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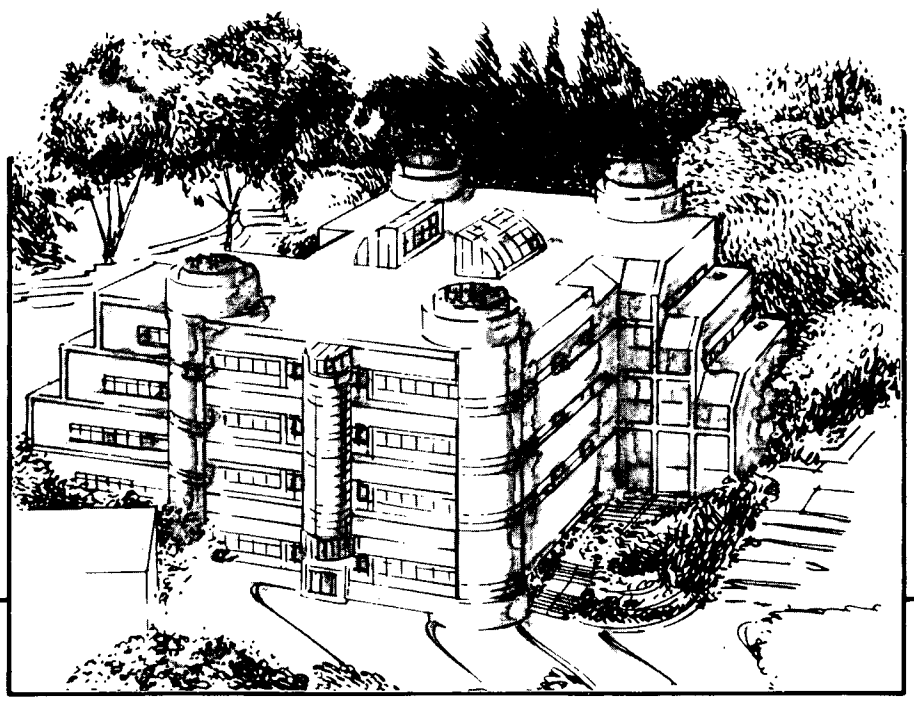

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Grain Growth in Al-2% Cu Thin Films

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ABSTRACT

The grain size and grain growth kinetics in sputter deposited Al-2% Cu films on silicon substrates were determined by TEM for various film thicknesses and anneal times, temperatures and methods. Grain sizes were found to be typically lognormally distributed. The as-deposited grain size (d_o) dependence on film thickness (TH) was found to be $d_o = C\ TH^{1/2}$, due to competitive grain growth during film formation. Annealed grain size (d) after Rapid Thermal Annealing (RTA) for time (t) at temperature (T) is described by the general equation $d - do = C\ TH^{0.7}\ \{t \exp(-\Delta E_a/kT)\}^{1/8}$, where $\Delta E_a = 0.85$ ev for 0.4 μm films and $\Delta E_a = 1.1$ ev for 0.8 μm films. Grain growth is largely saturated for these anneals. Grain growth is shown to be more extensive during RTA anneals than furnace annealing and more extensive in 0.4 μm films than 0.8 μm films for equivalent RTA cycles. The results are discussed in terms of models, simulations and previous results of grain growth in thin metal films.

INTRODUCTION

Al alloy thin films are the predominant class of material systems used for the passive interconnections in integrated circuit technologies, primarily due to the ease in film deposition and patterning and the relatively low Al electrical resistivity. Al-Cu alloys, in the range 0.5% to 4.0% Cu (wt.), are particularly important since it has been shown that Cu additions dramatically reduce the interconnection electromigration (E-M) failure rate [1]. Since the usual conditions for circuit operation or electromigration lifetime testing occur near to or below $T/T_m \approx 0.5$, the diffusion processes during E-M occur mainly along Al grain boundary pathways. Accordingly, interconnects with larger average Al grains lead to longer electromigration lifetimes. Further, more uniform grain size distributions [2,3] and improved fiber (111) crystallographic textures [2,4] also increase the resistance to electromigration failure. The evolution of the grain structure in Al-Cu films is therefore important for integrated circuit technology.

