



ELECTROMIGRATION ACTIVATION ENERGY IN PURE ALUMINUM FILMS DEPOSITED BY PARTIALLY IONIZED BEAM TECHNIQUE

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Introduction

The grain boundary diffusion activation energy E_g is one of the most important characteristics of electromigration in aluminum polycrystalline thin films interconnects. The activation energy defines the mobility of an aluminum ion in a grain boundary under electric stress. The higher E_g the less is the probability of motion and hence a reduced material flux.

It is known from Borisov's equation [1] that the less the grain boundary energy, the less is the difference between the diffusion parameters (diffusivity and activation energy) of the grain boundary and those of the lattice. Thus the energy of a grain boundary ensemble in polycrystalline aluminum films must strongly influence the electromigration in them.

Recent studies of grain boundaries have shown that they can be in the equilibrium and nonequilibrium states when they are saturated with extrinsic defects. It is well known from theory and experiment that certain special equilibrium grain boundaries possess an extremely low energy. Diffusion along these grain boundaries is considerably less than along nonequilibrium grain boundaries [2,3].

A nonequilibrium state of grain boundaries is most typical for polycrystalline films used in industry. Strained, short-time annealed polycrystalline films and those treated with particle beams are in the nonequilibrium state. The change from the nonequilibrium to the equilibrium state occurs during relaxation processes. Boundary motion is one of them [4,5].

Recently we showed [6] that the use of the partially ionized beam (PIB) deposition technique and subsequent annealing allows the preparation of aluminum films with nonequilibrium and equilibrium grain boundary ensembles. It was also shown [7] that an interruption lifetime was unusually long in the films with equilibrium grain boundary ensembles.

The aim of this work is to clarify the film microstructure - electromigration activation energy relationship.

Experimental Procedure

The test structure which was used to measure the aluminum drift velocity in order to determine the electromigration activation energy is similar to that described in [8]. The structure was fabricated as follows.

Silicon wafers were oxidized to form a SiO_2 layer about 100 nm thick. Then TiN was rf-sputtered to form a film 250 nm thick on the oxide. After that, high purity aluminum was deposited by the partially ionized beam (PIB) technique at a rate about 2 nm/s onto an unheated substrate with a TiN layer. The ion-to-atom ratio was about 6%. The ions were accelerated by a potential of 3 and 6 kV applied to the accelerating electrode located near the substrate. The thickness of the Al film was 300 nm.

The aluminum was photolithographically etched in a two mask process to form the stripes 670 μm long and 8 μm wide on TiN conductors. These stripes partly cover the titanium nitride. The TiN stripes have the same width as the aluminum ones and they end in Al covered bonding pads. In the first photolithographic step Al was etched with a mixture of H_3PO_4 and H_2O , and TiN with a 1:2 mixture of EDTA (ethylenedinitrilotetraacetic acid tetrasodium salt) and H_2O_2 . Then Al was etched to form aluminum free TiN windows in order to split the Al stripe from the bonding pads. Subsequently, one half of the stripes patterned from Al films deposited at 3 and 6 kV were annealed for 1 hour at 480°C in a vacuum of 10^{-4} Pa. All the experiments were performed in an air atmosphere in the temperature range from 250 to 360°C. The stripes were stressed by a current density of $6 \cdot 10^5 \text{ A}\cdot\text{cm}^{-2}$. The edge displacement was measured with an optical or scanning electron microscope.

Samples for TEM examination were prepared by etching a crater on the back of the substrate with a 5:3:3 mixture of HNO_3 , HF and CH_3COOH to the TiN layer. The TiN layer was removed by ion-milling. Microstructures were examined using the JEOL JEM-2000FX transmission electron microscope at an acceleration voltage of 200 kV. An average grain size was calculated as follows. The area under 1 or 2 hundred grains was determined and then an average area for one grain was derived. An average grain diameter was determined as the square root of an average grain area.

