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Effects of direct current and pulse-reverse copper plating waveforms on the incubation behavior of self-annealing

Min-Yuan Cheng [a], Kei-Wei Chen [b,*], Tzeng-Feng Liu [a], Ying-Lang Wang [b], Hsien-Ping Feng [c]

[a] Department of Materials Science and Engineering, National Chiao Tung University, Hsinchu, 300 Taiwan, ROC
[b] Department of Materials Science, National University of Tainan, Tainan, 700 Taiwan, ROC
[c] Department of Chemical Engineering, National Tsing Hua University, Hsinchu, 300 Taiwan, ROC

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ABSTRACT

This study investigates spontaneous microstructural evolution in electroplated Cu films with various plating current densities involving direct current and pulse-reverse waveforms and various possible driving forces. Studies have explained the grain growth and resistivity decrease during the self-annealing of as-deposited Cu film, but the incubation behavior of self-annealing under various direct current and pulse-reverse current waveforms at a certain film thickness is unknown. In this study, it was found that pulse-reverse current retards the incubation behavior more significantly than does direct current. According to the measurements of resistivity, stress, and secondary ion mass spectrometer, the large stress difference between the initial and critical values and the low impurity content of pulse-reverse current postponed the incubation, and led to a slow self-annealing rate. The combination of the stress difference and the impurity effect explains the incubation behavior of self-annealing under various plating current densities. The resistivity and X-ray diffraction results suggest that stress is the primary driving force that dramatically speeds up grain growth above the critical stress, and that high current density with a rapid grain growth rate enhances the (200) texture for strain energy minimization in electroplated Cu film.

1. Introduction

In most production processes, electroplating (ECP) is the primary method of copper metallization. In general, it is implemented using direct current (DC) density or pulse-reverse (PR) current density methods. Researchers [1,2] have pointed out that electroplating copper using PR plating produced films with finer grain size, smaller size, and lower porosity than those of films obtained using DC plating.

The grain size, texture, and impurity of polycrystalline Cu film are known to be critical factors that affect the resistivity of metallization. It has been reported that electroplated Cu films experience self-annealing. Toomey et al. [3] suggested that the copper self-annealing process has an incubation region and a critical stress point. The resistivity dramatically decreases when this critical point is exceeded [3]. During the self-annealing process, both the physical and electrical properties dramatically change, including an increase in grain size (grain growth), changes in preferred crystallographic texture, a decrease in resistivity by about 20%, and a stress change in the tensile direction [4–11]. Harper et al. [9] demonstrated that stress is the driving force of grain growth in Cu films.

For face-centered-cubic (FCC) Cu films, the orientation with the lowest surface energy is (111), and the lowest elastic strain energy is (100) [7]. Zielinski [12] reported that the texture remains unchanged during the self-annealing process until the stress in the film reaches the critical value (σ_T), which varies with the film thickness. After the stress exceeds the critical value, the (111) texture starts to decrease and the (200) texture begins to increase; they lead to strain energy minimization in the film [4,8]. This clearly demonstrates that stress development induces texture changes and grain growth during self-annealing.

The defect density within a film decreases at room temperature due to grain growth. Chang [13] suggested that the defects within porous films accelerate self-annealing. Yoon [14] claimed that grain growth during self-annealing provides a driving force for impurity redistribution. Several studies have reported the effect of the current density on self-annealing behavior. Haebum et al. [4] suggested that films plated at higher current densities take less time to complete self-annealing; compared with direct current density, self-annealing behavior is retarded in films plated with pulse-reverse current. This is believed to be due to films plated at a higher deposition rate having higher defect densities, which provide a higher driving force for self-annealing [15]. Brongersma et al. [16] stated that self-annealing behavior is intimately linked to the composition of the plating bath and the density of the sequential incorporation of organic additives in the Cu layer.

* Corresponding author.
E-mail address: kwchena@aa.nctu.edu.tw (K.-W. Chen).

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While self-annealing has been studied in terms of grain size, stress, texture, impurity, and current density, the combination of these characteristics on self-annealing behavior has not been fully identified; it thus is the main subject of this article. The objective of this paper is to describe the relative roles of stress, texture, and impurities and their effect on the incubation time of the self-annealing process at various plating waveform density conditions using sheet resistance, stress, secondary ion mass spectrometer (SIMS), X-ray diffraction (XRD), and focused ion beam (FIB) measurements to provide a detailed understanding of self-annealing kinetics in copper thin film.

2. Experimental procedure

Cu electrochemical deposition on blanket 8 in. Si (100) wafers was performed using a 100 nm thermal oxide substrate and 30 nm TaN/ 150 nm Cu seed layers, which were deposited by physical vapor deposition (PVD) at room temperature without vacuum breaking. Electroplating was carried out on APPLIED iECP plating hardware using 15 °C bath control with a 12 L/min steady overflow rate. Electroplating studies were performed in an acid copper sulfate electrolyte containing 50 g/L copper metal, 20 g/L sulfuric acid, and 80 mg/L chloride ions. Two additives, 5 ml/L of a proprietary organic accelerator additive of the sulfide type (Shipley company, similar to SPS) and 15 ml/L of a proprietary organic additive suppressor of the polyglycol type (Shipley company, similar PEG), were added to the electrolyte.

