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Very Large Grained Aluminum Alloy Thin Films for Interconnects

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The rate of electromigration-induced interconnect failure is strongly affected by grain sizes. Abnormal grain growth, also known as secondary grain growth, can lead to very large grains in alloyed aluminum films. For example, initially layered films of Al-2.0%Cu-0.3%Cr will, upon annealing for 30 minutes at 480°C, develop grains with sizes on the order of 100μm. We report here the conditions required for abnormal grain growth in Al-Cu-Cr films.

I. Introduction

Electromigration and Microstructure

It is well known that the rate of electromigration-induced interconnect failure is strongly affected by crystal or grain sizes and grain size distributions. Ames and D'Heurle[1] showed that the mean time to failure (MTF) in lines made from single crystal films were essentially immeasurably long. Gangulee and D'Heurle[2] later showed that very long lifetimes were obtained in lines with grain sizes which were very large compared to the linewidth. More recent research on lines with grain sizes approximately equal to the linewidth has also demonstrated substantial improvements in mean times to failure.[3,4] In the latter case, lines can develop "bamboo" structures in which the planes of all grain boundaries are perpendicular to the line axis. In all of these cases the key to lifetime improvement is the reduction of the number and areas of grain boundaries which provide high diffusivity paths for electromigration.

In developing interconnect technologies it is important that the *deviation* in the time to failure be large as well an the *mean* time to failure. Attardo et al.[5] predicted that electromigration failure times would be lognormally distributed if grain sizes are lognormally distributed. This expectation has most recently been verified by LaCombe and Parks.[6] This relationship between failure time and grain size distributions should hold qualitatively even when grain sizes are larger than or comparable to line sizes. Therefore, the mean time to failure should increase and the deviation in the time to failure should decrease if the mean grain size is increased and the deviation in the grain size is small. We anticipate that during post-deposition annealing abnormal grain growth, also known as secondary grain growth, can lead to such structures.

Grain Growth in Thin Films

If the as-deposited grain size of a film is small compared to the film thickness, then during annealing will result in *normal grain growth*, driven by the reduction of grain boundary area and energy, which can lead to lognormal distributions of columnar grains.[5,7] A *columnar* grain structure is one in which all grain boundaries extend from the top to the bottom of the film and all grain boundary planes are roughly perpendicular to the plane of the film. Once a columnar structure with a mean grain size equal to the film thickness has developed, normal grain growth usually stops. This is known as the "specimen thickness effect"[8] and is known to occur in semiconductors[7] as well as metals. Termination of normal grain growth is usually ascribed to the formation of grooves[9] at the lines of intersection of grain boundaries and film surfaces.

Subsequent grain growth in thin films usually occurs through a process known as abnormal or secondary grain growth.[10] In abnormal grain growth a minor fraction of the grains continue to grow while most grains remain constant in size. These abnormal grains grow at the expense of stagnant normal grains and continue to grow until they impinge on one another. At some intermediate stage, therefore, there is a bimodal distribution of grain sizes. In the final structure, however, the <u>new</u> grain size distribution is again monomodal with a mean grain size that can be many times larger than the film thickness.

Films that have undergone abnormal grain growth are very often characterized by uniform fiber texture. That is, all grains have the same planes parallel to the substrate but have random in-plane orientation. This result strongly suggests that surface energy minimization provides an important part of the driving force for abnormal grain growth[10] and provides for the selective growth of abnormal grains. Therefore, key conditions leading to abnormal or secondary grain growth are:

i) normal grain growth is impeded, and

ii) surface energy anisotropy provides for selective growth of a minor fraction of the grains.

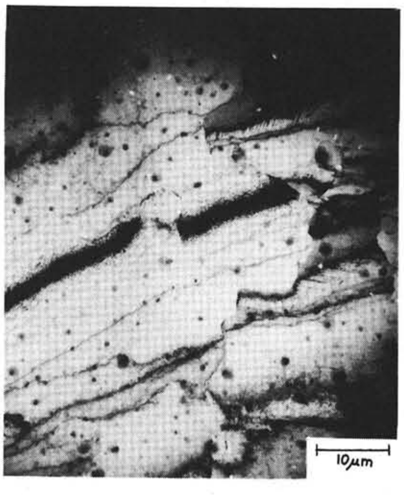
In this paper we report studies of abnormal grain growth in alloyed aluminum films. We expand on earlier work by Gangulee and D'Heurle[11] who reported abnormal grain growth and greatly improved mean times to failure in layered films.

