$ by the mesh replica method and XPS depth profiling, respectively.

By the mesh replica method, $R_{Al_2O_3(film)}/R_{SiO_2}$ was 0.59, while $R_{Al_2O_3(sapphire)}/R_{SiO_2}$ was 0.45. It is thought that the difference in density between the sapphire single crystal and the film obtained by sputtering deposition affected $R_{Al_2O_3}$. While $R_{Al_2O_3(film)}/R_{SiO_2}$ was 0.50 by depth profiling, this tendency agree with the thermally oxidized SiO_2 film in SERD report [1].

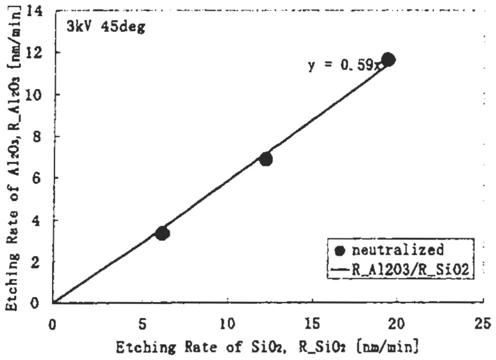


Fig. 8 Etching rate of Al_2O_3(film) as a function of the rate of SiO_2; incident ion species was Ar^+ and incident angle was 45 deg. Ion energy was 3keV.

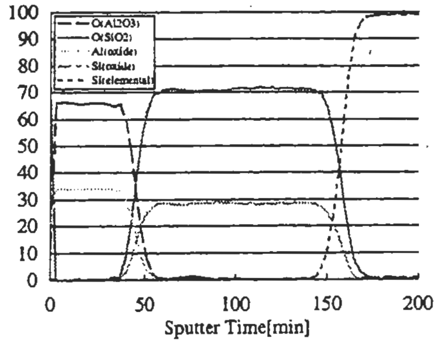


Fig. 9 XPS depth profile of $Al_2O_3/SiO_2/Si$-sub.

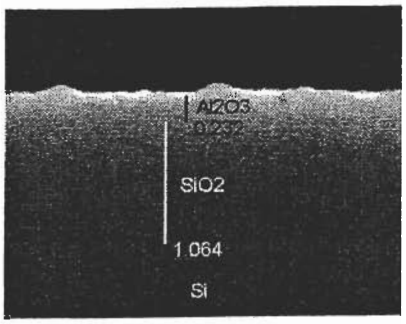


Fig. 10 SEM image of a cross-section of $Al_2O_3/SiO_2/Si$-sub.

Table 2 Etching rate ratio $R_{Al_2O_3}/R_{SiO_2}$

	Sapphire	Sputtering deposition
Mesh replica method	0.45	0.59
Depth profiling	—	0.50

4. Conclusions

1) Surface charging is a contributing factor to the decrease in the etching rate observed for an insulator such as Al_2O_3.
2) It is thought that the lattice plane affects $R_{Al_2O_3}$.

References

[1] SERD Project Group of SASJ, J. Surf. Anal., **8**, 76 (2001).
[2] M. Suzuki, K. Mogi and H. Ando, J. Surf. Anal., **5**, 188 (1998).
[3] A. W. Czanderna, *Methods of Surface Analysis*, pp. 12 13, Elsevier (1989).