Relatively little experimental work has been done to characterize Al thin film grain growth kinetics. In one study of Al-4% Cu thin films McBee and McComb [5] measured the kinetics of grain growth for anneals in the range 400°C to 500°C. Thompson and Maiorino [6] observed normal and abnormal grain growth in Al-Cu and Al-Cr-Cu alloy films, and measured the abnormal grain growth kinetics.

There have been, however, many studies that show the extent of grain growth, i.e., final grain size and distribution, after extensive anneals. Gangulee and d'Huerle [7,8] showed that Cu, Mg and Cr additions to Al films produced abnormally large grains when the alloying additions were added as elemental layers to the pure Al film matrix. Roberts and Dobson [9] examined Al grain structures for films deposited at different thicknesses and temperatures with several deposition methods. More recently, in an extensive study, Longworth and Thompson [10] observed normal and abnormal grain growth in Al films with W, Mn, Cu, Ag and Cr films in binary and ternary layered combinations.

The results presented here are a portion of the work undertaken to describe the detailed precipitate reactions, Al grain growth behavior and possible growth-precipitate interactions in Al-2% Cu thin films during thermal treatments, including accelerated electromigration testing. Previously we have reported [11] on the Θ ($CuAl_2$) reactions in these films during annealing between 345°C and 472°C. Recently, Θ coarsening and the effects of concurrent Al grain growth were studied [12] in more detail. We presently report the grain growth kinetics in the Al-2% Cu thin films for anneals between 345°C and 472°C. The results include the effects of film thickness and annealing time, temperature, and annealing method (hot wall furnace annealing (FA) and Rapid Thermal Annealing (RTA)) on the average grain size, grain size distribution and kinetics of grain growth.

EXPERIMENTAL

Al-2 (± 0.02)% Cu (wt.) alloy thin films were deposited by MTI, Inc. (San Jose, CA) in a magnetron sputtering system with a base pressure of 2×10^{-5} mTorr. Composition was verified by high temperature plasma mass spectrometry. Films were deposited at ~ 1.2 kÅ per minute in argon at 2 mTorr on unheated substrates. Final thicknesses were 0.4 μm and 0.8 μm. Substrates were (100) 100 mm Si wafers coated with a 1.65 μm cvd borosilicate glass deposited on the front side. (See references [11,13] for details of thin film sample fabrication). The FA cycle was an industry standard "alloy" or "sinter" cycle, 425°C for 35 minutes with an automated slow (20 minutes each) heat up and and cool down during insertion or withdrawal from the constant temperature furnace chamber. RTA was performed at 345°C, 405°C and 472°C for 5 and 15 minutes each. RTA ramp up was approximately 3 seconds and cool down took about 15 seconds. Temperature control was about the same for both systems, ~ ± 2°C. However the overall cycle control is more precisely controlled for RTA than for FA because of the fast heat up and cool down steps. All anneals were performed in forming gas (N_2/H_2) ambients. Grain sizes are the averages of 300 to 600 intercept lengths measured on TEM micrographs taken from several foils for each film condition. Grain size distributions for several films were measured using a digitizing pad and computer software for easy data reduction.

RESULTS

The results of all grain size measurements are listed in Table I. Plan view TEM microscopy of the as-deposited films, figure 1 for the 0.4 μm thick film, shows the Al grain morphology is relatively equiaxed with a fine dispersion of incoherent $CuAl_2$ Θ phase, approximately 200Å in diameter, evenly

Table I. Mean Al grain size (d) by intercept method in Al-2% Cu films from TEM micrographs.