Two series of Cu films were prepared: (1) 1 μm Cu films plated with direct current (DC) at 15 mA/cm^2, 30 mA/cm^2, and 60 mA/cm^2; (2) 1 μm Cu films plated with pulse-reverse (PR) current at 15 mA/cm^2, 30 mA/cm^2, and 60 mA/cm^2. The PR plating method used continuous and alternate forward and reverse pulses to produce sequential deposition and etching processes by controlling the magnitude and direction of the electrical current. For DC plating, the waveform was 10 s of forward current with 3.14 A; for PR plating, the waveform was 10 s of forward current with 3.14 A and 1 s of reverse current with 25 A, repeated for 5 cycles. The variation of plating current distribution across the wafer was controlled at below 3%.

The sheet resistance of Cu films was measured using the four-point probe method on Prometrix RS-75 tools. An optical curvature tool was used to measure film stress. Cu film grain sizes were determined using a focused ion beam scanning electron microscope (FIB-SEM). X-ray diffraction method was applied to determine film textures. The impurities in Cu films including carbon, oxide, sulfur, and chloride atoms were detected by secondary ion mass spectrometer (SIMS) analysis.

3. Results and discussion

In order to investigate the effect of plating current density on the rate of the self-annealing process, a number of 1 μm-thick Cu films were prepared at essentially the same time. Film resistivity was monitored over time at room temperature. For each plating condition, the final resistivity decreased by approximately 20% compared to the values of the initial film regardless of conditions, as shown in Fig. 1. This value is consistent with the data reported in the literature. Fig. 1 also shows the sheet resistance change of Cu film formed at various current densities at a constant thickness (1 μm) in the self-annealing process. A higher current density leads to a higher self-annealing rate. As Toomey et al. [3] suggested, there are an incubation time and a critical stress point in the curves. The resistivity dramatically decreases when this critical point is exceeded. A shorter incubation time below the critical point was found in the plating film formed at a higher direct current density. This was also found for films with various PR current densities. Moreover, the film formed using PR current required a longer incubation time than that of the film formed using direct current for the same density. High DC plating led to a faster self-annealing rate than that of low DC and PR current plating.

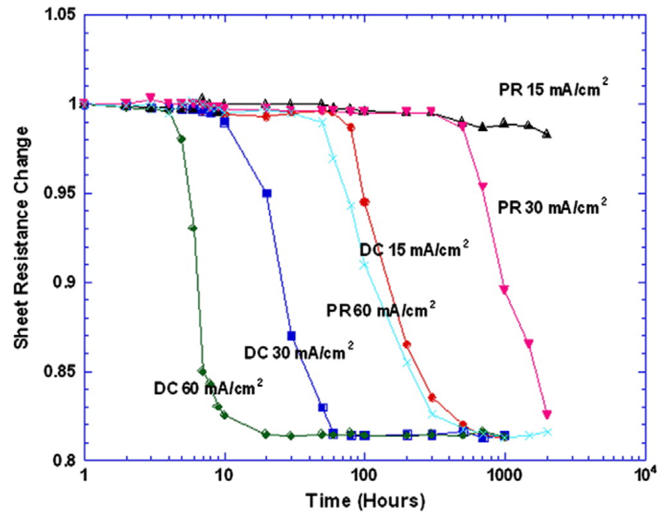

Fig. 1. Evolution of the sheet resistance change for 1 μm Cu films at various current densities.

The grain structures, textures, and impurities of the incubation region and the abnormal grain growth region are next discussed to understand the self-annealing behavior at various plating currents.

3.1. Grain structure

Fig. 2 shows the FIB-SEM images of the four phases of the Cu self-annealing process (initial (Fig. 2a), incubation (Fig. 2b), above critical point (Fig. 2c), and final (Fig. 2d)), plated using 15 mA/cm^2 DC and 60 mA/cm^2 PR waveforms, respectively. The results demonstrate the existence of grain growth during the copper self-annealing process. The magnitude of current density did not affect the initial or final grain size during self-annealing. Plating current conditions only affected the grain growth rate from the initial to final states under a constant film thickness. Grain growth of the incubation region resembled piecemeal islands, as shown in Fig. 2a and b. Elimination of the grain boundaries during grain growth causes film shrinkage and produces tensile stress. As stated in previous studies [12,17], grain growth rate is accelerated by an additional driving force when the critical stress is exceeded (Fig. 2c) for minimization of strain/surface energy.

Table 1 summarizes the initial, critical, and final stress values of 1 μm-thick Cu films for various plating currents. The data demonstrates that using high DC density to deposit Cu films results in large initial stress. Previous studies proved that the critical stress value is determined by film thickness. Moreover, the final stress value was not significantly affected by the type of waveform or current density. Table 2 shows that larger initial stress, which means a smaller stress difference between the initial and critical value, resulted in shorter incubation time and faster self-annealing. The same trend was observed for DC and PR plating conditions. Similarly, the critical point trigger time increased with lower current density. The driving force of self-annealing was the stress in the film during the grain growth process.