Results

The activation energy E_g can be calculated from the average drift velocities using the following equation:

$$v = j (eZ^*/kT)(\delta/d) D_0 \exp(-E_g/kT),$$

where v is the average drift velocity, ρ - the metal resistivity, j - the current density, eZ^* - the effective ion charge, δ - the effective width of grain boundaries, d - mean grain size, D_0 - the diffusion constant, E_g - activation energy, k - Boltzmann's constant, T - absolute temperature. The Arrhenius plots for the Al films deposited at the accelerating voltages of 3 and 6 kV are shown in Fig.1 a,b. The activation energy E_g was calculated from the slope of the fitted curves.

As-deposited Films

The results of the tests of the as-deposited Al films prepared at the bias 3 and 6 kV are shown in Fig.1 a,b (curves 1). It can be seen from Fig.1 b (curves 1), that the drift velocity of the 6 kV-films is slowed down at the test temperatures higher than 280°C.

TEM studies of the structure of these films revealed numerous defects in the film grains such as dislocations, vacancy clusters and others (Fig.2). The density of the defects is about $2 \cdot 10^{11} \text{ cm}^{-2}$. Most of the grain boundaries in the films have a bent shape and contain numerous extrinsic defects. An average grain size in as-deposited films is 0.45 and 0.23 μm for 3 and 6 kV respectively (Table 1). No texture was observed in the as-deposited 3 and 6 kV-films. After testing, the average grain size in the 3 kV-films remained the same (0.47 μm) in the test temperature range, whereas in the 6 kV-films it depended on the test temperature (Table 2). Probably, this effect causes a slowed down drift velocity in the 6 kV-films at the test temperatures higher than 280°C. Therefore only the lower temperature data points (up to 280°C) were fitted in order to obtain an electromigration activation energy.

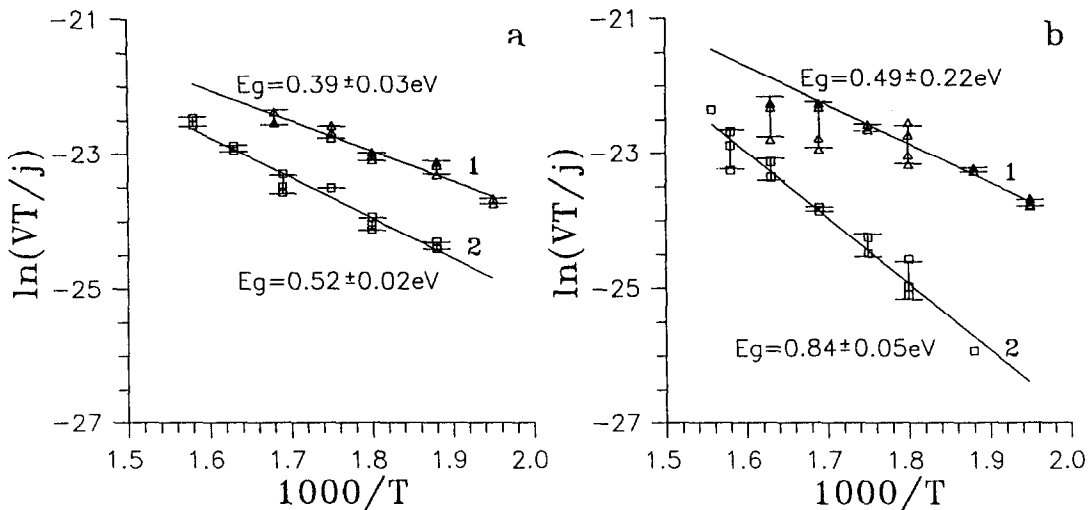


Figure 1. Arrhenius plots of $\ln(VT/j)$ vs $1000/T$ for Al films deposited at the bias potential 3 kV (a) and 6 kV (b). curves 1 - as-deposited and curves 2 - annealed films.

Annealed Films

Drift velocities in the annealed films are substantially slower (this has been observed in [9]) especially in 6 kV-films. The activation energies are equal to 0.52 and 0.84 eV for the 3 and 6 kV-films, respectively (Fig. 1 a,b; curves 2). The structure of the films is markedly different as well. After annealing an average grain size in the 3 kV-films increased up to 0.66 μm (standard deviation $\sigma = 0.55$); however, the defect density was rather high (about $5 \cdot 10^{10} \text{ cm}^{-2}$). Annealing twins were found in the films (Fig.3). Twin boundaries also contained many extrinsic defects (Fig.4 a).

