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Measurement of the Strength at Grain Boundaries in Electroplated Copper Thin-Film Interconnections

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Abstract

In this study, a micro-tensile test method that can measure the strength at a grain boundary has been developed by applying a focused ion beam system. In order to clarify the relationship between the micro structures of grain boundaries and their strength, the crystal quality of grain boundaries was evaluated by electron back-scatter diffraction (EBSD) analysis method. It was confirmed that the strength of grain boundaries with low crystallinity was much lower than that with high crystallinity.

1. Introduction

Electroplated copper thin-films have applied to the interconnection material in TSV structures of 3D semiconductor modules because of its low electric resistivity and high thermal conductivity. However, electrical and mechanical properties of electroplated copper thin-films have been found to vary drastically depending on their microtexture. In particular, the crystallographic quality (crystallinity) of grain boundaries in the thin-films dominates the variations of these properties and their long-term reliability. Since grain boundaries are the area where the atomic alignment of materials is disordered and various defects such as vacancies, dislocations and impurities easily concentrate, the deterioration of the reliability of the interconnection is concerned around grain boundaries. For example, several voids mainly appeared in the vicinity of random grain boundaries during the EM (electro migration) test, as shown in Fig.1. This indicates that the grain boundary has become a diffusion path of atoms by EM. The crystallinity around grain boundaries is degraded by EM and electrical open failure often occurs due to the growth of void around grain boundaries with low crystallinity. Therefore, it is very important to evaluate the characteristics of a grain boundary quantitatively in order to control and assure the electrical performance and reliability of the electroplated copper thin-film interconnections.

Recently, a new micro-tensile test method using a focus ion beam (FIB) system has been developed for measuring the strength of a grain boundary [1]. In this study, the strength of grain boundaries in the electroplated copper thin-film interconnections was evaluated by using the developed method. In addition, we evaluated the crystallinity of grain boundaries by using an Electron Back Scatter Diffraction (EBSD) method to investigate the relationship between the crystallinity of grain boundaries and their strength quantitatively.

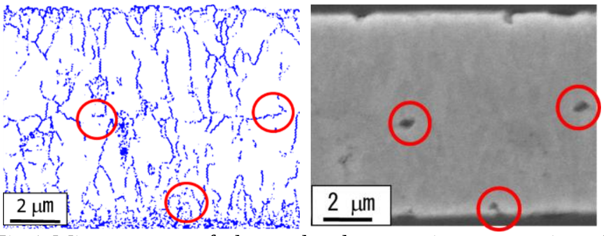


Fig.1 Micro texture of electroplated copper interconnection after the EM test, (a) distribution of random grain boundary and (b) SEM image at the same area of (a)

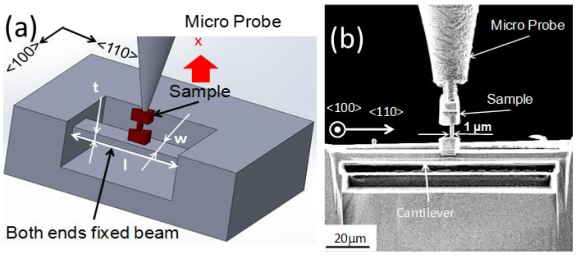


Fig. 2 (a) Schematic image of the proposed micro tensile test and (b) SIM image of completed test system before the tensile test.

2. Method of micro tensile test

The micro-tensile test system consists of a three-point bending loading tool which is cut from a single crystalline silicon substrate and a test sample. The schematic image and scanning ion microscope (SIM) image of the micro tensile test is shown in Fig. 2. A loading tool which consisted of a single-crystalline silicon beam was fabricated by using a FIB system (Hitachi, FB2200). The in-situ micro tensile test was performed in the FIB system at room temperature. An axial tensile strain was applied by pulling-up the microprobe along the vertical direction continuously. The load applied to the beam was estimated by the deflection of the beam, monitored by SIM in real- time. The spring constant k of the beam can be written by

$$k = \frac{4Ewt^3}{l^3} \qquad (1)$$

where E (= 169.7 GPa) is Young's modulus of the single crystal Si along <110> direction and w, l, and t are the width, length, and thickness of the beam respectively.

The stress of the sample σ can be calculated by using the spring constant k.

$$\sigma = \frac{kx}{A} \qquad (2)$$

where x is the displacement of the beam and A is the cross-sectional area of the sample.

3. Results of Micro-tensile test

In this study, the strength of a grain boundary in the electroplated copper thin-films with different thermal hysteresis after the electroplating was measured. After electroplating, some films were annealed in pure argon gas at 400°C for 30 min. The samples for the tensile test were cut out from the prepared electroplated copper thin-films by using the FIB. The length and width (slim part) of the dumbbell specimen were approximately 5 µm and 1 µm, respectively.