In a completely recrystallized metal, the driving force for grain growth is a decrease in surface energy of the grain boundaries by atomic arrangement. Elimination of the grain boundaries during grain growth generates stress in the film. Doerner et al. [18] proposed a relationship between the grain size and the intrinsic biaxial film stress (σ) during grain growth.

$$\sigma = \left(\frac{E}{1-\nu}\right)\Delta\alpha\left(\frac{1}{L_0}-\frac{1}{L}\right) \quad (1)$$

where E is the Young's modulus of the film and ν is the Poisson ratio of the film. $\Delta\alpha$ is the excess volume per unit area of the grain boundary,

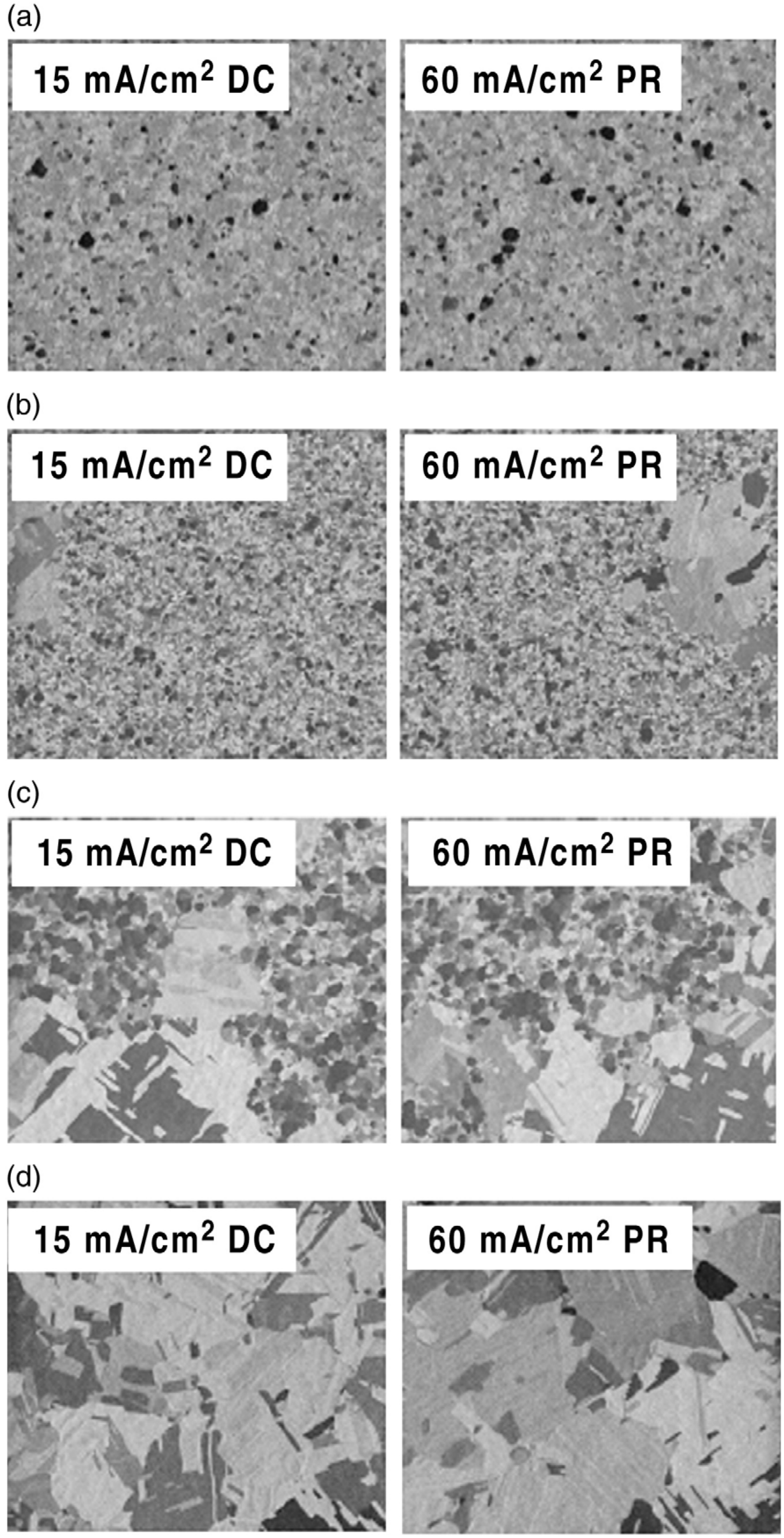

Fig. 2. FIB images of grains in 1 μm-thick electroplated Cu films for (a) initial, (b) incubation, (c) above critical point, and (d) final stages, plated by 15 mA/cm^2 DC and 60 mA/cm^2 PR conditions, respectively.

Table 1
Summary of the initial, critical, and final stress values of 1 μm-thick Cu films plated at various current densities (only Cu film, excluding TaN/seed layer).

