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Mechanical strength lowering in submicron Cu thin films by moderate DC current

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Abstract This letter reports an experimental investigation into the direct current (DC) induced reduction in the yield strength of 60 $\sim$ 700-nm-thick Cu films. Results show that the larger the current density and the thinner the film, the greater the reduction when the film thickness is below about 340 nm. This reduction could be described on the basis of dislocation buckling, which, caused by the electron wind of the current flow, induces an increase in the dislocation length and a decrease in the critical stress for multiplying the dislocation.

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Lower resistivity and improved electromigration resistance have been putting Cu rapidly forward to replace Al for advanced interconnects in multilevel metallization of ultralarge scale integrations. As the dimension of the integrated circuits continues to shrink, it is becoming ever more crucial to understand the operational properties of Cu thin films with size in submicron scale [1], in order to ensure the reliability of Cu interconnects and favor the design and fabrication of integrated circuits. Most important properties of the submicron-thick Cu films, such as tensile properties [2, 3], mechanical fatigue [4, 5], thermal or thermomechanical fatigue [6], and electromigration behaviors [7], have been elaborately investigated and well revealed. Electromigration is termed the phenomenon of atomic flux in metals under an applied current, which could induce a stress gradient and then initiate voids after a somewhat long duration, e.g., several hundreds of hours. For the metals used in the microelectronic interconnects, the required current density that could cause an appreciable electromigration is usually in the range of 10^5–10^6 $\mathrm{A/cm^2}$. As one of the major failure mechanisms of the Cu films, the electromigration-induced void formation and growth have attracted extensive attention and some statistically based life-predicted models have been suggested [8–10] that might be referred to as design criteria.

However, on another important failure mechanism, the weakening in strength of Cu films under applied electric current, little systematic work has been carried out, which results in a lack of the design criterion on strength requirement. Because the shrinking in size and resultant increase in current density make the electricity-related mechanical failure of thin films more and more important in integrated circuits, it is urgent to well understand the yield strength of Cu films and its size effect in application of an electric current, which could be helpful for strength design in Cu metallization.

The yield strength of films could be determined by a variety of methods, such as tensile testing, indentation, microbeam bending, and bugle testing [11–13]. In comparison, tensile testing has the advantage of providing mechanical parameters directly, without the need of a model-based data analysis. However, the tensile testing of freestanding films, especially with size in submicron scale, is complicated and difficult. Recently, thin films deposited on compliant polymer substrates have been used to determine the mechanical properties of films [3, 4, 14], because the polymers usually have large elastic limits and their response could thus easily be subtracted from the overall measurement. On the other hand, many flexible electronics that are

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being developed for diverse applications, such as paper-like displays [15] and sensitive skins [16], comprise the same configuration of thin metal films deposited on compliant polymer substrates. Therefore, the investigations on systems of metal film/polymer substrate should have a multiplicity of purposes.

Using polyimide as substrate, the yield strength of Cu films has been determined and an increase of yield strength with decreasing film thickness has been observed [3, 14]. Nevertheless, the actual service conditions of Cu films used in integrated circuits involve also the electric current pulse and the influence of electric current on the yield strength has not become well known. In this letter, we perform experiments to investigate the current-affected size effect in the yield strength of Cu films with a series of thicknesses, with the aim of mapping the electroplastic effect in thin films used in integrated circuits. Because of the application of a much lower current density and a much shorter duration, the present experimental conditions are distinctly different from those used to investigate the dynamic electromigration-induced failure.

The Cu films, with a thickness range from 700 nm down to 60 nm as determined by Rutherford backscattering, were deposited through a dogbone-shaped mask on a rectangle polyimide substrate (4 mm × 20 mm) by magnetron sputtering. The sputter current was 0.45 A and the bias voltage −80 V, which resulted in a deposition rate of about 5.9 nm/min. This low-energy slow deposition rate was experimentally determined to have almost no effect on the mechanical properties of the 125-µm-thick polyimide duo to its good temperature-endurance. Prior to deposition, the polyimide was firstly cleaned by Ar ion bombardment. The as-deposited films were in-situ annealed at 100°C for two hours to eliminate the residual stress. X-ray diffraction (XRD) measurements revealed that all the Cu films were polycrystalline and no obvious texture was found even in the thinnest 60-nm film. Based on quantitative microscopy, the average grain size of the films was determined using transmission electron microscopy (TEM) (see the inset in Fig. 2(a)).