After annealing, the average grain size in the 6 kV-films increased to 0.62 μm (standard deviation $\sigma = 0.7$). The defect density decreased by more than two orders of magnitude and was less than 10^9 cm^{-2} . Annealing twins were also found in the films but the number of them was smaller than in the 3 kV films. Most of the grain boundaries were free from extrinsic defects (Fig.4 b). No texture was observed in the 3 and 6 kV-films after annealing.

Discussion

As has been shown above, the electromigration activation energy after annealing increases from 0.39 to 0.52 eV in the 3 kV-films and from 0.49 to 0.84 eV in the 6 kV-films. The drift velocity in annealed films is slower in the 6 kV-films than in the 3 kV-films. Since the average grain size in the 6 kV-films is somewhat less than that in the 3 kV-films, and the drift velocity is substantially slower in the 6 kV-films than in the 3 kV-films, this decrease can be due to a decrease in the grain boundary diffusion coefficient (owing to the improvement of the grain boundary structure) rather than to a decrease in the total cross-sectional area of the grain boundaries. An abrupt decrease in the defect density in grain boundaries in the 6 kV-films supports this interpretation. Thus, relaxation processes in the films during annealing influence the immunity to electromigration.

From our viewpoint, the effects observed in these films can be explained in terms of self-cleaning, which was observed during the deposition of Al, Ag and Cu films by PIB [10-12]. This self-cleaning is possible

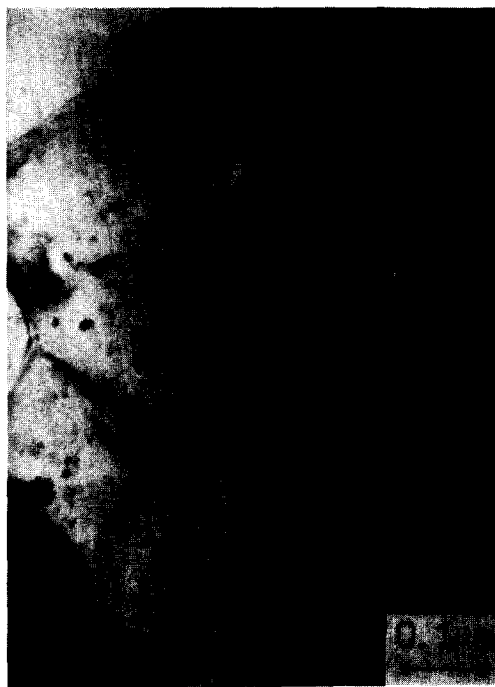


Figure 2. Transmission electron micrograph of the as-deposited film prepared at 6 kV.



Figure 3. Annealing twins in the 3kV-film annealed at 480°C for 1 hour.

owing to the desorption of residual gas impurities through sputtering by the self-ions. The efficiency of self-cleaning is likely to increase with an accelerating voltage applied to substrate holder or accelerating electrode. Therefore, the number of impurities in the 3 kV-films is greater than in the 6 kV-films. These impurities (as a rule it is oxygen which forms oxide surrounding Al grains [13]) may exert a drag force on the boundary and defect motion [14], slowing the rate of grain growth and the migration of the defects to sinks. Indeed, a high defect density was observed in the 3 kV-films after annealing and the ratio d_a/d_b of the average grain size after annealing to that before annealing was 1.4.

TABLE I
Parameters of Grain Structure of the PIB Aluminum Films Tested at the Temperature 300°C.

Bias, kv	as-deposited films before tests		as-deposited films after tests		annealed films after tests		d_a/d_b
	average grain size d , μm	standard deviation of grain size distribution σ	average grain size d , μm	standard deviation of grain size distribution σ	average grain size d , μm	standard deviation of grain size distribution σ	
3	0.45	0.40	0.47	0.40	0.66	0.55	1.4
6	0.23	0.35	0.35	0.44	0.62	0.70	2.7

TABLE 2
Average Grain Size of the As-deposited 6-kV Films Electromigration Tested at Different Temperatures.