Film	mA/cm^2	Initial (MPa)	Critical (MPa)	Final (MPa)
DC plated film at various current densities	15	63.6	285	332
	30	83.1	285	328
	60	102.8	285	328
PR plated film at various current densities	15	32.1	285	333
	30	47.6	285	339
	60	61.9	285	331

which derives mainly from the separation between adjacent grains caused by an imperfect packing of atoms at the boundary. L_0 is the average grain diameter of a thin film in the initial stage and L is the average grain diameter during grain growth with the assumption that the grains are spherical. Under the same initial grain size (L_0) and after the same interval, a film with higher stress (σ) will have larger grain size (L), which means that stress is the driving force of grain growth during the self-annealing process. This explains the results in Fig. 2 and Table 2; a large initial film stress resulted in a short incubation time.

However, there was a significantly shorter incubation time for the 60 mA/cm^2 PR waveform (20 h) even though it had the same stress difference as that of the 15 mA/cm^2 DC waveform (40 h). It means that the stress effect cannot completely explain the incubation time difference during self-annealing.

3.2. Film texture

The film texture for various current densities measured by XRD is plotted in Fig. 3. The dramatic degradation of the (111) texture intensity matched the development of tensile stress over the critical value, as mentioned by Haebum et al. [4]. For FCC metal film, the orientation with the lowest surface energy is (111) and the lowest elastic strain energy is (200). Texture can change to a low (111)/(200) ratio depending on the stress that has developed in the films during grain growth. Cu film prefers the (111) texture to release surface energy during the incubation period of self-annealing. Grain growth starts from the film surface (lowest surface energy barrier) and then expands to the bottom [19]. During grain growth, the grain structure in the top zone has few grain boundaries and a shrinking volume compared to those in the bottom zone. The top zone compresses the bottom zone, producing tensile stress. The (200) texture becomes remarkable for strain energy elimination when the critical stress is exceeded. Fig. 3 shows that the high current density film took a shorter time to reach the critical stress; this means a faster self-annealing rate and lower (111)/(200) ratio in the final stage (refer to Table 3).

The texture of Cu plating film is significantly correlated with the stress variation. Haebum et al. [4] suggested that during the self-annealing process, the (111)/(200) ratio starts to decrease above a critical stress and that this critical stress varies with the film thickness. The critical stress of all conditions in this study is all the same for the

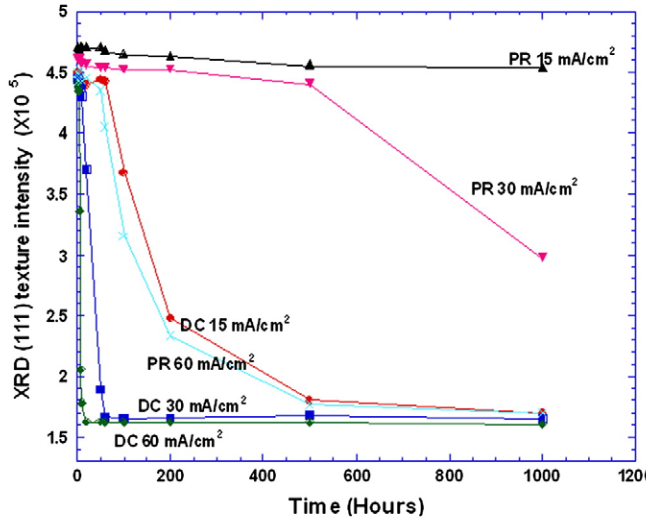

Fig. 3. Change of X-ray (111) peak intensity with time for DC plated and PR plated films at various current densities.

1 μm-thickness condition. As shown in Table 3, for both DC and PR current conditions, all films had an almost equal (111)/(200) ratio in the initial textures and films plated at high current density had high stress. The (111) texture dramatically decreased while the (200) texture increased when the critical stress value was exceeded. The high stress films took less time to reach the critical stress. Finally, the high stress films had the lowest (111)/(200) ratio because of the high driving force of grain growth. Comparing the performance of the 60 mA/cm^2 PR waveform to that of the 15 mA/cm^2 DC condition, although they had equal final (111)/(200) ratio, their incubation times were different. It appears that the texture evolution can be explained by the stress effect, but this effect cannot completely explain the incubation time behavior of self-annealing for various plating current conditions. This implies that there are other variables that affect the incubation behavior of self-annealing.

3.3. Impurity effect

In an aqueous plating bath, as-deposited film remains defect rich and fine-grained, which induces self-annealing behavior. The development of Cu film stress, as previously mentioned, is clearly correlated with texture orientation. A rapid plating rate with high current density generated high film stress, as proved by XRD analysis. As many researchers reported, a variety of proprietary additives can be used to influence the impurity of copper plating film. However, the effect of impurity of Cu film on self-annealing behavior is not very clear.

Fig. 4 shows the secondary ion mass spectrometer results for the distributions of three impurities in the film formed at 15 mA/cm^2 DC plating current density under four bath life conditions. The interface between the plated copper and substrate was at the position of 400 s sputter time. As the bath aged, certain byproducts formed as a result

Table 2
Summary of the stress difference between the initial and critical values and the self-annealing rate of 1 μm-thick Cu films plated at various current densities.