II. Experiments

We have studied grain growth in films of Al alloyed with Cr and/or Cu. Elemental films were electron beam evaporated onto room temperature substrates in a vacuum system with a base pressure of 5 x 10^{-7} torr. Substrates were silicon wafers that had been thermally oxidized to form a 1000Å-thick oxide film. These wafers were subjected to an RCA[12] clean but not a dehydration bake. Films were deposited, one component at a time, to a total thickness of 0.75μm. Best results were obtained for "sandwiched" or layered films for which first 3720Å of pure Al was deposited followed by deposition of 50Å of pure Cu, 10Å of pure Cr and finally another 3720Å of pure Al. These particular films had final overall compositions of Al-2%Cu-0.3%Cr (weight percentages are reported).

After deposition, wafers were sealed in evacuated (~5 x 10^{-7} torr) fused silica ampoules and subjected to isothermal oven anneals for various times and temperatures. Identical results were obtained for samples sealed in 20%H_2 and 80% He. Grain sizes were determined by optical examination of thermal grooves (when samples were annealed above 500°C), backscattered scanning electron microscopy, or transmission electron microscopy (TEM). The crystallographic textures of as-deposited and annealed films were determined via x-ray analysis. Textures of individual secondary grains were also determined via electron diffraction in a TEM. Impurity distributions were monitored via cross-sectional TEM and energy dispersive x-ray analysis.

(a) undergoing abnormal grain growth.



(b) after abnormal grain growth

Figure 1. TEM Micrograph of an Al-2.0%Cu-0.3%Cr-Al film.

III. Results

Figure 1 shows an intermediate state (a) and final result (b) of abnormal grain growth in an initially layered film of Al-2.0%Cu-0.3%Cr-Al. In Figure 2(a) a single grain with (112) texture occupies about two-thirds of the picture and grew from the lower left-hand corner. This grain grew at the expense of stagnant normal grains (upper right) with dominant (111) texture and sizes on the order of a few thousand angstroms. Figure 2(b) shows a similar film after abnormal grain growth is complete. Note that there is a completely occluded grain approximately in the center of the picture and that the grain boundary running down the length of the picture has complex curvature.

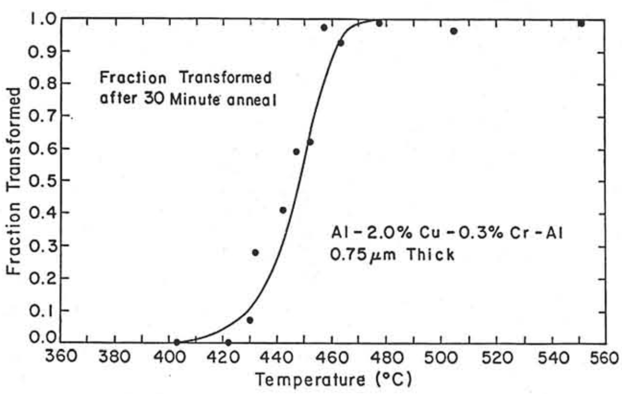


Figure 2. Fraction of the area of a film transformed to secondary grains after 30-minute isothermal anneals. Results are for a 0.75μm thick Al-2.0%Cu-0.3%Cr-Al film.

Table 1 summarizes results for various film compositions and initial structures. Film constituents are indicated in the order in which they were deposited. (Cu and Cr were either deposited in the middle of the film or at the bottom or top.) Note that growth of large abnormal grains leads to a change of dominant crystallographic texture from (111) to (112). Also note that abnormal grain growth was only observed in films containing Cr. Addition of Cu alone did not lead to abnormal grain growth but Cu does appear to catalyze abnormal grain growth in films containing both Cu and Cr. These films have larger final grain sizes and undergo abnormal grain growth at lower temperatures. It is also important to note that abnormal grain growth was only observed in those films which initially had Cu and Cr in the middle.

Table I.
Summary of Grain Growth Results for 0.75μm Films

Film	Dominant Texture	Grain Growth	Approximate Average Grain Size
Al	(111)	Normal	<5 μm
Al-2.0%Cu	(111)	Normal	<5 μm
2.0%Cu-Al	(111)	Normal	<5 μm
Al-2.0%Cu-Al	(111)	Normal	<5 μm
Al-0.3%Cr	(111)	Normal	<5 μm
Al-0.3Cr%-Al	(112)	Abnormal	~ 50 μm
Al-2.0%Cu-0.3%Cr	(111)	normal	<5 μm
2.0%Cu-0.3Cr-Al	(111)	normal	<5 μm
Al-2.0%Cu-0.3%Cr-Al	(112)	Abnormal	~100 μm

Because of the texture change accompanying abnormal grain growth, the fraction of the area of the film transformed to abnormal grains could be determined by monitoring the Al (111) and Si (100) x-ray reflections. The fraction transformed was determined by comparison to Al (111) intensities for as-deposited films. Figure 2 shows the fraction transformed versus annealing temperatures for Al-2.0%Cu-0.3%Cr-Al films subjected to 30 minute anneals. Fully transformed, large-grained films were obtained after annealing at about 480°C. Figure 3 shows the time dependence of the fraction transformed for films annealed at two different temperatures. Note that the rate of abnormal grain growth rapidly decreases with time.