Test temperature, °C	240	260	280	300	320	340
Average grain size, m	0.32	0.33	0.33	0.45	0.48	0.52

On the contrary, in 6 kV-films a great number of self-ion bombardment -induced defects, which are free from impurities, promotes increase in the grain growth rate [15]. A population of large grains, sizes of which exceed the film thickness by about an order of a magnitude, appears in the films. The grain growth rate increases ($d_g/d_0 = 2.7$) in comparison with the 3 kV-films. Due to the high mobility of defects their density drops after annealing (defect density is less than 10^9 cm^{-2}). A greater relaxation takes place in the 6 kV-films in comparison with the 3 kV-films.

Thus, the immunity to electromigration of the 6 kV-films is higher than that of the 3 kV-films in spite of the fact that the average grain size is lower and the standard deviation of the grain size distribution is higher in the 6 kV-films than in the 3 kV-films. Therefore, the grain boundary structure is probably more important for electromigration in these films than the average grain size and the standard deviation of grain size distribution. Obviously, the ratio d_g/d_0 (or of the grain growth rate) is related to the degree of relaxation of the grain boundary structure. The higher d_g/d_0 , the higher the immunity to electromigration.

A similar behaviour of pure Al films deposited by the PIB technique onto SiO_2/Si substrates was observed earlier [7]. The interruption lifetime increased in those films with ratio d_g/d_0 (23, 65 and more than 200 hours for 1.3, 4 and 11.8 respectively). The grain growth rate was higher, probably due to a higher vacuum during deposition ($3 \cdot 10^{-5} \text{ Pa}$ in ref.[7] and $3 \cdot 10^{-4} \text{ Pa}$ in this study) and hence lower impurity content, because the duration and the temperature of annealing were the same. The effect of polycrystalline TiN in this study and amorphous SiO_2 in ref.[7] used as substrates should not be ignored, but it requires further investigations.

Conclusion

The electromigration activation energy E_g increases with the accelerating voltage during the deposition of pure aluminum films by the PIB technique. The defect density in the annealed films decreases with the increase in

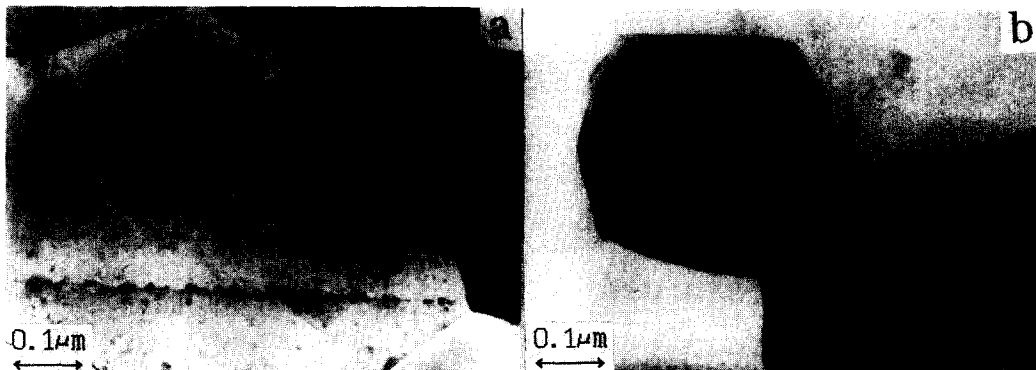


Figure 4. Transmission electron micrographs of the annealed films. (a) 3kV-film, (b) 6kV-film.

the accelerating voltage; the structure of grain boundaries is improved. It was found that the drift velocity of the annealed films becomes slower and E_g higher with increasing accelerating voltage. Thus, the improvement of the grain boundary structure and the decrease in the defect density in the films reduces the material flux during electromigration in polycrystalline aluminum conductors.

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