Film	mA/cm^2	Stress diff. (MPa)	Incubation time (h)	Critical point (h)
DC plated film at various current densities	15	221.4	40	80
	30	201.9	6	10
	60	182.2	2	4
PR plated film at various current densities	15	252.9	>1000	N/A
	30	237.4	200	500
	60	223.1	20	50

Table 3
Summary of the (111)/(200) ratio of electroplated films with various current densities.

Condition		I(111)/I(200)			
Film	mA/cm^2	Initial (<1 h after dep)	Final (>1000 h)	Stress diff. (MPa)	Incubation time (h)
DC plated film at various current densities	15	107.3	18.5	221.4	40
	30	106.9	9.3	201.9	6
	60	105.5	5.6	182.2	2
PR plated film at various current densities	15	109.7	103.7	252.9	> 1000
	30	108.6	56.9	237.4	200
	60	107.5	17.8	223.1	20

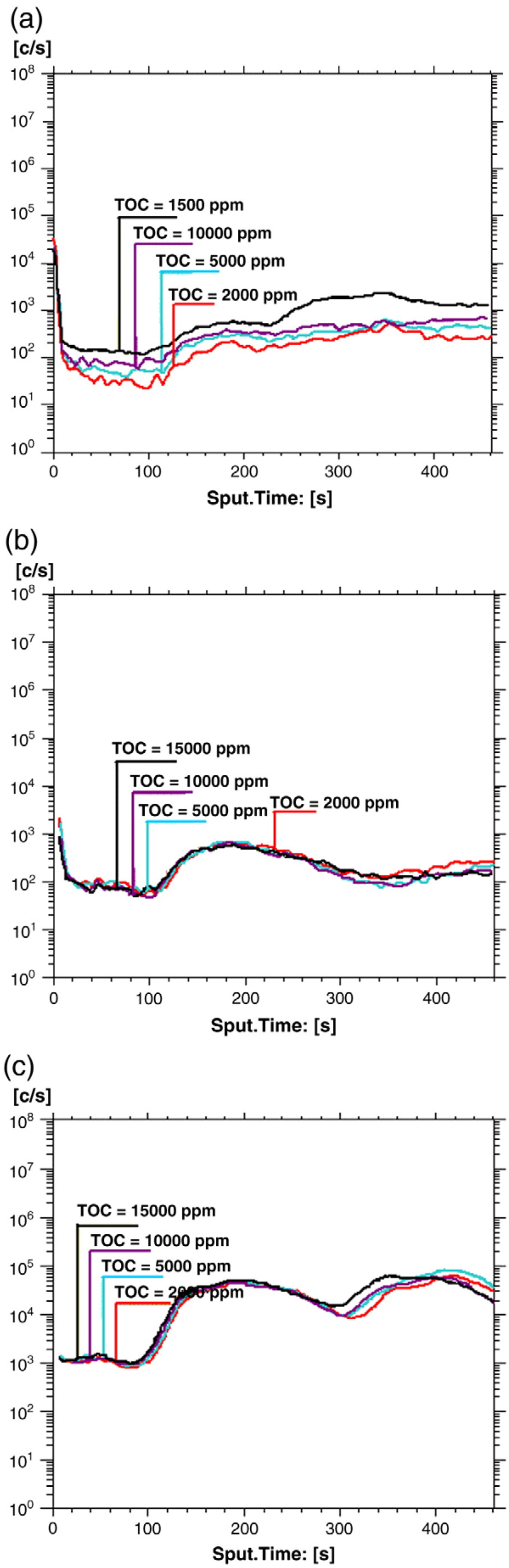

Fig. 4. SIMS measurement for Cu films plated at a 15 mA/cm^2 DC current density at various bath life times: (a) carbon, (b) sulfur, and (c) chloride content.

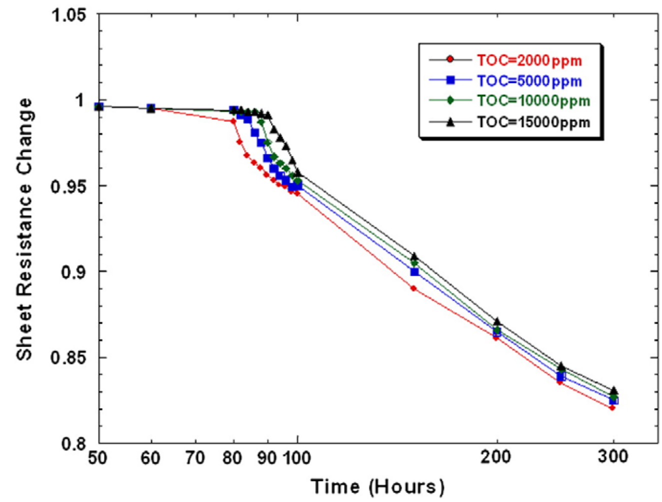

Fig. 5. Sheet resistance change of Cu films plated at a 15 mA/cm^2 DC current density versus bath life time.

of additive decomposition during the plating process [20,21]. TOC (total organic concentration) measurements showed byproduct accumulation when bath life increased [9]. These byproducts were incorporated into the plating film, increasing carbon impurities, as shown in Fig. 4. The concentration of sulfur and chloride were the same versus bath life times. Fig. 5 demonstrates the sheet resistance change for 15 mA/cm^2 DC current for the four bath life conditions. Plating in the aging bath postponed the incubation time period at the same plating current. Harper et al. [9] explained the incubation time from the impurity aspect; impurities incorporated into Cu film during plating pin the grain boundaries and retard the grain growth process. Table 4 summarizes the incubation time and initial stress values of the four bath life conditions. Impurities co-deposited into plated film were one of the factors that changed the incubation time during self-annealing, but its contribution to stress was not significant.