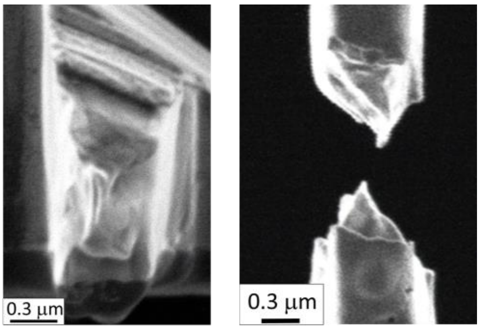
In the micro tensile test, as-electroplated and annealed samples showed completely different behavior. Figure 4 shows a fracture surface of an as-electroplated copper thin-film. This sample mainly showed brittle fracture mode with little necking around the fracture surface. The fracture surface was rather rough, and the fracture mainly occurred at grain boundaries. The estimated strength of this sample was about 320 MPa. This value agreed with the average strength of as-electroplated copper thin-films (350 MPa) measured by the macro tensile test [2]. This result indicates that the strength of the as-electroplated copper thin films was determined mainly by the strength of grain boundaries in the films.

Figure 5 shows a fracture surface of a sample of the electroplated copper thin-film annealed at 400°C. The fracture surface of this sample has a partially smooth surface, indicating that transgranular fracture occurred. It is because that average grain size of the film was larger than 1 µm and thus, the fracture surface of this sample consisted of a few grains at most. The estimated strength of the sample was about 428 MPa.

4. Evaluation of crystallinity of grain boundaries

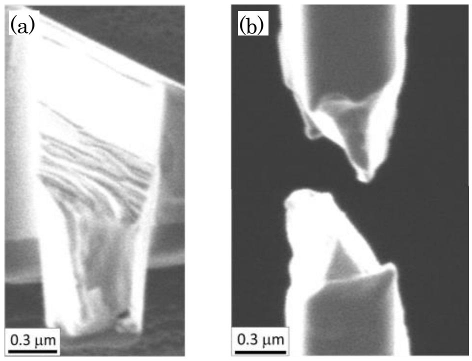
In this study, the crystallinity of grain and grain boundaries was evaluated by using image quality (IQ) value obtained from the observed Kikuchi pattern [2]. Fig.5 shows the two-dimensional distribution of the area with low crystallinity (blue area) in the electroplated copper films. There are many low crystallinity area in grains and along grain boundaries in the as-electroplated film, as shown in Fig. 5(a). This result indicates that the as-electroplated film consisted of both grains and grain boundaries with low crystallinity.

When the film was annealed at 400°C, the average grain size increased due to recrystallization and the number of grains and grain boundaries with low crystallinity decreased drastically, as shown in Fig. 5(b).This result indicates that the quality of the electroplated copper thin films was improved by the annealing at 400°C. In addition, since the fracture of the annealed sample occurred in grains, the strength of grain boundaries of this sample should have been higher than the measured strength of grains. Thus, the strength of grain boundaries in the annealed sample was much higher than that of as-electroplated sample. This result clearly indicates that the strength of grain boundaries varies drastically depending on their crystallinity.



(a) Top view (b) Side view

Fig. 3 Image of the fractured surface of an as-electroplated copper thin-film



(a) Top view (b) Side view

Fig. 4 Image of the fractured surface of an electroplated copper thin-film annealed at 400°C.

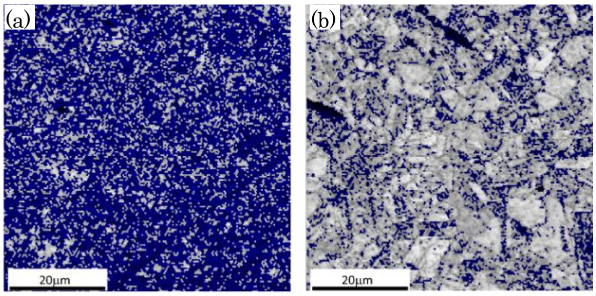


Fig. 5 Distribution of the low crystallinity (IQ < 4000) area in the films: (a) as-electroplated and (b) annealed at 400°C.

5. Conclusions

The effect of the crystallographic quality of a grain boundary in electroplated copper thin-films on their strength was evaluated by using a new micro tensile test and EBSD method. It was confirmed that the strength of a grain boundary with high quality was much higher than the average strength of the films, while that with low quality was close to the average strength. Brittle fracture of the films was found to be dominated by the grain boundaries with low quality. Thus, the strength of grain boundaries of electroplated copper thin films varies drastically depending on their crystallinity.

Reference

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2. Murata, N., Suzuki, K., and Miura, H., Proc. of the ASME IMECE2012, No. IMECE2012-87426, (2012), pp. 1-6 (CDROM)