Anneal Temperature	Anneal Time	d, 0.4 μm film	d, 0.8 μm film
as-deposited	- - - - -	0.10 μm	0.14 μm
345°C, RTA	5 min	0.74 μm	1.12 μm
"	15 min	0.84 μm	1.22 μm
405°C, RTA	5 min	0.87 μm	1.39 μm
"	15 min	0.99 μm	1.62 μm
472°C, RTA	5 min	0.98 μm	1.66 μm
"	15 min	1.17 μm	1.84 μm
425°C, FA	30 min	1.02 μm	1.50 μm

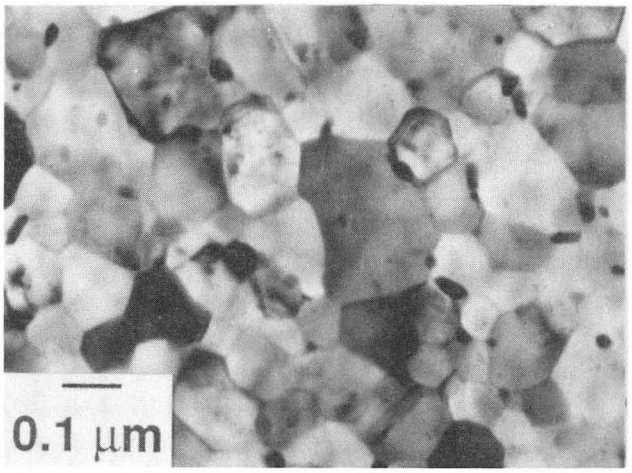

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Figure 1. TEM of 0.4 μm thick Al-2% Cu thick film in as-deposited condition; note small dark Θ phases and largely columnar Al grain structure.

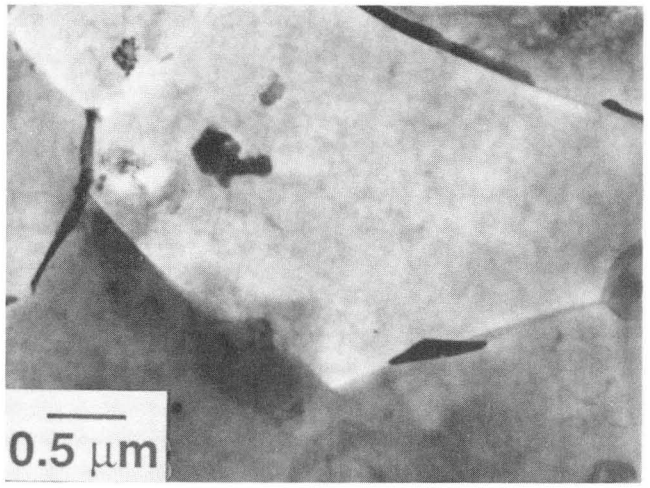

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Figure 2. TEM of 0.8 μm Al-2% Cu film after FA (30 min. at 425°C); note substantial Al grain growth and elongated Θ at boundaries.

distributed at grain boundaries and grain interiors. Cross-section TEM microscopy [11] showed the relatively even distribution of Θ through the film thickness. The Al grain sizes are 0.10 μm and 0.14 μm for the 0.4 μm and 0.8 μm films, respectively. Cross-section TEM also showed small Al grains at the interface. The general morphology is consistent with the structural zone model [14] predictions if it can be assumed that the temperature of these films reached at least approximately 100°C (~ 0.35 T/T_m) during deposition. Further analysis of the processes producing the as-deposited grain structure is presented elsewhere [13]. However the functional dependence of as-deposited grain size (d_o) on film thickness (TH) may be evaluated by assuming

