東北大学機関リポジトリ
TOUR
Tohoku University Repository

Atomic force microscopy study of plastic deformation and interfacial sliding in Al thin film: Si substrate systems due to thermal cycling

著者	Chen M. W., Dutta I.
journal or publication title	Applied Physics Letters
volume	77
number	26
page range	4298-4300
year	2000
URL	http://hdl.handle.net/10097/51834

doi: 10.1063/1.1332098

Atomic force microscopy study of plastic deformation and interfacial sliding in Al thin film: Si substrate systems due to thermal cycling

M. W. Chen and I. Dutta[a)]
Center for Materials Science and Engineering, Department of Mechanical Engineering, Naval Postgraduate School, Monterey, California 93943

(Received 28 August 2000; accepted for publication 18 October 2000)

A method is proposed to measure the plastic deformation of thin metallic films on Si substrates induced by thermal cycling. The cross-sectional profiles of pattern-grown square Al films with a thickness of ~ 250 nm and a size of $\sim 6\ \mu\text{m} \times 6\ \mu\text{m}$ were measured before and after thermal cycling by employing an atomic force microscope. With the assistance of statistical analysis, the change in the size and shape of the thin films were determined. Based on theoretical considerations, the thermal cycling deformation of thin films is attributed to creep and plasticity effects, accommodated by diffusion-controlled interfacial sliding. © *2000 American Institute of Physics.*
[S0003-6951(00)04250-9]

Mechanical properties of thin films on semiconductor substrates are of much interest to the microelectronic industry. It is well known that thin films have properties different from their bulk counterparts.[1,2] Because traditional testing techniques cannot be applied to the measurement of thin films, developing new techniques to characterize the mechanical behavior of thin films is an active area of research.[2] Owing to differences in thermal expansion between thin metallic films and semiconductor substrates in microelectronic devices, high stresses can develop during thermal excursions experienced in processing steps or service, which may induce plastic deformation of the thin films accompanied by creep, and possibly, interfacial sliding. These stresses and deformation processes can have a pronounced effect on the reliability of microelectronic devices and components. Various methods have been proposed to estimate the thermal and/or intrinsic residual stresses, such as curvature measurement, indentation, etc.[1,2] In particular, substrate-curvature measurements have been widely utilized to study stress evolution during thermal cycling of thin metallic films on Si substrates.[1,3,4] However, methodologies for the measurement of plastic deformation of thin films induced by thermal stresses have not received much attention to date. To date, no direct measurement of plastic deformation of thin films associated with thermal cycling has been reported.

Previous studies have noted that commensurate with stress/temperature changes during thermal cycling, a multitude of plastic deformation mechanisms, such as dislocation glide, dislocation creep and diffusional flow may appear.[3,4] In addition, near the edges of the film, where interfacial shear stresses are significant, film plasticity may be accommodated by diffusionally controlled interfacial sliding, resulting in relative size changes between the film and susbtrate. It has been demonstrated theoretically and experimentally that such interfacial sliding is prominent near fiber ends during thermal cycling in fiber reinforced metal-matrix composites, and occurs via interface-diffusion-controlled diffusional creep.[5,6] Experiments on thermal cycling of high-density Cu/Ta/polyimide interconnect structures have also shown evidence of diffusionally accommodated interfacial sliding between Cu lines and Ta liners, driven by the interfacial shear stresses generated due to out-of-plane thermal expansion mismatch between Cu and polyimide.[7] Clearly, since significant interfacial shear stresses only exist near the edges of a thin film, interfacial sliding is an edge effect, and as such, can be ignored for large-area films. However, with decreasing lateral film dimensions (e.g., in electronic applications where linewidths are at the submicron level) these effects are likely to become important. Therefore, an understanding of the roles of film plasticity, as well as interfacial sliding, is essential for developing and designing reliable electronic devices.

In this letter, we directly measure plastic deformation of thin Al films on Si substrates by using an atomic force microscope (AFM). Based on the experimental results, the effects of film plasticity and interfacial deformation on the dimensional stability of thin films are discussed.