Uniaxial tensile testing was performed using a MTS microtensile tester with a 50-N force transducer at a constant displacement rate of 0.002 mm/s. The force and strain were automatically recorded by machine and a high-resolution laser detecting system, respectively. When testing the Cu film/polyimide systems with different film thicknesses, the total force on the systems is a sum of the forces on the Cu film and the polyimide substrate. In order to obtain the strain-stress data of Cu films, the force on the substrate should be subtracted from the total one [14]. Therefore, pure polyimide with the same width and length as those of Cu film/polyimide systems was also tested and the strain-force data were recorded for subtracting. Figure 1(a) typically shows the derived stress-strain curves of the Cu films

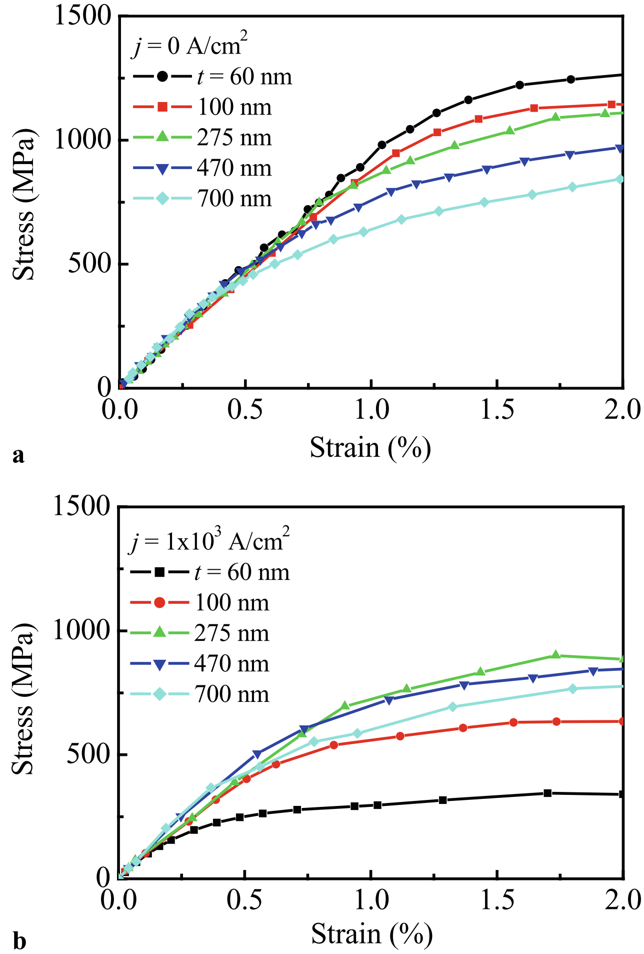

Fig. 1 Stress-strain curves of Cu films with different thicknesses: (**a**) under no DC and (**b**) under a DC of 1×10^3 A/cm^2

with different thicknesses. Direct currents with different current densities were applied on the films using a home-made attachment to realize the coupled application of both electric loading and force loading, and the influence of DC on the stress-strain curves could clearly be observed in (b) by comparing with (a). In order to avoid the oxidation of Cu films, only the somewhat low current density of less than 1×10^4 A/cm^2 was applied, which is much lower than one that could induce electromigration. These low current densities, according to our finite element calculations as similar to Mönig et al.'s work [6], could induce only a temperature increase less than 2 K in the Cu films. This has also been confirmed by a Raytek miniature infrared sensor (MI) measurement, where the experimentally determined temperature increase of the film surface was surprisingly found to be within only 1 K. After testing, the surface morphology of the tested samples was observed using scanning electron microscopy (SEM). It was found that there were no oxides formed on the surface of the Cu films, which was further verified by XRD and Auger electron spectroscopy (AES) measurements on the tested samples. In order to make clear whether any creep