As indicated above, both stress and impurity influence the incubation time during self-annealing. Film stress is affected by dislocation and vacancy concentration with various plating currents, but it is not affected by small numbers of impurities. Hence, stress and small numbers of impurities are regarded as two independent factors for self-annealing kinetics. Impurities including carbon, sulfur, oxygen, and chloride within plated films were also correlated with plating current (DC and PR waveform, bath TOC = 15,000 ppm), as shown in the secondary ion mass spectrometer analysis of Fig. 6a to d. The interface between the plated copper and substrate was at the position of 600 s sputter time. A higher DC current density produced more impurities. PR conditions showed the same trend of impurity variation as that of DC waveforms. However, the same impurity level between 60 mA/cm^2 DC and 30 mA/cm^2 DC plating currents led to different incubation time behavior. This implies that the impurity effect does not completely explain the self-annealing behavior between DC and PR waveforms.

The stress effect and the impurity effect can explain the incubation time behavior of the self-annealing process in DC and PR plating

Table 4
Summary of the incubation time and stress versus various bath life time expressed as TOC values.

Film	TOC (ppm)	Stress (MPa)	Incubation time (h)
15 mA/cm2 DC plated film for various bath life times	2000	221.4	80
	5000	221.7	87
	10,000	221.9	90
	15,000	221.8	96

