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Characterization of Electroplated Cu Films by Combination of PALS, HIM, and EBSD

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Abstract

Characterization of the Cu film grains such as size, orientation, and defects plays a crucial role to understand the electroplated Cu films and their performance in terms of the electrical resistance, reliability of electro and stress migrations of interconnects. In this study, we attempted quantitative evaluation of electroplated Cu grains by positron annihilation lifetime spectroscopy (PALS), helium ion microscopy (HIM), and electron back scattering diffraction (EBSD), where 550 and 1000 nm thick Cu films plated with two different electrolytes containing additives A (EA) or B (EB) were used. The PALS and EBSD observations clarified crystalline differences among them. Focusing on the 550 nm thick Cu films, the EA sample showed higher <111> orientation and more pore-accumulation which indicate faster gran growth rate than the EB.

In addition, we firstly applied HIM to the Cu films observation and confirmed that the higher helium ion dose by HIM induced more Cu swelling, along with appearance of cracks. This phenomenon might suggest that accumulating helium ions into nano defects promoted visualization of the defects in the Cu. Thus, HIM is expected to have a potential for accelerated observation of nano defects in interconnect metals. Moreover, Gain Orientation Spread (GOS) maps of EBSD revealed higher strains at the swelled Cu areas in the EB than that in the EA, suggesting the same tendency of the PALS and EBSD data. These results indicate that the EA should have a better interconnect reliability such as electro migration.

1. Introduction

Copper has been used as the primary material for the interconnect applications due to its low resistance, mechanical properties, and cost. Electrical plating has been intensively used as one of the main deposition methods due to the fact the Cu film can be plated on different features and patterns of a substrate such as damascene and TSV using different types of additives to control the filling property. Although the quality of Cu films has been studied by measurements of electrical resistance, mechanical strain, and impurity rate etc., it has not been investigated well enough to characterize Cu films and prove the quality of films in terms of electrical resistance and reliability. It has been well proposed that the orientation of grains and the defects, such as dislocation, in Cu films have been the keys for the performance and the reliability of the material. However, these factors have not been quantitatively investigated enough and proved their effects yet.

2. Experimental

550 nm and 1000 nm thick Cu films were electroplated onto test coupons with 100 nm of Cu seed layer by PVD. Copper sulfate electrolytes containing additives A (EA) or B (EB) were used (hereafter 550 films plated with EA or EB are denoted A1 and B1, respectively, and 1000 nm films plated with EA or EB are indicated A2 and B2, respectively). The Cu films were annealed at 200 °C for 2 min.

EBSD was employed to characterize the orientation of grains, grain size distribution, and strain mapping of the Cu films. PALS was used for evaluation of the crystallinity and grain growth [1]. HIM, which is known for its high spatial resolution up to 0.35 nm [2], was utilized to dose helium ions into Cu films and induce the dislocations (defects). We evaluated the Cu samples with various helium ion dose amount from 1.17×10^{17} to 1.17×10^{18} ions cm^{-2}.

3. Results and Discussion

Figure 1 shows the inverse pole figure maps (IPF-ND) of Cu films. The preferred orientation was <111> (blue color) for all the samples, and the A1 film showed more than 80 % of <111> area. The 1000 nm films showed some grains with <101> direction indicating that the thicker films tend to grow the direction due to their lower activation or surface energy of the grains [3, 4]. The B1 film showed less crystallinity compared to the other three films, which was a good agreement with the result of PALS (Table 1) showing the higher intensity of the atomic size pore by 3.9% than A1. These intensities were extracted from the two lifetime components fittings of the spectrum (shown in Fig. 2).

Figure 3 shows the HIM images of Cu films after the dose of 7×10^{17} ions cm^{-2} by HIM. The area of ion dose for all Cu films was swelled. It was assumed that the accumulation of both vacancies and helium atoms occurred during the irradiations. Further study on swelling generated by vacancies and helium ions will be required. Figure 4 shows the GOS maps

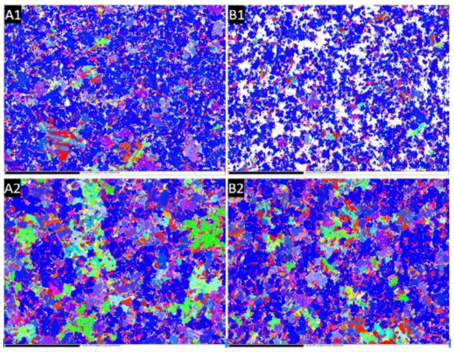

Fig. 1 Inverse pole figure maps (ND) of Cu films, A1: 550 nm with EA, B1: 550 nm with EB, A2: 1000 nm with EA, and B2: 1000 nm with EB. The scale bar is 20 μm.