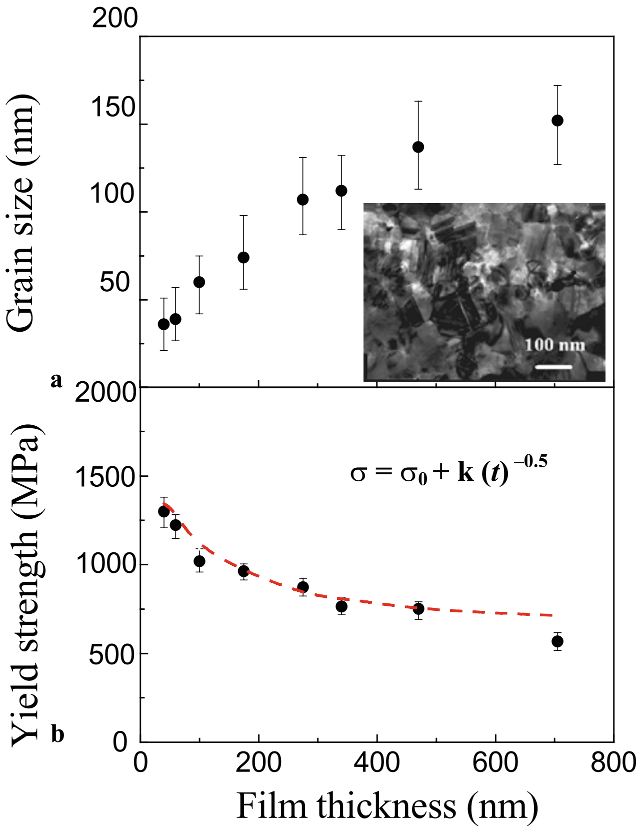

Fig. 2 Thickness dependence of (**a**) grain size, and (**b**) yield strength of Cu films without application of direct current. The inset in (**a**) is a typical TEM image illustrating the grains, and the lines in (**b**) are calculation fits

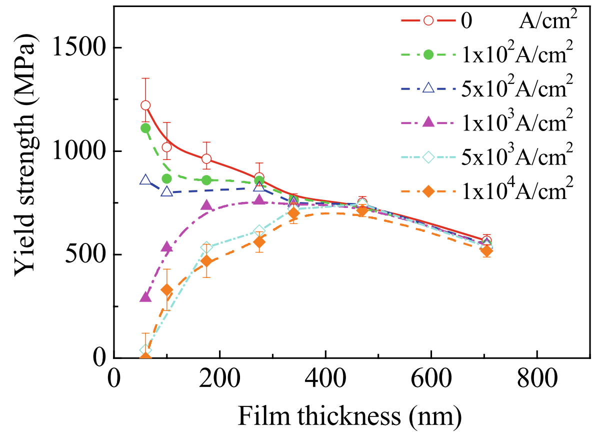

Fig. 3 Thickness dependence of yield strength of Cu films with application of direct currents with different current densities

effect existed in the measurement, some supplement experiments were also performed, where the films were tensed to a stress level and then kept changeless in the same stress condition. This stress level was higher than the yield strength of the films. In this course, DC was applied all the time and the electrical resistance of the samples was monitored using the Keithly source-meter to relate it to the change in longitudinal strain. It was revealed that, even after 22 hours in application of DC, no additional elongation appeared, as long as the thin films tensed in a stationary stress condition. This indicates that no creep effect existed and therefore the present experimental results are not induced by the creep effect.

