Temperature-dependent inelastic response of passivated copper films: Experiments, analyses, and implications

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The temperature-dependent mechanical behavior of passivated copper films is studied. Stresses in copper films of thickness ranging from 40 to 1000 nm, deposited on quartz or silicon substrates and passivated with silicon oxide were measured by using the substrate curvature method. The thermal cycling spans a temperature range from -196 to 600 °C. It was observed that the passivated films do not exhibit a significant stress relaxation at high temperatures that is typically found in unpassivated films. The measured mechanical behavior was found to be rate insensitive within the heating/cooling rate range of 5 – 25 °C/min. Furthermore, a significant strain hardening during the course of thermal cycling was noted. Analyses employing continuum plasticity show that the experimentally measured stress–temperature response can only be rationalized with a kinematic hardening model. Analytical procedures for extracting the constitutive properties of the films that were developed on the basis of such model are presented. To emphasize the importance of the appropriate choice of constitutive model, results of finite element modeling for predicting thermal stresses in copper interconnects are presented. The modeling assumed parallel copper lines embedded within the combined low k /oxide dielectric materials. It was found that ignoring plastic strain hardening of copper can lead to significant errors in the stress and strain developed in the interconnect.

I. INTRODUCTION

The progressive replacement of copper (Cu) in place of aluminum (Al) for interconnect metallization in integrated circuits has necessitated the need for understanding the thermomechanical behavior of thin Cu films. Considerable progress has been made in recent years.^{1–12} Passivated Cu films behave significantly differently from their unpassivated counterparts. Thermal mismatch induced stresses in passivated Cu films do not relax significantly at elevated temperatures during thermal cycling.^{1,2,5,8} This has been attributed to the prevention of surface atomic diffusion from relaxing the film stress and to the constrained dislocation motion due to the passivation. (In the case of Al, even the unpassivated film is naturally coated with a thin native oxide layer.) The overall stress–temperature response of Cu films, from which the constitutive behavior of the film can be extracted is, therefore, highly influenced by the passivation. As a result, great care has to be exercised in selecting the constitutive model for simulating the thermomechanical behavior of the Cu films.

The objective of the present study is to develop a detailed understanding of the mechanical behavior of passivated Cu films, in particular, the temperature-dependent elastic–plastic response within the continuum framework. For this purpose, temperature excursion experiments on Cu films of varying thickness, sandwiched between a substrate and a thin passivation layer, were conducted and analyzed. Special attention

is given to the plastic hardening characteristics during cyclic loading. The constitutive response extracted from the film experiment is used for numerical modeling of thermal stresses in a model interconnect structure with the objective of illustrating the importance of choosing appropriate material models in predicting the thermal stresses in Cu interconnects.

II. EXPERIMENT

The experiments were performed with electron beam deposited films on quartz substrates. The Cu films with thicknesses of 400, 250, and 40 nm were passivated with a silicon oxide layer whose thickness is, typically, 20% of that of the Cu film. A 15-nm-thick chromium interlayer is applied both between the Cu film and the substrate and between the Cu film and the passivation layer to serve as diffusion barrier. After thermally cycled to a stabilized condition, the Cu films showed columnar grains with an average grain size approximately equal to the film thickness. Many annealing twins and dislocations were also existent, as were shown in other studies of such films.^{7,12} In addition to the above specimens, 1000-nm-thick Cu films were also studied. The films were sputter deposited on oxidized silicon wafers and passivated with 50 nm silicon oxide. In this case, the interlayer is 50-nm-thick tantalum.

The curvature measurement technique that is routinely employed for measuring the thermomechanical response of thin films^{13,14} was employed for measuring stresses in the Cu film during thermal cycles between ambient temperature

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FIG. 1. Variation of stress in the passivated 400 nm Cu film during thermal cycling.

(nominally, 20 °C) and 600 °C. Unless otherwise stated, the nominal heating/cooling rate was maintained at 10 °C/min. During the cooling phase of the thermal cycle and below ~150 °C, the prescribed cooling rate was maintained through the utilization of liquid nitrogen in the temperature control system. In selected cases, the temperature range was extended to subambient temperatures by interrupting the curvature measurement and slowly immersing the specimen in liquid nitrogen and then reheating to ambient followed by the resumption of subsequent measurements under controlled thermal cycles.