Physical vapor deposition was employed to grow pure Al films on Si (100) wafers covered by Ni masks with nominally $6\ \mu\text{m} \times 6\ \mu\text{m}$ square holes. The surfaces of the Si wafers were cleaned prior to film deposition in accordance with standard microelectronics practice. A whole array of square Al films with a thickness of ~ 250 nm was deposited at a substrate temperature of 428 K, and a deposition rate of about 20 nm/min. The samples were subsequently annealed at 623 K for 15 min prior to removal of the Ni masks in order to promote diffusion bonding at the interface, and stabilize the film microstructure. After removing the Ni masks, the samples were subjected to thermal cycling from 293 to 623 K in a vacuum furnace at a pressure of $\sim 5.0 \times 10^{-7}$ Torr. The thermal cycle began with a ramp-up at ~ 20 K/min to 623 K, and concluded with a cooling rate of ~ 2.5 K/min to ~ 373 K. The cooling rate from 373 to 293 K could not be controlled, but the average cooling rate was below 1 K/min. This cycle was repeated five times for each sample. To identify the deformation induced by thermal cycling, the cross-sectional profiles of the square films were measured before and immediately after the thermal cycling by an AFM at room temperature in air. All the profiles were measured at the middle of the square films with the direction

[a)]Author to whom correspondence should be addressed; electronic mail: idutta@nps.navy.mil

Downloaded 01 Sep 2011 to 130.34.134.250. Redistribution subject to AIP license or copyright; see http://apl.aip.org/about/rights_and_permissions

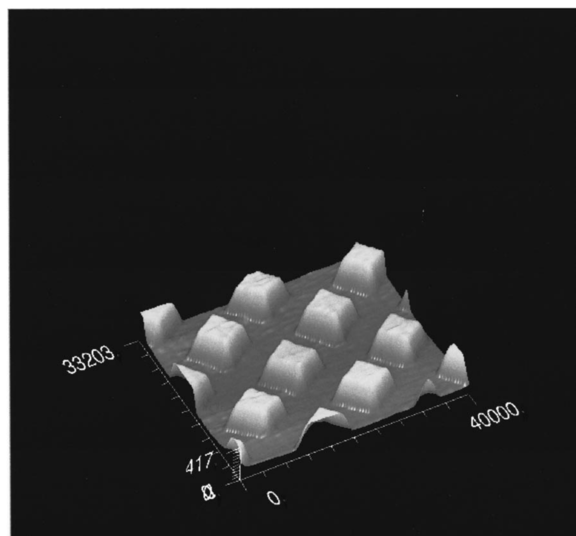

FIG. 1. 40 000 nm×33 203 nm AFM image of pattern-grown Al films on a Si substrate.

normal to their side faces. The AFM measurements were carried out in noncontact mode, using high aspect ratio silicon tips, yielding a lateral displacement resolution of better than 15 nm with a 20 μm scan range.

Figure 1 shows a typical three-dimensional image of the square Al films on a Si substrate on a 40 000 nm ×33 203 nm scanning scale. Each film with a size of ~6 μm×6 μm is displayed on a Si (100) surface with a regular distribution. Small scale scanning revealed that the surface roughness of the Si substrate is below 1 nm, suggesting that the interfaces between Al films and Si substrates is very smooth. Following thermal cycling, no evidence of interfacial debonding between the films and Si substrates or cracks at the film edges was observed. Typical cross-sectional profiles of the films before and after thermal cycling are shown in Fig. 2, where the Si–Al interface is at the bottom. The edges of the square islands of Al film were found to vary within a standard deviation (2σ) of ± 0.35 μm from the mean of 6 μm. Generally, after thermal cycling, the film width close to the interface becomes larger and the slope of the film edges becomes shallower, indicating that (i) there is a gradient of plastic deformation along the through-thickness direction of the film, and (ii) more plastic deformation occurs close to the interface. Since there is statistical variation in the size of the film islands, we randomly measured the widths of about 70 square films and plotted the size distribution of the samples before and after thermal cycling in Figs. 3(a) and 3(b). All the values were measured at a height of 20 nm from the Si–Al interface. Statistical analysis shows that the size distribution histograms follow a Gaussian distribution. By fitting the histograms, it can be observed that the mean of the normal distribution (solid lines in Fig. 3) moves to a higher value after thermal cycling. The fitted parameters show that the mean width of the thermally cycled sample at a distance of 20 nm from the interface is about 6.20 μm, which is larger than that of the samples without thermal cycling, 5.99 μm. Thus, it is believed that the difference, ~0.2 μm, comes from plastic deformation of the film, induced by thermal cycling. The average lateral strain of the square films is ~3.3%. After subtracting the elastic strain induced by residual tensile stresses, the plastic deformation of the films is about 3%.

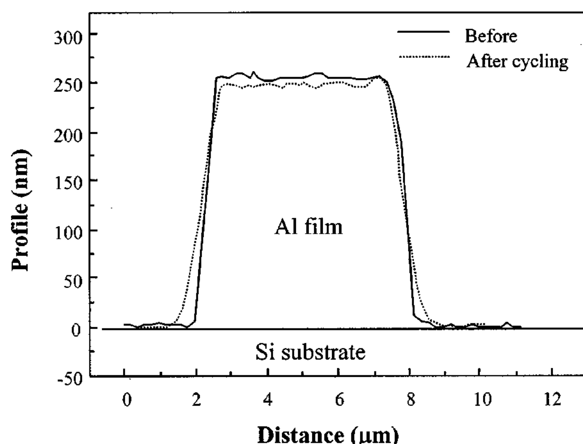

FIG. 2. Representative cross-sectional profiles of the square Al films before and after five thermal cycles from 293 to 623 K.