IV. Discussion and Conclusion

The data shown in figures 3 and 4 can be analyzed using a modified form of the Johnson-Mehl-Avrami procedure.[10,13] The fraction transformed is given by

$$x = 1 - \exp(-x_{ex})$$

where x_{ex} is the extended fraction transformed, i.e., the expected fraction transformed if impingement is ignored. The extended fraction transformed is given by

$$x_{ex} = N_a \pi \bar{r}_a^{\,2}$$

where abnormal grains have been idealized as cylinders and average radii $\bar{r}_a$, t is time, and N_a is the number of abnormal grains per unit area, assumed to be constant. The time dependence of the abnormal grain radius can be cast in the general form

$$\bar{r}_a = (\alpha t)^n$$

where

$$\alpha = \alpha_o \exp[(Q)/(kT)$$

and Q is an activation energy, k is Boltzman's constant, T is temperature and α_o is a temperature independent constant. Normally, the rate of abnormal grain growth is taken to be time independent so that n is equal to one.[10] However, if a groove develops at moving boundaries, n should equal 0.25.[9]

By replotting the data of Figure 3 as $\ell n[-\ell n(1-x)]$ vs. t and finding the best line, we determined n to be 0.2 which is sufficiently close to 0.25 to suggest that grain boundary grooving leads to the observed decreasing rate of abnormal grain growth. This interpretation would be consistent with the optical observation of grooves after annealing above 500°C.

Cross-sectional TEM of the Al-2.0%Cu-0.3%Cr-Al films revealed two types of precipitates in the middle of the film, Al-Cu precipitates and Al-Cu-Cr precipitates (determination of the structure or exact composition of these precipitates has not yet been made).[14]

Based on the preceding results and discussion we propose the following tentative, qualitative explanation of the observed abnormal grain growth. Normal grain growth is probably impeded by precipitates retained at the annealing temperature. The appearance of precipitates in the middle of the film implies that the Cu and Cr never fully dissolved and dispersed during annealing. Development of grain boundary grooves may also retard normal grain growth although larger normal grain sizes are obtained in the absence of alloying elements. Since normal grain growth is retarded at a fairly early stage, an unusually large amount of grain boundary energy can drive abnormal grain growth.

The initial films often have dominant (111) textures, possibly the result of grain growth during deposition.[15] Abnormal grain growth leads to a change in texture, suggesting that Cr alloying may additionally lead to modification of the surface energy of the film. This could be due to modifications of the atomic structure of the surface. Similar changes in texture have been previously observed to accompany surface energy modification in Fe-3%Si sheet.[16] Much further work is necessary to test these ideas and clarify the roles of Cr and Cu in promoting abnormal grain growth. It is possible that other alloy additions would have

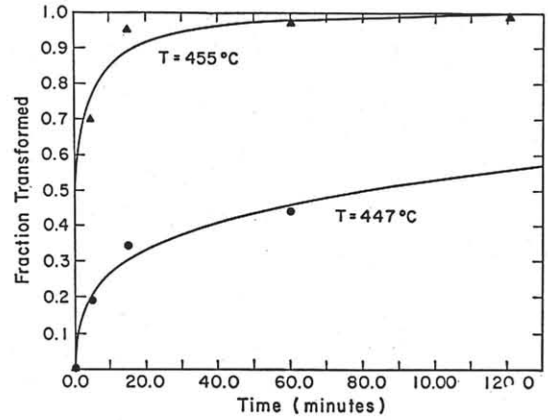


Figure 3. Fraction transformed versus time for films annealed isothermally at 447°C and 445°C. Results are for a 0.75μm thick Al-2.0%Cu-0.3%Cr-Al film.

similar effects.

Whatever the mechanism, Gangulee and D'Heurle demonstrated that lines patterned from similarly large grained films have high mean times to electromigration-induced failure. Their results and the results reported here suggest that substantial modification of grain sizes and grain size distributions are possible through controlled abnormal grain growth.

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