The measured thickness dependence of grain size, d, and yield strength, σ_y (determined as the 0.2% offset) of Cu films without the application of electric current are shown in Fig. 2(a) and (b), respectively. The grain size is found to decrease with decreasing film thickness, $\boldsymbol{t}$, while the yield strength enhances with decreasing $\boldsymbol{t}$. These trends are well consistent with previous results [4, 12, 17]. Moreover, the values of yield strength we determined for the Cu films are found to be close to those determined either from freestanding Cu films or from Cu film/polyimide systems [14]. The strong dependence of the yield strength on the Cu film thickness of submicron size has been suggested [14] as a result of the thickness effect that could be quantitatively described by strain gradient plasticity theory [18]. The same dependence of the yield strength on the film thickness was also found in our experiments, which follows the expression of $\sigma_y = \sigma_0 + k(t)^{-n}$, where σ_0 is the lattice friction stress (116 MPa for Cu [14]), and exponent n $(= 0.5)$ and k $(= 362\ \text{MPa}\ \mu\text{m}^n)$ are constants that are close to those given by Yu and Spaepen [14]. In a word, the normal inverse thickness-dependent yield strength was observed in the present submicron Cu films without application of electric current. This close dependence of the yield strength on film thickness should be partly induced by the finite size of the films, i.e., the pinning effect of the film surface and film/substrate interface on the deposited dislocations segments, and it should partly result from the strengthening of the fine grain size, which is almost in scale with the film thickness, as shown in Fig. 2.

On application of direct current, however, the yield strength of the films reduces with increasing current density and the dependence of the yield strength on the film thickness may exhibit some trends distinct from that without direct current, as shown in Fig. 3. When the applied current density, j, increases up to values larger than $5 \times 10^2\ \text{A/cm}^2$, the monotonic inverse dependence of the yield strength on the film thickness will no longer be found. More precisely, the yield strength still increases with decreasing thickness within the thickness range of larger than about 340 nm, while for a thickness less than about 340 nm, the yield strength reduces with decreasing thickness. This means that, on application of a current density larger than $5 \times 10^2\ \text{A/cm}^2$, a significant reduction in yield strength will be induced when the film thickness reduces down to less than about 340 nm.

It was also observed that the larger the current density in the films and the thinner the films, the greater the reduction of the yield strength. Compared to the normally inverse

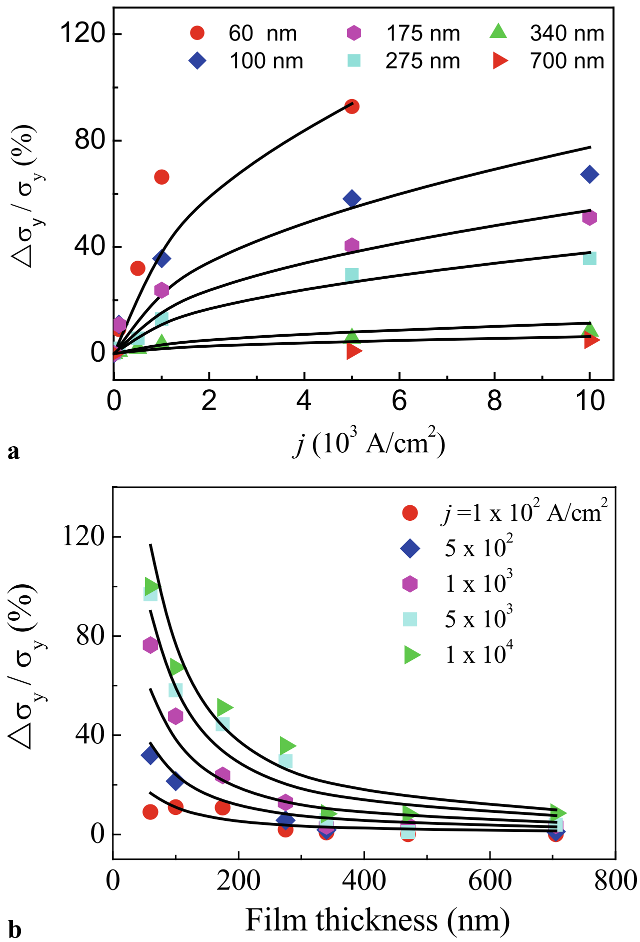

Fig. 4 (**a**) Dependence of relative reduction in yield strength, $\Delta\sigma_y/\sigma_y$, on the current density as a function of film thickness; (**b**) thickness dependence of relative reduction in yield strength, $\Delta\sigma_y/\sigma_y$, as a function of current density. All the curves are calculation fits