III. RESULTS OF STRESS MEASUREMENT

Figure 1 shows the variation of biaxial stress in the 400 nm Cu film as a function of temperature between 20 and 450 °C. The stress in the film, initially of tensile nature at ambient temperature, tends to be more compressive upon heating because of the constraint to thermal expansion of Cu imposed by the substrate that has a lower coefficient of thermal expansion *vis a vis* Cu. During initial heating, however, a reduction in the magnitude of stress is seen from 150 to 250 °C. This is due to the evolving microstructure, typified by grain growth.^{1,5} A stabilized response ensues upon subsequent cooling. Throughout the rest of this paper, only the stabilized thermomechanical response is discussed.

The initial slopes observed during heating and cooling define the elastic response of the Cu film. The deviation from the initial slope indicates plastic yielding. It is noted that, at elevated temperatures, the passivated Cu film can support reasonably large stresses. This is in sharp contrast with unpassivated Cu films which exhibit a significant stress relaxation at temperatures above ~250 °C.^{1–5,8} At room temperature, the passivated film can support a tensile stress close to 700 MPa (Fig. 1).

An important feature observed in Fig. 1 is that, in a stabilized loop, yielding can commence at a “negative” stress. This is manifested by the heating curve that shows “com-

FIG. 2. Stress change as a function of time during isothermal hold. The beginning state was attained during cooling from 450 °C.

pressive” yielding at a tensile stress of about 175 MPa. The early yielding upon load reversal is generally referred to as Bauschinger effect in bulk metals. Qualitatively similar behavior has also been shown for passivated Cu films in other studies.^{1,2,5,8} Implications of this experimental feature in constructing the constitutive model will be discussed in Sec. IV.

In order to examine whether the highly asymmetric stress range about the zero axis (or equivalently, the very strong Bauschinger-like effect) observed in the stabilized loop in Fig. 1 was indeed the film’s response and not an artifact of the methodology employed for measurement, passivated 400 nm Cu films were thermally cycled between 20 and 450 °C to achieve stabilization and isothermally held at various temperatures during the cooling phase. The results are shown in Fig. 2 in the form of change in stress as a function of time. At 300 to 375 °C, significant stress relaxation can be seen over a period of 4 h. However, at 400 °C there is essentially no change in stress with time. This means that the stress in the Cu film is very close to zero at 400 °C, which is consistent with the cooling response shown in Fig. 1. As temperature decreases, stronger isothermal relaxation occurs (Fig. 2) owing to the increasing initial tensile stress (Fig. 1).

Figure 3 shows a comparison of stabilized stress–temperature response for passivated Cu films with thicknesses 400, 250, and 40 nm. In all cases the Bauschinger-like effect is evident. It can be seen that the hysteresis becomes slightly smaller with decreasing film thickness, implying increasing difficulty of plastic deformation due to the dimensional constraint. At room temperature, a thinner film can support a higher tensile stress.

The 1000 nm Cu films were subject to thermal cycles up to 600 °C. A representative stress–temperature response is shown in Fig. 4. It is clear that there is no sign of stress relaxation for passivated Cu films even at 600 °C during continuous thermal excursions. An intermediate cycle is included in Fig. 4(a) for a temperature range of 20 to 300 °C and in Fig. 4(b) for a range of 400 to 600 °C. In contrast with the case of Al films where intermediate cycling near the

FIG. 3. Stabilized stress-temperature response for passivated Cu films of various thicknesses.

maximum temperature results in temporary cyclic hardening,¹⁵ the variation of thermal history shows essentially no effect on the overall stabilized stress-temperature response of passivated Cu films. In addition, the Bauschinger-like effect exists for all the loops in Fig. 4.

To further extend the temperature range considered above, a slow cooling to liquid nitrogen was conducted on the 400 nm film after cycled to stabilization between 20 and 450 °C. The result is shown in Fig. 5. Although the curvature change at below room temperature was not monitored, linear extrapolation of the cooling to and reheating from -196 °C leads to a smooth connection of the measurable response, as evidenced by the model fit shown in Fig. 5 (to be discussed in the following section). Subsequent cycling between 20 and 450 °C shows a loop similar to those exhibited prior to the cryogenic excursion, suggesting that there is minimal effect of thermal history on the hysteresis. It is noticed from Fig. 5 that, upon reheating from liquid nitrogen, plastic yielding commences while the overall stress is still tensile.

