Oblique Argon Ion Etching for Copper at Elevated Temperature

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In order to smooth the uneven surface of polycrystalline copper, argon ion etching at the elevated temperature was conducted. The polycrystalline copper was obliquely irradiated by argon ion beam with an ion energy of 1 keV and an incident angle of 70°. The substrate temperatures during argon ion irradiation were room temperature (RT) and 573 K. Before and after the irradiation, the surface morphology was observed using an atomic force microscope. After the irradiation with fluence of $3.4 \times 10^{16}$ Ar/cm² at RT, the change of surface morphology was not clearly observed. After the irradiation with the same fluence at 573 K, the protuberances disappeared and the surface became very smooth. These results suggest that an increase of substrate temperature is effective for the surface smoothing.

Keywords: Ion etching, Argon, Copper, Substrate temperature, Surface smoothing

1. Introduction

Polycrystalline copper has been widely used to the wire material of large-scale integration (LSI) [1]. In order to miniaturize the LSI device, the surface smoothing is necessary in each steps of the production process. For this purpose, chemical mechanical polishing (CMP) has been widely used [1]. However, the surface after the CMP has some structures of the scale of 50-100nm. As an alternative process for the surface smoothing, we suggest the oblique ion etching [2]. Instead of CMP which is a wet process, the present ion etching is a dry process, so that the use of the ion etching is expected to simplify the production process because the process can be performed in the vacuum devices. In our previous study, it was shown that the oblique argon ion etching was effective for the surface smoothing at room temperature (RT), especially in the case of the incident angle of 70 degrees [2]. This smoothing was associated with the preferential sputtering for convex surface by argon ion and re-deposition of sputtered copper atoms [2]. If the substrate temperature increases, the re-deposited particles migrate on the surface [3]. Consequently, the increase of the substrate temperature during the irradiation might result in the smoother surface compared with the case of irradiation at RT.

In the present study, the oblique ion etching with Ar⁺ at the elevated temperature was conducted in order to smooth the uneven surface of polycrystalline copper. After the irradiation, surface morphology was observed to evaluate the surface smoothness.

2. Experiments

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The sheet of polycrystalline copper with a purity of 99.99 at.% (Nilaco Ltd.), was used as the sample. The size was 20mm×5mm×0.05mm. The sheet was mechanically polished by Al₂O₃ powder with an average particle size of 3 μm. After the polishing, the sample was cleaned in ultrasonic bath with ethanol. Before and after irradiation the surface morphology was observed using an atomic force microscopy (AFM) (Shimazu Ltd).

Figure 1 shows the schematic diagram of the ion irradiation apparatus used for the present study. The polished sample was irradiated by argon ion by using an ion gun. The ion energy was 1 keV and the incident angle, θ, was 70 degrees to the normal incidence. In our previous study, it is known that the oblique argon ion etching with the incident angle of 70 degrees is most effective for the surface smoothing [2]. The flux of argon ion measured by the sample current was approximately $10^{17}$ Ar/cm²s. The ion fluence was in the range of $(0.6-5.3) \times 10^{16}$ Ar/cm². The substrate temperature during argon ion irradiation was taken RT and 573 K.

Fig.1 Apparatus of argon ion irradiation.
Fig. 2  Surface morphologies of polycrystalline copper before the irradiation (a), after the irradiation at RT (b), after the irradiation at 573 K (c) and after only heating at 573 K (d). The ion fluence was $3.4 \times 10^{16}$ Ar/cm$^2$.

3. Results

Figures 2 show the AFM images of the surface morphology of samples, before the irradiation, i.e. after the polishing (a), after the irradiation at RT (b), after the irradiation at 573 K (c), and after only heating (d). The groove structures with one direction existed on the sample surface just after the polishing. The structure consisted of large protuberances with approximately 200 nm in width and approximately 30 nm in height, and small ones with approximately 5 nm in height. After the irradiation at RT (fig. 2 (b)), the surface morphology was almost the same as that before the irradiation. Little change in the surface morphology is due to the low ion fluence. After the irradiation at 573K (fig. 2 (c)), however, the surface morphology clearly changed, compared with that before the irradiation. The large protuberances became unclear and the small ones disappeared. The change in the surface morphology was also observed for the sample after only the heating at 573 K (fig. 2 (d)). In this case, the large protuberances still remained. These results indicate that the increase of the substrate temperature is effective for the surface smoothing. In fact, the surface after the irradiation at 573 K was clearly smoother than the surface before the irradiation.

![Surface morphology of polycrystalline copper after the irradiation with different ion fluence at 573 K. The ion fluence was $0.6 \times 10^{16}$ Ar/cm$^2$, $3.4 \times 10^{16}$ Ar/cm$^2$ and $5.3 \times 10^{16}$ Ar/cm$^2$ for (a), (b) and (c), respectively.](image)

![Surface morphology of polycrystalline copper after the irradiation with different ion fluence at 573 K. The ion fluence was $0.6 \times 10^{16}$ Ar/cm$^2$, $3.4 \times 10^{16}$ Ar/cm$^2$ and $5.3 \times 10^{16}$ Ar/cm$^2$ for (a), (b) and (c), respectively.](image)
Fig. 4 The average height of grooves versus argon ion fluence at incident angle of 70 degrees. The height at null point of ion fluence at 573 K is shown average height after only heating.

The AFM images of samples after the irradiation with different ion fluence at 573 K were shown in Fig. 3. With increase of ion fluence, the groove structures at the surface became unclear, namely most of protruding parts and groove structure disappeared in the case of $5.3 \times 10^{16}$ Ar/cm$^2$.

Figure 4 shows the average height of grooves as a function of the ion fluence. The average height decreased to 28 nm by the irradiation with the fluence of $3.4 \times 10^{16}$ Ar/cm$^2$ at RT. However, the average height largely decreased in the case of the irradiation at 573 K, 24 nm at the fluence of $3.4 \times 10^{16}$ Ar/cm$^2$ and 18 nm at $5.3 \times 10^{16}$ Ar/cm$^2$. This corresponds to the change in the features of the surface morphologies shown in fig. 2 and fig. 3.

From figs. 3 and 4, it was found that the polycrystalline copper surface was smoother by the ion irradiation at the elevated substrate temperature. The surface smoothing mechanism by oblique ion irradiation at the elevated temperature is still uncertain. One of the possible reasons of effective smoothing is seemed to be the enhancement of the surface migration of re-deposited sputtered atoms at the elevated temperature.

4. Conclusion

In order to smooth the uneven surface of polycrystalline copper, the oblique ion etching with 1 keV argon ion at the elevated temperature was conducted. The surface of polycrystalline copper was significantly smoothed by the oblique ion etching with the fluence of $3.4 \times 10^{16}$ Ar/cm$^2$ at 573 K. Compared with the case of RT, the surface smoothness was significantly improved. The present study shows that the etching at elevated temperature is useful for the surface smoothing.