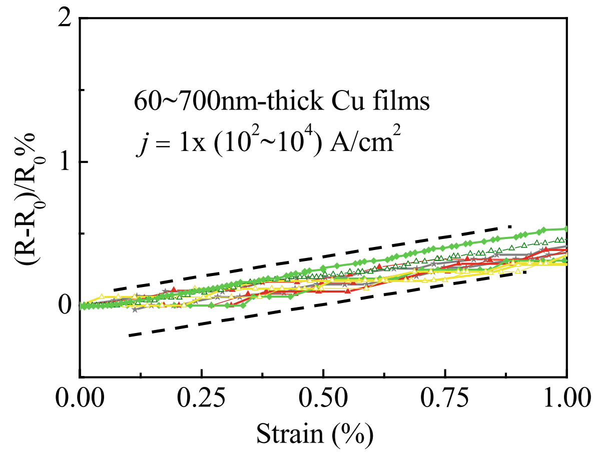

Fig. 5 Dependence of relative change in electric resistance, $(R - R_0)/R_0$, of Cu films (60–700 nm) on the applied strain under different current densities (1×10^2–1×10^4 A/cm^2)

dependence of the film yield strength upon the film thickness without DC, i.e., the thinner the films the larger the yield strength and vice versa, as shown in Fig. 2(a), the yield strength increases abnormally as the film thickness increases for the thinner Cu films under the higher DC density, leading to only less than 5% yield strength of a 60-nm-thick Cu film, retained at a current density of only 5×10^3 A/cm^2, compared to its counterpart without DC, much lower than that of the 700 nm thicker film at the same current density.

In an application of direct current, we define the reduction in yield strength of the films, $\Delta\sigma_y$, as the difference between the yield strengths without (σ_y) and with (σ_y^c) direct current, i.e., $\Delta\sigma_y = \sigma_y - \sigma_y^c$. The dependence of the relative reduction in the yield strength, $\Delta\sigma_y/\sigma_y$, on the current density is depicted in Fig. 4(a) as a function of film thickness, where the dots are the measured results and the curves are calculated ones that will be described later. It is clearly shown that the increasing current density induces more reduction in the yield strength, especially in thinner films. A significant size effect is then found in the current-induced reduction in the yield strength, as shown in Fig. 4(b), where $\Delta\sigma_y/\sigma_y$ is used as a vertical axis. Understanding this size effect may be very helpful for strength design in Cu film metallization, because it is surprisingly revealed that, when the film thickness reduces to less than about 340 nm, a rapid reduction in the yield strength could be induced with application of a low direct current of only 10^2 A/cm^2. The application of such a low current density, as we mentioned above, will induce a very limited temperature increase of less than 1 K. As a result, the relative variation of the electrical resistance, $(R - R_0)/R_0$, is almost linear for various film thicknesses with applied strain up to 1%, as shown in Fig. 5, and the relative difference in $(R - R_0)/R_0$ among different film thickness is found to be within the range of $0 \sim 0.18\%$ at any identical strain.

Conrad and Yang [19] have found that the application of an electric field yielded a 20–25% decrease in the flow stress of the Cu foil, and they attributed this mechanical strength lowering to the reduction in the dislocation density during straining, which was believed to be related to the electric charge density at the specimen surface. In comparison, the decrease of the strength in present experiments was caused by the application of electric current, where electromigration-induced damage may be a very well possible softening mode. However, predictions based on the classic Black equation [20] reveal that, for the present thinnest 60-nm film with the present largest current density of 1×10^4 A/cm^2, the median time to failure induced by electromigration should be about 5×10^6 hours, very much longer than the time (t_n, about 10 minutes) needed for samples tensed to yield a point in the present experiments, even though the effects of the refined grain sizes in the thinner Cu films were taken into account, where many grain boundary pathways were available to accommodate the electromigrating atoms.