In addition to the aforementioned experiments, the effect of heating/cooling rate on the evolution of stress was also investigated by varying the rate from 5 to 25 °C/min. It is observed that the stress-temperature response is unaltered within the temperature range between 20 and 600 °C. Therefore, within the range of the heating/cooling rate considered here, the rate-dependent effect is insignificant and therefore may be ignored for practical purposes. This is similar to the case of aluminum films.¹⁵

IV. CONSTRUCTION OF CONSTITUTIVE MODEL

All the stress-temperature loops presented in Figs. 1, 3, 4, and 5, obtained from passivated Cu films with various thicknesses and under different temperature ranges, revealed the same qualitative features: rate-independent elastoplasticity with cyclic stabilization and Bauschinger-like phenomenon. Such a behavior cannot be represented, within the framework of continuum mechanics, by the elastic-perfectly plastic

FIG. 4. Stress-temperature response of the 1000 nm Cu film within the range of 20–600 °C. Different intermediate cycles are also shown in (a) and (b).

model with temperature-dependent yield strength such as in the case of Al films, because it will be impossible to endow the Cu film with a compressive yield strength that matches the heating response of the film at elevated temperatures. Necessarily, strain hardening has to be involved in the model for the films investigated in this work. However, typical *isotropic hardening*, with the yield surface in stress space expanding uniformly with no shape change or translation during deformation,^{16,17} is incompatible with the Bauschinger effect upon load reversal and predicts an eventual elastic shakedown without a hysteresis loop. Therefore, the isotropic hardening model cannot be applied to the Cu film. On the other hand, the classical *kinematic hardening* model, which assumes that the yield surface does not change its size and shape but simply translates in stress space in the direction of the outward normal,^{16,17} can be used to describe the stress evolution in Cu films. This is evident because the kinematic hardening model does indeed predict the existence of

FIG. 5. Stress-temperature response of the 400 nm Cu film, with the thermal cycling conducted extending to -196°C . Experimental data are fitted with the kinematic hardening model discussed in Sec. IV.

Bauschinger effect, and a steady state of alternating plastic deformation sets in after the first loading cycle, in full agreement with the experimental measurements reported in this article.

In the following, construction of a constitutive law of the passivated Cu film is sought by invoking the kinematic hardening model with linear hardening. The temperature dependence of yield strength is taken into consideration. In particular, a linear dependence of initial yield strength with temperature is employed,

$$\sigma_y = \sigma_0 \left(1 - \frac{T}{T_0} \right), \quad (1)$$

where σ_y is the initial yield strength, T is temperature, and σ_0 and T_0 are reference constants. Note that within the context of this formulation, the size of the yield surface will be a linear function of temperature but the essence of kinematic hardening, featuring a translating yield surface, is nevertheless preserved. For illustration purposes attention is devoted to the 400 nm film. The stabilized loop between 20 and 450°C is highlighted with four points in Fig. 5: A and C are the reversal points and B and D approximately define the transitions from elastic to plastic deformations. Since segments AB and CD represent the elastic behavior (the stress state traverses diagonally inside the yield surface), the two unknowns in Eq. (1), σ_0 and T_0 , can then be determined by the following two equations:

$$[\sigma_y]_A + [\sigma_y]_B = |[\sigma]_B - [\sigma]_A|, \quad (2)$$

$$[\sigma_y]_C + [\sigma_y]_D = |[\sigma]_D - [\sigma]_C|, \quad (3)$$

where $[\sigma_y]$ denotes the magnitude of yield strength at the specified temperature and $[\sigma]$ represents the stress value at the specified point obtained from the plot (Fig. 5). The hardening rate (or tangent modulus, defined to be the slope of the uniaxial stress-strain curve beyond yielding), then follows:

$$H_{D \rightarrow A} = \frac{|[\sigma]_A - [\sigma]_D| - \{[\sigma_y]_A - [\sigma_y]_D\}}{\Delta\alpha\Delta T}, \quad (4)$$

$$H_{B \rightarrow C} = \frac{|[\sigma]_C - [\sigma]_B| - \{[\sigma_y]_B - [\sigma_y]_C\}}{\Delta\alpha\Delta T}, \quad (5)$$

where $\Delta\alpha$ is the difference in coefficient of thermal expansion between Cu and the substrate, and ΔT is the temperature change involved. The solid curves shown in Fig. 5 are a result of the model with $\sigma_0 = 305\text{ MPa}$, $T_0 = 1090\text{ K}$, and $H = 77\text{ GPa}$. It satisfactorily fits the measured loop ABCD. With simple extrapolation to lower temperature, it can be seen that the model also matches the cryogenic thermal cycle remarkably well. (For reference, the thermoelastic properties of Cu at room temperature are: Young's modulus 110 GPa, Poisson's ratio 0.3, and $\Delta\alpha = 16.5 \times 10^{-6}\text{ K}^{-1}$.)