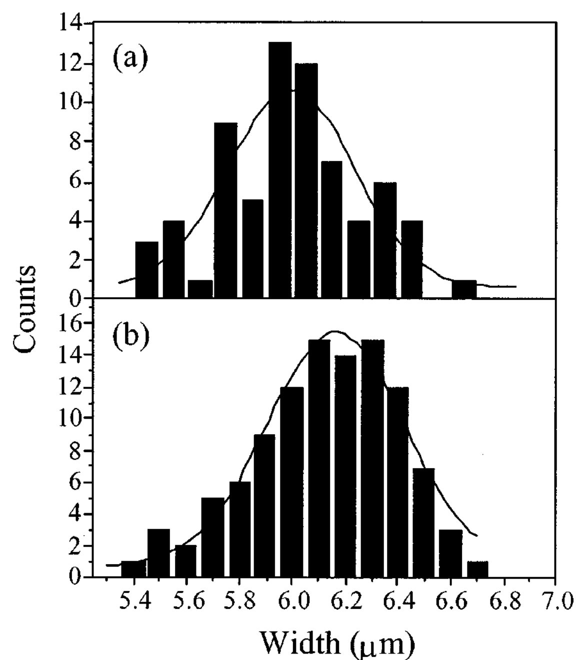

FIG. 3. Histograms and the associated Gaussian fits of the width distribution of the square Al films at a distance of 20 nm from the interface; (a) before thermal cycling; (b) after thermal cycling.

Because of the paucity of lattice dislocations and the difficulty of dislocation glide in thin films,[1] plastic deformation via dislocation glide at low temperatures is likely to be limited during thermal cycling. The dominant mechanism of plastic deformation of the thin film is therefore believed to be creep/stress relaxation. Indeed, Cu films on Si have been noted to creep at temperatures as low as 333 K during thermal cycling,[3] whereas Al films on Al_2O_3 have been thought to creep at even lower temperatures.[8] Since the film is constrained by the adjoining Si substrate which does not undergo plastic deformation, permanent relative change between the dimensions of the Al film and the Si substrate at the interface would not be possible, were it not for interfacial sliding. It is therefore believed that near the edges of the film, creep relaxation of the film is accommodated by interfacial sliding due to the presence of interfacial shear stresses, thereby allowing the film dimensions to change. The perma-

Downloaded 01 Sep 2011 to 130.34.134.250. Redistribution subject to AIP license or copyright; see http://apl.aip.org/about/rights_and_permissions

nent strain measured in the present work is $\sim 3\%$, which is much larger than the differential thermal expansion of the Al film relative to Si over the entire test temperature range, suggesting that the observed plastic strain accrues cumulatively during thermal cycling due to continuous stress and temperature revision.

The shape change of films wherein the slope of the side faces become shallower after thermal cycling indicates that plastic deformation of the square films is nonuniform. The film is strained to a greater extent close to the Al/Si interfaces. This can be attributed to two factors. First, the film has a through-thickness gradient of the in-plane normal stresses, with the stresses being largest at the interface, thereby allowing maximum creep/plasticity near the interface. Second, near the interface, film plasticity may be accommodated by interfacial creep, allowing the film dimensions to alter more.

Finite element modeling of unpassivated Al interconnect lines on Si annealed at 644 K and subsequently cooled to the ambient has shown that at ambient temperature, the films are subjected to large biaxial in-plane tensile stresses close to the interface and through most of the film thickness, but the stresses decrease to very small compressive values at the free surface.[9] The stress gradient is confined only to regions close to the free surface for very thin films (<100 nm), and becomes more pronounced with increasing film thickness, consistent with previous reports of gradients of in-plane stresses along the thickness direction.[10] Also, it was noted that with decreasing film aspect ratios (ratio of linewidth to line thickness), an increasingly larger proportion of the interface is subjected to shear stresses. Furthermore, at ambient temperature, large peeling stresses (normal tensile stresses) act on the interface near the film edges, the extent of the film subjected to such peeling stresses increasing with decreasing film aspect ratio. Indeed, for a film thickness of 0.1 μm and a linewidth of 1 μm, interfacial shear stresses prevail over the entire film width, and tensile peeling stresses exist over a distance of about 20% of the film width from each film edge.[9]