$$d_o = C\,TH^{\alpha} + h \qquad (1),$$

where C and h are constants and α indicates the order of dependence. If we assume that the dependence is linear, that is $\alpha = 1$, the data give c = 0.1 and h = 0.6 μm. The parameter h may be interpreted in this analysis as the size of the stable nuclei during the initial stages of film formation. However this dependence predicts a continuous increase in grain size with film thickness, contrary to the zone model [14] for microstructural development during film formation and the results of Roberts and Dobson [9]. In both the model [14] and the previous experimental results [9] the grain size is shown to saturate with increasing thickness, but increases primarily with increasing deposition temperature. We therefore assume the dependence (equation 1) is more general. Calculation of α, for these limited data, by the relation $\alpha = \ln(d_1/d_2) / \ln(TH_1/TH_2)$, yields $\alpha = 0.5$ with C = 0.16 for both film thicknesses. This as-deposited grain size dependence on film thickness ($\alpha = 0.5$) is identical to the results of Roberts and Dobson (9). Similar analysis of their data, for evaporation depositions of pure Al films 2 nm to 450 nm thick at 295K to 625K, show $\alpha = 0.6$, $\alpha = 0.5$, and $\alpha = 0.5$ for the 295K, 475K and 625K deposition temperatures, respectively. For the RTA annealed films, a similar relation is found for the final grain size dependence on film thickness for each annealing cycle. Analysis of the data in Table I for the film thickness effect for 5 and 15 minute anneals yields an average value $\alpha \sim 0.7$. This dependence is slightly larger than for the case of as-deposited grain sizes, above.

Grain growth during FA at 425°C is extensive as shown for the 0.8 μm film, figure 2. Also shown are the elongated Θ precipitates along grain boundaries [11]. No remnant small as-deposited Θ particles are evident. (TEM *in-situ* annealing experiments [11] confirmed the Θ dissolution-heterogeneous reprecipitation processes for annealing at these temperatures.) Grain growth has also been extensive during all the RTA anneals, Table I. The microstructures after 15 minute RTA anneals at 405°C are shown in figures 3, 4 for the 0.4 μm and 0.8 μm films, respectively. Annealed grain size

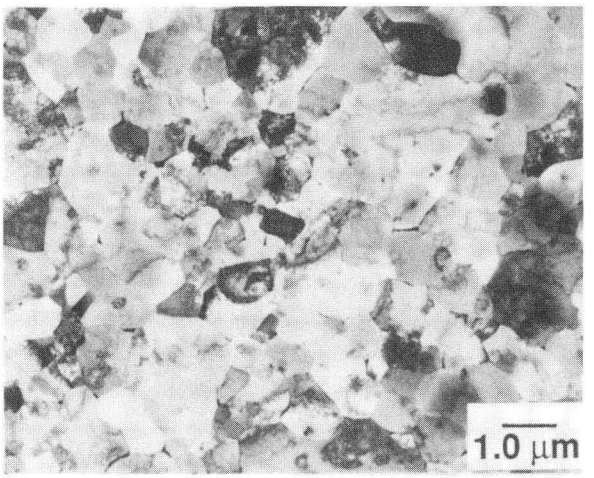

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Figure 3. Plan view TEM of 0.4 μm film after RTA annealing at 405°C for 15 min.

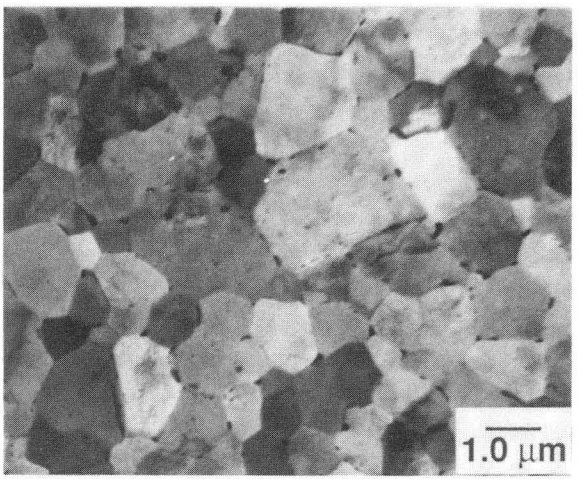

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Figure 4. Plan view TEM of 0.8 μm film after RTA annealing at 405°C for 15 min.