The same methodology can be used to obtain the constitutive response of passivated Cu films with other thicknesses. In the above analysis, it is coincidental that the value H is 77 GPa during both heating (B to C) and cooling (D to A). The constitutive law does not require the two hardening rates to be the same. Nevertheless, this hardening rate appears to be very high (greater than one half the magnitude of Young's modulus of Cu). The strong hardening may be partially attributed to the small grains containing twin boundaries that can interact with dislocations and serve as additional barriers to slip.¹⁸ The concept of "back stress" normally associated with the kinematic hardening model may also be reasoned as such. More characterizations are necessary to establish a firm structure-property relation in such films. To this end, it is worth mentioning that the very strong hardening is not at much variance with the tensile stress-strain response of Cu films measured mechanically by Hommel *et al.*¹⁹ Within the tensile strain range of about 0.005 (approximately the same order as the thermally induced strain of interest here), the measured tangent modulus was reported to be only moderately smaller than the apparent Young's modulus.

V. MODELING STRESSES IN Cu INTERCONNECTS

One important purpose of developing the constitutive law based on experiments is to utilize it to numerically predict the thermal stress generated in Cu interconnect lines. To a first approximation the encapsulated line can be assumed to have the same properties as the passivated film. In this section, finite element analyses of an interconnect system, featuring Cu lines and a low dielectric constant material, are presented to illustrate the importance of choosing appropriate constitutive behavior in the modeling.

For the above-mentioned purpose, a series of parallel Cu lines, embedded within the dielectric on top of the silicon (Si) substrate, are considered. The metal lines are perpendicular to the paper. Because of the periodicity and symmetry in the lateral (across-the-line) direction, only one half of a unit segment is required in the calculation, as shown in Fig. 6. In the model w and h were both taken to be 400 nm, and a full Si substrate thickness of $500\text{ }\mu\text{m}$ was included. (The exact substrate thickness will not affect the modeling result if

FIG. 6. Schematic of the interconnect structure used in the numerical modeling.

it is greater than about $100 \mu\text{m}$ in the present case.) For simplicity the thin diffusion barrier layers between Cu and the surrounding material were not included. The dielectric arrangement follows an embedded scheme^{20,21} such that in between the metal lines a polymer based low-dielectric constant (low- k) material is used and conventional silicon oxide (SiO_2) exists directly above and below the metal/low- k dielectric layer. The width of the Cu line and low- k dielectric is taken to be the same.

In all calculations, attention was confined to deformations that preserve the mirror symmetry of the array. At the reference point ($y=0, z=0$), the displacements along the y and z directions are taken to be zero. Along the symmetry axis, $y=0$, the displacement in the y direction vanishes. The other symmetry axis, i.e., the right boundary, is free to move but is forced to remain straight. All other boundaries are not constrained during deformation. The finite element program ABAQUS (Ref. 22) was used for the calculation. The discretizations used linear quadrilateral elements. The conver-

gence of results was checked through various degrees of mesh refinement. The result presented in this paper is from calculations using a total of 1440 elements. A generalized plane strain formulation was used. This is an extension of the plane strain framework (with the yz plane being the plane of deformation), achieved by superimposing a longitudinal normal strain along the line (x) direction on the plane strain state. The generalized plane strain model is capable of yielding more realistic field quantities than the strict plane strain formulation.²³ Such type of model has been used to calculate thermal stresses in single- and multilevel Al interconnects^{24–27} and compared favorably with the full three-dimensional analysis.^{28,29}

The evolution of thermal stresses was modeled by imposing a spatially uniform temperature change from the initial stress-free temperature of 350°C (presumably, the film annealing temperature) to 20°C . The SiO_2 , Si, and low- k dielectric phases were taken to be isotropic linear elastic solids. The material properties, extracted from Refs. 21, 23, and 24, used in the analysis are listed in Table I. The properties of the low- k dielectric followed those of divinyl-siloxane-bis-benzocyclobutene reported in Ref. 21. Two constitutive behaviors for Cu are considered: the presently obtained elastic-plastic response with kinematic hardening (described in Sec. IV) and an elastic-perfectly plastic model, which fits the cooling portion of the loop in Fig. 5. Although Cu crystals are mechanically anisotropic, the direction dependence and texture effects were ignored because the present modeling serves as a straightforward illustration of comparisons of two simple constitutive behaviors. The thermoelastic properties of Cu are listed in Table I. The plastic response of the kinematic hardening model is given by Eqs. (1), (4), and (5) with the parameters $\sigma_0=305 \text{ MPa}$, $T_0=1090 \text{ K}$, and $H=77 \text{ GPa}$ (see the discussion in Sec. IV). The temperature-dependent yield strength for the perfectly plastic model, in accordance with the stress-temperature response during cooling (Fig. 5), is given in Table I. It is noted that the latter model is typically adopted for modeling stresses in Al interconnects^{24–32} and on occasion for Cu;³³ we show below

TABLE I. Material properties used in the numerical analysis. E : Young's modulus, ν : Poisson's ratio, α : coefficient of thermal expansion (CTE), and σ_y : yield strength. Unless otherwise stated, a linear variation of properties with temperature between the indicated temperatures is assumed.