It is this combination of interfacial shear stress and the normal peeling stress near the edges of the film that is thought to drive interfacial sliding in order to accommodate creep deformation of the film. Funn and Dutta,[5,6] conducted experiments on model interfaces sliding under shear creep conditions, and modified the classical model for grain boundary sliding[11] to describe the interfacial sliding rate $\dot{\gamma}_i$ as[6]

$$\dot{\gamma} \approx \frac{4\delta_i D_i \Omega}{kTh^3}\left[\tau_i + 2\pi^3\left(\frac{h}{\lambda}\right)^3 \sigma_n\right], \quad (1)$$

where τ_i is the shear stress acting on the interface, D_i is the interface diffusivity, Ω is the atomic volume of the diffusing species (film), δ_i is the thickness of the interface, λ and h are the morphological periodicity and width (i.e., twice the amplitude), respectively, of the interface, and σ_n is the normal (peeling) stress acting on the interface. Clearly, a tensile (i.e., positive) σ_n would add to the shear stress acting at the interface, and hence enhance the sliding rate, allowing the film to undergo more lateral expansion via creep/plasticity effects. It is further noted that $\dot{\gamma}_i$ increases with decreasing interfacial (i.e., substrate surface) roughness (i.e., smaller values of h). Since the surface roughness of our substrate is less than 1 nm (as indicated by the AFM scans), the interfacial sliding rate in the present samples is likely to be large, accounting for the significant size change noted experimentally.

It should be noted that the in-plane normal stress in the film has a large tensile value at ambient temperature, is relieved with increasing temperature, and reaches a small compressive value beyond that.[3,4,8,9] The creep/plastic strain in the film assumes the same sign as the stress at any given temperature. Clearly, the expansion of the film dimensions noted in the AFM measurements suggest that the overall tensile strain induced during a complete cycle is greater than the compressive strain. Since tensile stresses exist at the lower temperatures, this indicates that the majority of the observed creep/plasticity effects occur at the lower temperatures, because of the larger film stresses in this regime. In a biaxial stress state, creep along lateral directions may be accomplished by lattice diffusion (N–H creep) and/or grain boundary diffusion (Coble creep).[11–14] Since most of the creep/plasticity occurs at lower temperatures, Coble creep is likely to be the dominant deformation mechanism in the present experiments, consistent with other observations of thin films during thermal cycling.[7]

In summary, thin Al films deposited on Si were found to undergo plastic deformation via creep during thermal cycling, the net strain during a complete cycle being tensile. The associated elongation of the film was accommodated at the interface by diffusion-driven interfacial sliding. Because of the gradient of in-plane film stresses in the through–thickness direction, the film elongated closer to the interface, resulting in not only a size change, but also a shape change. Interfacial sliding, which is driven by interfacial shear stresses, and is assisted by interfacial normal or peeling stresses near the film edges, is believed to play a key role in allowing the size change to occur, and is thought to assume greater importance the smaller the lateral film dimensions. Further experimental and modeling efforts are envisaged to quantitatively delineate the role of interfacial sliding and film creep on the dimensional stability of small thin-film features found in microelectronic applications.

This work was supported by the National Science Foundation, Division of Materials Research, under Grant No. DMR-0075281 with Dr. B. A. MacDonald as program monitor. Partial support for this work was also obtained from NSF Contract No. DMR-9423668. M. W. C. also acknowledges the support of the National Research Council Postdoctoral Associateship Program.

[1] W. D. Nix, Metall. Trans. A **20A**, 2217 (1989).
[2] F. R. Brotzen, Int. Mater. Rev. **39**, 25 (1994).
[3] M. D. Thouless, J. Gupta, and J. M. E. Harper, J. Mater. Res. **8**, 1845 (1993).
[4] Y.-L. Shen and S. Suresh, J. Mater. Res. **10**, 1200 (1995).
[5] I. Dutta, Acta Mater. **48**, 1055 (2000).
[6] J. V. Funn and I. Dutta, Acta Mater. **47**, 149 (1999).
[7] D. V. Zhmurkin, T. S. Gross, and L. P. Buchwalter, J. Electron. Mater. **26**, 791 (1997).
[8] Y.-L. Shen and S. Suresh, Acta Mater. **44**, 1337 (1996).
[9] I. Dutta (unpublished).
[10] M. F. Doerner and S. Brennan, J. Appl. Phys. **63**, 126 (1988).
[11] R. Raj and M. F. Ashby, Metall. Trans. **2**, 1113 (1971).
[12] H. J. Frost and M. F. Ashby, *Deformation Mechanism Maps* (Pergamon, Oxford, 1982).
[13] C. Herring, J. Appl. Phys. **21**, 437 (1950).
[14] R. L. Coble, J. Appl. Phys. **34**, 1679 (1963).

Downloaded 01 Sep 2011 to 130.34.134.250. Redistribution subject to AIP license or copyright; see http://apl.aip.org/about/rights_and_permissions