is consistently larger in the 0.8 μm film than in the 0.4 μm film for a given anneal, Table I, as expected. The annealed grain sizes are larger than film thicknesses. However comparisons of the ratios of annealed average grain size to the film thickness (d/TH) show that it is generally larger for the thinner (0.4 μm) film, for equivalent anneals. Note also from the data (Table I) that grain growth has been more extensive in the RTA films that for the FA films. For example, RTA for 15 minutes at 405°C produced equal to or larger grain sizes than for the 425°C 30 minute FA cycle for both films. This suggests that the RTA process is more "efficient" in promoting grain growth in these films.

No significant population of very large "abnormal" grains was seen. Grain size distributions were lognormal for as-deposited and annealed films [13]. Further, film texture determination by x-ray techniques showed primarily (111) fiber orientation for both as-deposited and annealed films [13].

Grain growth for RTA annealing of the 0.4 μm film is shown in figure 5. It is obvious that most of the grain growth has occurred during the 5 minute anneal, and that longer anneals only slightly promote further growth. Note that from the similarity of the 472°C and 345°C growth curves that the presence of a stable (albeit coarsening) Θ phase during the 345°C anneals does not appear to pin the migrating boundaries and retard growth. The data for equivalent RTA anneals of the 0.8 μm films similarly show both the extensive initial growth and the lack growth inhibition due to the Θ phase present at 345°C. The data may be kinetically analyzed by assuming a grain size dependence on annealing time

Table II. Activation energies (ΔE_a) of grain growth processes in Al-2% Cu thin films during RTA anneals at 345°C to 472°C, obtained from least-squares fitting of data; nominal $\beta = 1/8$ was used in calculations (see text).

Film Thickness	RTA Anneal Time	ΔE_a
0.4 μm	5 min.	0.80 ev
	15 min.	0.91 ev
0.8 μm	5 min.	1.10 ev
	15 min.	1.15 ev

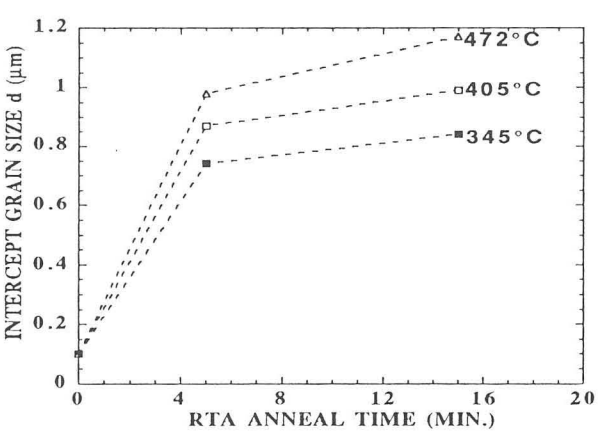

Figure 5. Plot of grain size during RTA annealing of 0.4 μm thick Al-2% Cu film.

$$[d(t) - d_o] / TH^{\alpha} = (M t)^{\beta} \quad (2).$$

Here M contains the thermally activated boundary mobility terms and is of the form $M = M' \exp(-\Delta E_a / kT)$, where M' is a constant, ΔE_a is the empirical activation energy for grain growth, k is Boltzman's constant, and T the absolute temperature. The kinetic rate parameter β may be determined for each anneal temperature by using equation 2 rearranged as

$$\beta = \ln \{ [d(t_1) - d_o] / [d(t_2) - d_o] \} / \ln(t_1 / t_2) \quad (3).$$