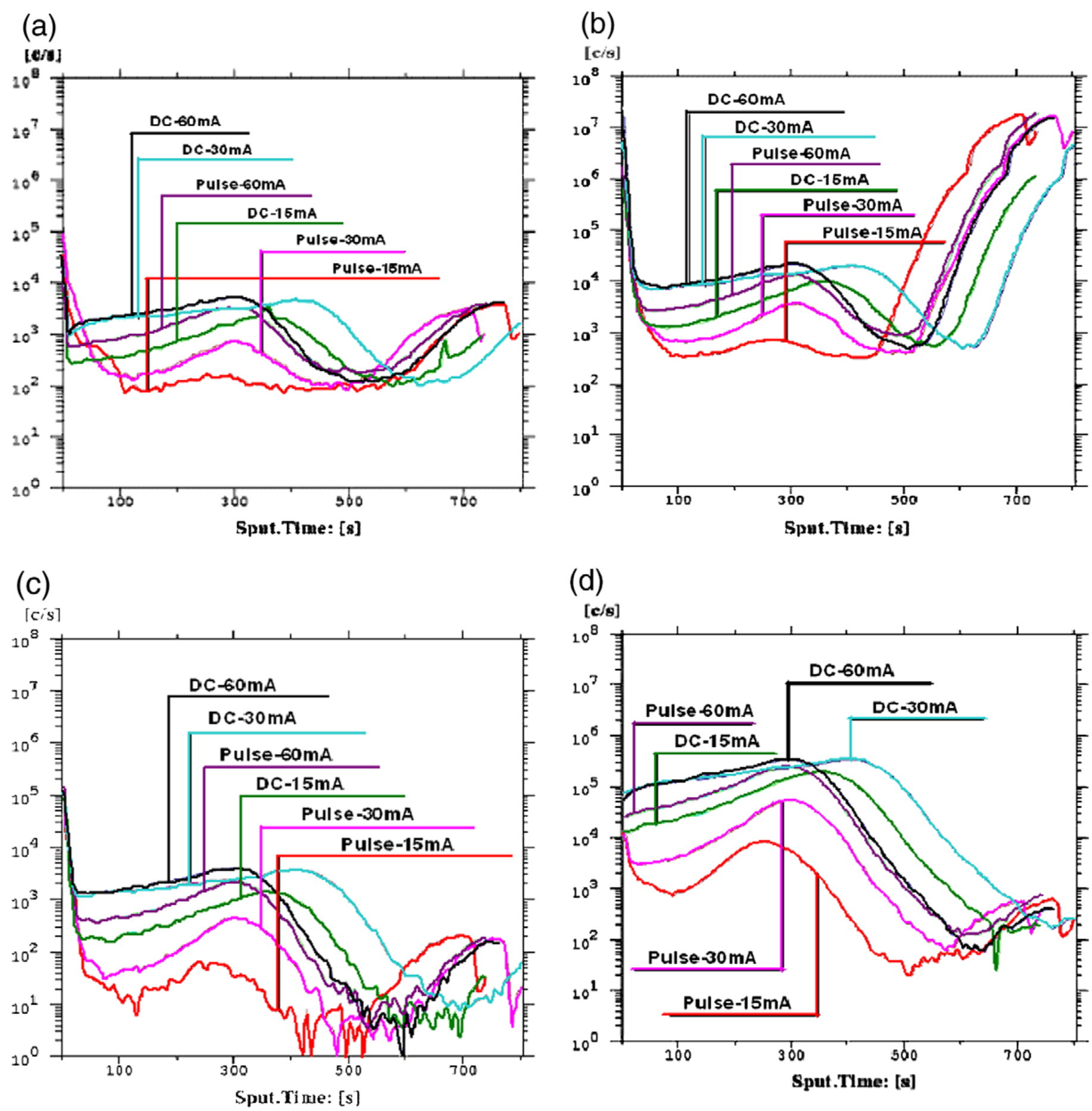

Fig. 6. SIMS analysis for 1 µm-thick Cu films plated with various DC and PR current densities: (a) carbon, (b) oxygen, (c) sulfur, and (d) chloride.

currents, respectively. However, they could not illustrate the whole differences of the incubation time between various DC and PR plating current conditions separately. This revealed that stress and impurity are two independent factors for self-annealing behavior. The incubation time, stress difference value, and impurity data for various DC and PR current densities are summarized in Table 5. The stress factors were normalized using the stress difference data of 60 mA/cm^2 DC as the standard. Similarly, the impurity factors were normalized using the carbon content before the 200 s sputter time of 60 mA/cm^2 DC as the criterion. The self-annealing rate was dominated by the incubation time. These two factors influenced the incubation time from the initial state to the critical point, affecting the self-annealing rate. For both DC and PR conditions, the incubation time was directly proportional to the stress difference between the initial and critical point and inversely proportional to impurity levels. Consequently, it is reasonable to use the stress difference per impurity as a combined factor to illustrate the incubation time difference, as shown in Fig. 7. It suggested a matched correlation to explain the incubation behavior of self-annealing for various plating waveform densities. Higher direct current density produced a plated film with a lower stress difference and higher impurity level for a shorter incubation time, which means a faster self-annealing rate. Film plated at a 60 mA/cm^2 pulse-reverse current density, which had the same stress factor as that plated at a 15 mA/cm^2 direct current density, showed a faster self-annealing rate because of its higher impurity amount. Similarly, the reason for different incubation behavior between 60 mA/cm^2 and 30 mA/cm^2 DC density films was the variation in stress difference. Film plated at a 60 mA/cm^2 DC condition had the same impurity level as that plated at a 30 mA/cm^2 DC density, but had a lower stress difference between the initial and critical point.

Table 5
Summary of the stress factor, impurity factor, and incubation time of 1 µm-thick Cu films for various current densities.

Film	mA/cm^2	Stress factor*	Impurity factor*	Stress/impurity factor	Incubation time (h)
DC plated film at various current densities	15	1.215	0.5	2.43	40
	30	1.108	0.95	1.17	6
	60	1.0	1.0	1.0	2
PR plated film at various current densities	15	1.388	0.09	15.43	> 1000
	30	1.302	0.15	8.69	200
	60	1.224	0.7	1.75	20

*: Used the data of 60 mA/cm^2 DC condition (the fast one) to be a standard as "1".

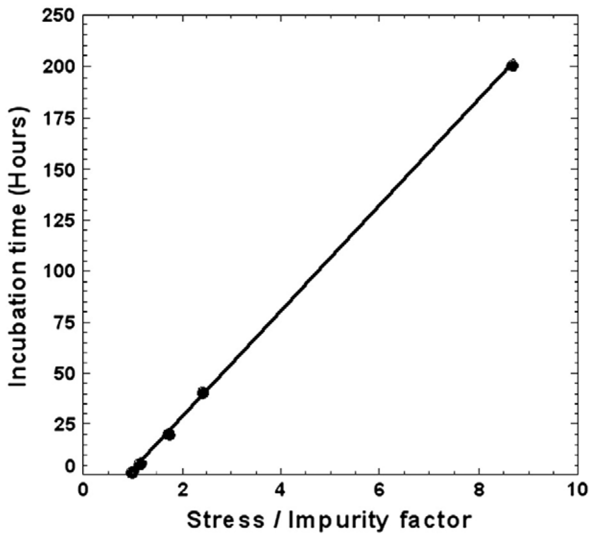

Fig. 7. Correlation of the ratios of stress and impurity factors with various plating current densities and the incubation time of self-annealing of 1 μm-thick Cu film.

As mentioned above, the incubation time of self-annealing was determined by stress and impurities in the plated film. These two factors correctly predict the incubation time below the critical stress point during self-annealing. When the critical stress generated by grain growth is exceeded, self-annealing behavior is mainly accelerated by the stress driving force, as stated in several studies. Additionally, the final film texture is determined by the stress driving force (above the critical point).