Besides, we have carried out two types of experiments for comparison. One was to apply DC on the Cu films for half an hour and subsequently tense the Cu films to determine the yield strength with no longer application of electric current. The other was to tense the Cu films directly to determine the yield strength without DC all the way. Comparing results revealed that there is not any difference in the yield strength values between the two cases. This suggests that the application of DC for half an hour (longer than t_n) could not result in any significant electromigration phenomenon. As to the present mechanical strength lowering, it seems to be most possibly associated with the dislocation buckling caused by the electric field. Suo [21] has suggested that, in application of DC, pinned dislocations in conduct metals could buckle when the current density exceeds a critical value. This is because the electric wind of current flow overcomes the line tension and drives the dislocation bow from its pinning points. The dislocation buckling will induce an increase in the length of dislocation and so decrease the critical stress needed to multiply the dislocations, which will cause the reduction in yield strength. Approximately, the increase in dislocation length ΔL should be proportional to the square root of the applied electric density j. For quantitative purposes, the pinned dislocation is suggested to have the length of L, and the critical stress is $\tau = Gb/L$, where G is the shear modulus and b Burger's vector. After the application of DC, the buckled dislocation has the increased length of $L + \Delta L$, and the critical stress $\tau' = Gb/(L + \Delta L)$. The relative reduction in critical stress is then as follows:

$$\frac{\Delta\tau}{\tau} = \frac{\tau - \tau'}{\tau} = \frac{\Delta L}{L + \Delta L} \approx \frac{\Delta L}{L} \propto \frac{\sqrt{j}}{L}. \tag{1}$$

The dislocation length L should be related to the spacing of pinning obstacles. In the thin films used in the present experiment, the most possible pinning obstacle is either the film surface (film/substrate interface) or the grain boundary and so L should be proportional to either the film thickness $\boldsymbol{t}$ or to the grain size d. In consideration of the aforementioned result that d is almost in scale with $\boldsymbol{t}$, the relative reduction in yield strength, $\Delta\sigma_y/\sigma_y$, should then be proportional to $\sqrt{j}/t$, i.e.,

$$\frac{\Delta\tau}{\tau} = Q\frac{\sqrt{j}}{t} \tag{2}$$

with the scaling constant Q representing the comprehensive influencing of material characteristics, temperature, etc. For present Cu films, Q is calibrated as $1.04 \times 10^{-11}\ \mathrm{m}^2/\mathrm{A}^{1/2}$ by fitting the measurement results, as shown in Fig. 3. The good agreement between calculations and measurements indicates that the influence of dislocation buckling could be responsible for the reduction in yield strength of Cu films. At this point, it must be especially noted that this significant reduction in yield strength with application of low DC could only be observed in thin films. In thick films and bulk metals even with nanometer grains, however, this phenomenon is hardly found because they both have dimensions much larger than those of the thin films. This could be easily understood by referring to Fig. 4(a), where the 700-nm thick film undergoes a weak reduction in yield strength even under a DC up to $10^4\ \mathrm{A/cm^2}$.

In summary, we deposited Cu films with a thickness range of 60–700 nm on compliant polyimide and investigated the influence of direct current on the yield strength of the films using a microtensile tester as well as some attachments of our own design. Without the direct current, the yield strengths of the films were found to monotonically depend on the film thickness, i.e., the thinner the films, the larger the yield strength and vice versa, especially when the thickness is below about 340 nm, revealing a significant thickness effect. However, the application of direct current reduced the yield strength of the films and changed the dependence of the yield strength on the film thickness. Of special interest to note is that, under a low current density only at the level of $10^2\ \mathrm{A/cm^2}$, a rapid reduction in the yield strength was found when the thickness also is below about 340 nm. It is suggested analytically that the electron wind of the current flow could cause the buckling of pinned dislocations and so induces an increase in dislocation length and a decrease in critical stress for multiplying the dislocation, which results in the DC induced reduction in the yield strength. The results for current-induced weakening in the yield strength of submicron-thick Cu films might be used for reference in strength design of Cu film metallization.

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