β values calculated in this way range unsystematically from 0.08 to 0.18, which are relatively small compared to the value ($\beta = 0.5$) found both theoretically and by simulation. Several factors may explain this discrepancy. The formation of grain boundary grooves [15] by surface diffusion may retard grain growth. Second phase particles may also inhibit grain boundary motion [16]. However only the RTA anneal at 345°C was carried out at temperatures where Θ are stable (with respect to dissolution) and therefore available to retard growth. β values at 345°C for the 0.4 μm and 0.8 μm films were not significantly smaller than for the other conditions. It is likely that grain growth at 345°C was not retarded by the small as-deposited Θ phases since they rapidly coarsen (and *shrink*) when impinged by migrating boundaries. Recent results [12] show such a process for these films. There it was found during annealing of Al-2% Cu films at 310°C that the rate of coarsening of small as-deposited Θ phases was enhanced by the concurrent motion of boundaries during grain growth, and that Θ phase pinning of boundary motion was negligible due to the rapid coarsening of the small Θ phases in favor of large Θ at triple points. The kinetics of Al grain growth were shown to give $\beta = 0.5$. Rather than boundary pinning by either surface grooves or second phases, a more likely explanation for the low β values here is that growth has largely saturated for these times and temperatures.

Arrhenius analysis of the data was performed using equation 2 rearranged for each RTA temperature and time as

$$\ln \{ [d(t) - d_o] / TH^{\alpha} \} = \beta \ln(M t) \quad (4).$$

Linear regression least-squares analysis of $\ln [d(t) - do] / TH^{\alpha}$ versus 1/T for each anneal time will yield the slope = $-\beta\Delta E_a / k$. A nominal value of $\beta = 0.125$ is chosen in order to calculate ΔE_a. The computed activation energies range from 0.80 ev to 1.15 ev, Table II. These compare to the 0.75 ev activation energy for grain boundary diffusion in Al.

Several other observations are relevant. *In-situ* annealings [11] carried out in a high voltage TEM (equipped with a heating stage) showed grain growth and boundary migration to be very jerky with a serrated type of motion. Boundaries were stationary until rapid boundary and related triple point motions occurred. This motion is similar to that described by Mullins [15]. He explained this as due to the pinning effect of grooves on boundary motion, which could lead to "spasmotic" motion as boundaries are alternately pinned or break away and migrate. Recently Frost et al. [17] also observed irregular boundary segment motion during simulations of two dimensional grain growth in thin films. These simulations included the effects of surface grooving leading to grain growth stagnation. The stop-start motions of boundary segments during simulations were due to the effects of changes in the topological features of neighboring boundary segments [17].

DISCUSSION

As described earlier, the kinetic parameter β was lower than expected both from theory and simulations. However other work has shown lower than expected β values. McBee and McComb [5] determined β to be ~ 0.18 for the Al-4% Cu films. A value of β ~ 0.25 was observed in Al-Cr thin films [6]. In this last case the growth kinetics were indicative of abnormal grain growth. However experiments in bulk Al consistently yield β values which are significantly lower than 0.5. In the present

case the low β values are presumed to be due to the saturation of growth at the times and temperatures studied here. Average d increased approximately only 10-15% during RTA annealing from 5 minutes to 15 minutes. However d increased 700-1000% during the first 5 minutes of RTA annealing, figure 5 and Table I. Thus grain growth largely saturates at or before the 5 minute RTA anneals studied. More data taken within the first 5 minutes of annealing, or data at lower temperatures, would better describe the initial rapid but normal grain growth kinetic behavior. Results of grain growth at 310°C in this system in fact showed [12] a kinetic exponent $\beta = 0.5$.

Little other data has been published for the kinetics of thin film grain growth for Al alloy systems. In a study of the mechanical properties of thin Al-Si films [18] isochronal anneals in the temperature range 250°C to 500°C were used to produce a variety of grain sizes. Analysis of their data without knowledge of their empirical β provide $(\beta\Delta E_a) = 0.26$ ev. Their data agree with the present only if their β is chosen to be ~ 0.25 - 0.3. In the previous study [5] of grain growth in Al-4% Cu films, ΔE_a was 1.3 ev (for $\beta = 0.18$), in fair agreement with the longer 15 minute RTA anneal data presented here.