It is known that the spontaneous self-annealing process at room temperature depends on film thickness [16]. At a fixed thickness, the plating waveform density determines the incubation behavior by the combined effect of stress and impurities. Film plated by the PR waveform has fewer dislocations, vacancies, and impurities in comparison with those of that plated by DC waveform. Based on metallurgy and the above study [22], the driving force for recrystallization is the stored energy of high-angle grain boundaries derived from point (vacancies and interstitial impurity atoms) and line (dislocations) defects. In the incubation region, spontaneous recovery removes defects, rearranging low-angle grain boundaries and forming large defect-free grains. The texture density of plated film increased with grain growth because the atoms are more closely packed in the grain boundary than they are in the crystal lattice. Shrinkage or densification of the film by elimination of the grain boundaries during grain growth generates tensile stress. When the critical stress is exceeded, abnormal grain growth is observed. Texture evolution is also determined by an additional stress driving force above the critical point.

4. Conclusion

The effect of the plating current waveform on the incubation behavior of self-annealing of Cu thin films was investigated using grain structure, texture, and impurity analyses. The data show that plating current density and waveform determine the plated film initial stress and impurity. At a fixed film thickness, the stress difference between the initial and critical values and the impurity effect influence the incubation time of self-annealing. However, the stress effect and the impurity effect cannot explain the incubation time behavior of self-annealing for DC and PR plating currents separately. That is, the stress discrepancy and a small number of impurities are regarded as two independent factors for self-annealing kinetics. Employing the stress difference and impurities as a combined factor illustrates the different incubation behaviors for various plating waveforms and current densities.

Correlations were found for all electroplating Cu films with various plating conditions. Cu film plated at a high current density had high stress and high impurity content. The PR plating waveform produces films with lower impurity. The texture evolution is determined by an additional stress driving force above the critical point. High current density films takes less time to reach the critical stress, which means a faster self-annealing rate and a lower (111)/(200) ratio in the final stage.

References

[1] J.C. Puippe, N. Ibl, Plat. Surf. Finish 6 (1980) 68.
[2] L.G. Holmbom, B.E. Bacobson, J. Electrochem. Soc. 135 (1988) 2720.
[3] J.J. Toomey, S. Hymes, S.P. Muraka, Appl. Phys. Lett. 66 (1995) 2074.
[4] H. Lee, S.S. Wong, S.D. Lopatin, J. Appl. Phys. 93 (2003) 3796.
[5] T. Ritzdorf, L. Graham, S. Jin, C. Mu, D. Fraser, IEEE, IITC 98 (1998) 166.
[6] C. Detavernier, S. Rossnagel, C. Noyan, S. Guha, C. Cabral Jr., C. Lavoie, J. Appl. Phys. 94 (2003) 2874.
[7] C.V. Thompson, Scr. Metall. Mater. 28 (1993) 167.
[8] V.A. Vas'ko, I. Tabakovic, S.C. Riemer, Electrochem. Solid-State Lett. 6 (2003) C100.
[9] J.M.E. Harper, C. Cabral Jr., P.C. Andricacos, L. Gignac, I.C. Noyan, K.P. Rodbell, C.K. Hu, J. Appl. Phys. 86 (1999) 2516.
[10] Q.-T. Jiang, R. Mikkola, B. Carpenter, M.E. Thomas, Proc. Adv. Metall. Conf., Colorado Springs, 1998;
Q.-T. Jiang, K. Smekalin, ibid (1998).
[11] Q.-T. Jiang, M.E. Thomas, G. Bersuker, B. Foran, R. Mikkola, B. Carpenter, J. Ormando, Mater. Res. Soc. Symp. Proc. 337 (1999) 209.
[12] E.M. Zielinski, R.P. Vinci, J.C. Bravman, Mater. Res. Soc. Symp. Proc. 391 (1995) 103.
[13] S.C. Chang, J.M. Shieh, B.T. Dai, M.S. Feng, Y.H. Li, J. Electrochem. Soc. 149 (2002) G535.
[14] M.S. Yoon, Y.J. Park, Y.C. Joo, Thin Solid Films 408 (2002) 230.
[15] H. Lee, S.D. Lopatin, A.F. Marshall, S.S. Wong, Proceedings of the IEEE International Interconnect Technology Conference, 236, 2001, Piscataway, NJ.
[16] S.H. Brongersma, E. Richard, I. Vervoort, H. Bender, W. Vandervorst, S. Lagrange, G. Beyer, K. Maex, J. Appl. Phys. 86 (1999) 3642.
[17] E.M. Zielinski, R.P. Vinci, J.C. Bravman, J. Appl. Phys. 76 (1994) 4516.
[18] M.F. Doerner, W.D. Nix, CRC Crit. Rev. Solid State Mater. Sci. 14 (1988) 225.
[19] M.W. Lane, C.E. Murray, F.R. McFeely, P.M. Vereecken, R. Rosenberg, Appl. Phys. Lett. 83 (2003) 2330.
[20] M. Stangl, V. Dittel, J. Acker, V. Hoffmann, W. Gruner, S. Strehle, K. Wetzig, Appl. Surf. Sci. 252 (2005) 158.
[21] K.R. Hebert, J. Electrochem. Soc. 148 (2001) C726.
[22] F.J. Humphreys, M. Hatherly, Recrystallization and Related Annealing Phenomena, Pergamon, Oxford, 1995.