		Cu	SiO_2	Si	Low- k dielectric
E (GPa) at	20°C	110	71.4	130.0	2.5
	350°C	104	71.4	130.0	0.3 ^a
ν at	20°C	0.3	0.16	0.28	0.34
	350°C	0.3	0.16	0.28	0.34
α ($10^{-6}/\text{K}$) at	20°C	17.0	0.52	3.1	63.6
	350°C	19.3	0.68	4.4	63.6
σ_y (MPa) ^b at	20°C	676	...	...	...
	350°C	165	...	...	...

^aLinear variation only between 20 and 180°C (see Ref. 21); a constant value (0.3 GPa) between 180 and 350°C .

^bFor the elastic-perfectly plastic model only. The kinematic hardening model is described by Eqs. (1), (4), and (5) with $\sigma_0=305 \text{ MPa}$, $T_0=1090 \text{ K}$, and $H=77 \text{ GPa}$ (see Sec. IV for details).

FIG. 7. Calculated variation of hydrostatic stress in the Cu line with temperature using the kinematic hardening model and the perfectly plastic model.

that this choice can lead to a large error in the case of Cu.

Figure 7 shows the evolution of hydrostatic stress, defined as $\frac{1}{3}(\sigma_{xx} + \sigma_{yy} + \sigma_{zz})$, as a function of temperature during cooling from 350 to 20 °C. The stress magnitude is averaged over the cross section of the interconnect. Both the kinematic hardening model and the perfectly plastic model are included in Fig. 7. It is seen that a significant difference develops when the temperature is below about 200 °C. The fact that the perfectly plastic model predicts a tensile stress higher than that of the kinematic hardening model is because a much steeper increase in yield strength with temperature in the former has to be imposed to match the measured cooling curve in Fig. 5. As a consequence, if one accepts the result on the basis of the experimentally determined kinematic hardening response for the Cu film, the perfectly plastic model shows an error in stress as large as 40% upon cooling to room temperature. This illustrates the importance of applying the appropriate constitutive response in modeling interconnect stresses.

Figure 8 shows the evolution of averaged equivalent plastic strain in the Cu line during cooling from 350 to 20 °C. Although the continuum approach is used in the present case of submicron material dimension, salient insights of the deformation process can still be obtained. In the present case, the equivalent plastic strain signifies the extent of irrecoverable deformation and the associated crystal defects. The perfectly plastic model predicts essentially no plasticity in Cu except close to room temperature. The kinematic hardening model, however, shows significant plasticity developed after an initial elastic response. The accumulation of plastic strain, given by the kinematic hardening model, is in line with the “back stress” concept observed in the passivated Cu films discussed in Sec. IV. The incorporation of the lateral low- k dielectric, with a very low modulus and very high coefficient of thermal expansion compared with both Cu and SiO₂, is known to suppress hydrostatic tension and enhance plastic

FIG. 8. Calculated variation of equivalent plastic strain in the Cu line with temperature using the kinematic hardening model and the perfectly plastic model.

deformation in the embedded Cu line. This feature cannot be captured by the perfectly plastic model.

VI. CONCLUSIONS

The generation of thermal stresses in passivated Cu films with various thicknesses and thermal cycling histories was measured. The stress does not relax at elevated temperatures during the temperature cycle. The film does not show any rate-dependent effect over a range of heating/cooling rate from 5 to 25 °C/min. An elastic–plastic model with kinematic strain hardening has to be employed to fit the experimental results showing a stabilized loop and the Bauschinger-like effect. This is drastically different from the unpassivated and passivated Al films and the unpassivated Cu films. The procedures of extracting the temperature dependent properties are presented. The film shows very high strain hardening beyond the elastic limit. It was also found that, with the inclusion of intermediate cycles within the stabilized loop and cryogenic excursion, there is minimal effect of thermal history on the already established hysteresis. The importance of utilizing an appropriate constitutive response in predicting thermal stresses in Cu interconnects was illustrated with finite element modeling. Treating Cu as an elastic–perfectly plastic solid with temperature dependent yield strength results in significant errors in the stress and strain developed inside the Cu line.

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