The lognormal grain size distributions found here, in the as-deposited and annealed films, corresponds to previous experimental results [9,19] in Al alloy thin films and realistic simulations [17] of grain growth in thin films which, as described earlier, include effects leading to growth stagnation. The normal grain growth behavior seen here, that is, the lack of abnormal grain growth, and the resultant lognormal grain size distributions, are presumed to be due to several factors. First, normal grain growth is not inhibited by the 2nd phases present, which could, under other circumstances, lead to abnormal grain growth. This due to the relative instability of the Θ second phases due to either complete dissolution at high enough temperatures or coarsening [12] and reduction of their "Zener drag" [10] at lower temperatures. Secondly, there exists no surface energetic or boundary mobility advantage for abnormal grain growth over normal growth [20]. Since the as-deposited grain texture is largely (111), the surface energy minimization driving force is not present. This texture also limits the possibilities where a specific boundary mobility advantage, for example a possibly faster mobility [10] for boundaries between (110)/(111) fiber oriented grains than for those between (111)/(111) grains, may lead to abnormal grain growth.

Interestingly, the longer FA cycles of both films (425°C for 35 minutes) produced a comparable or smaller grain size than for RTA at 405°C for 15 minutes, Table I. This may be attributed to the effects of the very fast RTA heating rate, about 120°C/second, while for automatic loading FA it is about 0.3 to 0.5°C/second. Large stresses [21] build up in the film due to the large difference in thermal expansion coefficients between Al (24×10^{-6}/°C) and the substrate Si (2.3×10^{-6}/°C). Time dependent stress relaxation during these anneals would be more extensive during the slower FA cycle, and would lead to generally lower stresses at equivalent temperatures as compared to the RTA cycles. The larger stresses during RTA may induce large numbers of defects (dislocations, etc.,) which may enhance boundary mobility. These results show that RTA processing is the more efficient process for producing larger grained interconnects via normal grain growth. However, no work has been reported which shows the effect of stress on boundary mobility or grain growth in thin films. Note that during typical anneals thinner Al films may sustain larger stresses than thicker films [21]. Thus a stress effect on (increasing) grain growth would be greater for thinner films. The more extensive grain growth in 0.4 μm films than in 0.8 μm films, as measured by (d/TH) for equivalently annealed films, is consistent with such an effect.

CONCLUSIONS

The dependence of as-deposited grain size on film thickness for 0.4 μm and 0.8 μm thick sputter deposited Al-2% Cu films varies as $d_0 = C\,TH^{1/2}$, in excellent agreement with previous results [9] for pure Al films and in correspondence with models [14] for film formation during deposition. As-deposited and annealed Al grain size distributions were lognormal. Both RTA and FA methods produced normal grain growth, which was unaffected by the presence at 345°C of rapidly coarsening

(shrinking) small Θ phases and growing Θ phases at triple points. RTA was more efficient for promoting grain growth than FA, with RTA anneals producing larger grain sizes than comparable FA anneals. *In-situ* TEM observations of grain growth included jerky boundary motion similar to that described as due to the pinning effect [15] of grooves where boundaries meet the surface and also to the boundary motion observed during recent simulations [17] of normal grain growth in thin films. The 0.4 μm films experienced more extensive grain growth than 0.8 μm films, for equivalent anneals. The kinetics of normal grain growth in the 0.4 μm and 0.8 μm films during RTA annealing are described by the equation $d - do = C\,TH^{0.7}\,\{t \exp(-\Delta E_a/kT)\}^{1/8}$, with $\Delta E_a = 0.85$ ev for 0.4 μm films and $\Delta E_a = 1.1$ ev for 0.8 μm films. The low value of the kinetic factor (1/8) is due to the largely saturated growth for these RTA times and temperatures. Finally, possible effects of thermal expansion mismatch induced compressive stresses, especially during RTA processing, may account for the more extensive grain growth in the thinner films.

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