for each Cu film plotting the mean misorientation angle per grain. The periphery of the ion dosed area for all the Cu films showed the lower misorientation angle (corresponding to lower strain) whereas the ion dosed area showed higher values. Interestingly, some of the center grains of B1 had the highest strain, more than 5 degrees, while other samples did not have grains with such high strains. The result indicates that B1 seems to have more dislocations (defects) compared to the other films, and hence Cu grains of B1 are strongly stressed by the dosed helium ions and swelled considerably. Such a high dislocation density of B1 leads to a short EM lifetime [5, 6], suggesting higher reliability of A1 than B1.

Table 1 Intensities of two positron life time components

Name	Additive	Thickness	Atomic size pore (t1 in Fig. 2)	Clustered pore (t2 in Fig. 2)
A1	A	550 nm	86.0 %	14.0 %
B1	B		89.9 %	10.1 %
A2	A	1000 nm	75.4 %	24.6 %
B2	B		72.2 %	27.8 %

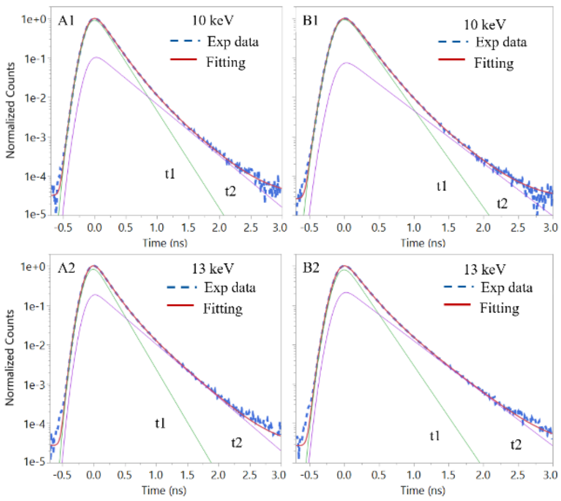

Fig. 2 PALS spectra of Cu films.

4. Conclusions

We studied the Cu films focusing the grains and defect behavior with the four different electroplated Cu films. The

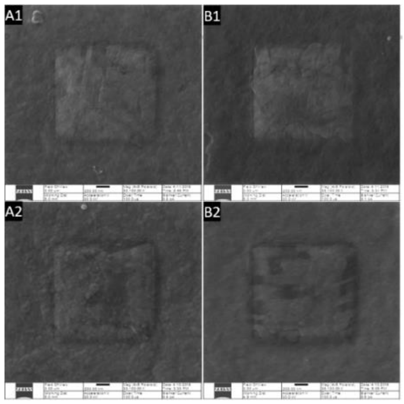

Fig. 3 HIM images of Cu films after the dose of 7×10^{17} ions cm^{-2} by HIM. The scale bar is 200 nm.

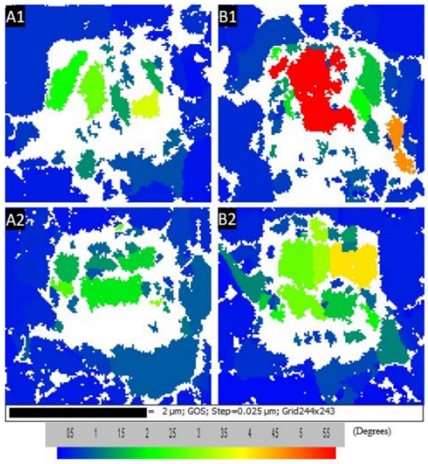

Fig. 4 Grain Orientation Spread (GOS) maps for each Cu film after the dose of 7×10^{17} ions cm^{-2} by HIM.

550 nm thick films showed the strongest <111> preferred grain orientation. The superior crystallinity and growth rate for the grains of A1 to B1 was confirmed by EBSD and PALS. Besides, the higher helium ion dose induced the more Cu swelling, along with the appearance of cracks. Those phenomena suggest that accumulating helium ions into nano defects might promote visualization of the defects in the Cu film. The GOS maps showed that the ion-induced area had a higher strain compared to the periphery. The grains of B1 showed the highest strain among its counterparts suggesting B1 grains had more dislocations, which probably lead to EM lifetime degradation of B1. Further experiments will be necessary to confirm our assumption.

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