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Internal stress and yield strength of copper films on substrates*

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Internal stress and yield strength of pure copper films on substrates were characterized by x-ray diffraction and thermal-cycle substrate curvature methods. The internal stress was of tension, and decreased with increasing working-gas (argon) pressure and increased with increasing film thickness. Tensile yield strength of copper films on steel substrate was reciprocal to the film thickness. Similarly, the compressive yield strength depended strongly on the film thickness: the thinner the film thickness, the larger the compressive yield strength.

Keywords: copper film, internal stress, yield strength, x-ray diffraction, thermal-cycle substrate curvature

PACC: 6110, 6114, 7340, 7360D

1. Introduction

Interest in the mechanical properties of adherent film has been accelerating with a need for the reliability of micro-structures and micro-devices made of films.[1,2] Failures of aluminium interconnects in integrated circuits, such as open circuits from cracking or voiding,[3,4] short circuits due to hillocks,[5,6] and both open and short circuits due to stress- and/or electro-migration, are believed to be associated mainly with the poor mechanical properties of the film. Failure prevention has been a continuing problem with aluminium-based metallizations for many years. It has been claimed in recent years that copper-based metallizations are prospective alternatives to aluminium-based alloys as multilevel interconnects in ultra-large-scale integration (ULSI) circuits.[7,8] The great advantage of copper interconnects lies in the desirable properties in terms of low resistivity and high melting temperature. There are, however, several issues that need to be addressed before copper can eventually replace aluminium as the metallization material of choice. Mechanical behaviour of the film has been identified as one of the key issues.[7]

Internal stress in thin film is inevitable due to differential thermal expansion between film and substrate and microstructure change.[7−11] Internal stress may build up and lead to a fairly large value that exceeds the yield strength of the film and results in plastic deformation as well as creep in thin film.[12] So, both internal stress and yield strength of copper film need to be investigated. In this paper, x-ray diffraction (XRD) is used to study the preliminary characteristics of internal stress and yield strength of the deposited film. On account of the fact that temperature change will be involved in both film fabrication and service, variations of internal stress and yield strength in the film with temperature are further explored by a thermal-cycle substrate curvature method.[13−15]

2. Experiments

2.1. Sample preparation

Pure copper films were deposited onto (100)-oriented silicon wafers by the magnetron sputtering method. A group of pure copper films of various thicknesses were coated on stainless steel in order to measure their yield strength by bending during x-ray diffraction analysis. The silicon wafer is too brittle to

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be used for the bending test.

Parameters used for copper film deposition are listed in Table 1. The deposited film thickness is measured by scanning electron microscope (SEM) in cross section mode. To investigate the effect of the deposition process on internal stress, some pure copper films are deposited on silicon substrates at different argon gas pressures.

Table 1. Sample deposition parameters.

Base vacuum	Sputtering-gas (Ar) pressure	Target-substrate distance	Target voltage	Target current
2.66×10^{-3}Pa	2.66×10^{-1}Pa	8cm	−520V	480mA

2.2. Yield strength measurement by using x-ray diffraction

Internal stress measurement by using x-ray diffraction has been well established in theory of elasticity, but the measurement of surface yield strength has not been widely understood. However, the stress σ_φ in the plane of sample surface can be deduced from the strain of the lattice spacing $\varepsilon_{\varphi\psi}$ based on the following equation:

$$\varepsilon_{\varphi\psi} = \frac{S_2}{2}\sigma_\varphi \sin^2\psi + S_1\left(\sigma_{11}+\sigma_{22}\right), \qquad (1)$$

in which

$$\sigma_\varphi = \sigma_{11}\sin^2\varphi + \sigma_{12}\sin 2\varphi + \sigma_{22}\cos^2\varphi,$$

where ψ is a tilt angle of the sample, S_1 and S_2 are termed x-ray elastic constants, the values of which depend on the diffracted (hkl) planes and can be calibrated experimentally. The subscript φ represents an angle between the direction of measured stress σ_φ and that of the stress σ_{11}. The internal stress can be obtained by a well-known $\sin^2\psi$-law

$$\frac{\partial\varepsilon_{\varphi\psi}}{\partial\sin^2\psi} = \frac{S_2}{2}\sigma_\varphi, \qquad (2)$$

in which, the strain $\varepsilon_{\varphi\psi}$ is measured in terms of displacement of diffraction profile based on Bragg's law.

If an external stress $\sigma_{\rm appl}$ is applied on the sample along the direction of σ_φ, the x-ray-measured stress σ_φ will be substituted by $\sigma_{\rm appl}+\sigma_\varphi$. Since elastic stress can only be detected by x-ray diffraction via the changes of the interplanar spacing, the linear relation is kept before the material deforms plastically. The transition from elastic to plastic strain can thus be employed to determine the yield strength of the tested material.

2.3. Stress measurement with substrate curvature

A strain will be introduced when the temperature of a film on a substrate is changed. This variation of thermal expansion is

$$\varepsilon_f = \left(\alpha_f - \alpha_s\right)\mathrm{d}T, \qquad (3)$$

where α_f and α_s are the linear-expansion coefficients of the film and substrate respectively. The integration can be made using polynomial expression of α obtained from Ref.[16]. Nevertheless, Eq.(3) can be approximated over a limited temperature range as

$$\varepsilon_f = \left(\alpha_f - \alpha_s\right)\Delta T.$$

The thermal strain (or stress) will cause bending of the coated sample if the sample is thin enough. The stress can be calculated from the substrate curvature

$$\sigma_f = \frac{E_s t_s^2}{6\left(1-\upsilon_s\right)t_f}\left(\frac{1}{R}-\frac{1}{R_0}\right), \qquad (4)$$

where σ_f is the stress in the thin film, E_s and υ_s are the Young's modulus and Poisson's ratio of the substrate, t_s and t_f are the substrate and film thicknesses, respectively; R_0 is the initial radius of curvature before film deposition, and R is the measured radius of curvature.

3. Results and discussion

Figure 1 shows the variation of internal stress as a function of sputtering-gas (argon) pressure. The internal stress is of tension and decreases with increasing argon-gas pressure. The effect of argon-gas pressure has been modelled by an atomic peening mechanism.[17] The higher the argon-gas pressure, the more the argon atoms contained in the film and the larger the compressive stress component induced by additional volume in the film. The other two components are intrinsic stress which is originated from the growth process as a result of in-grown defects or structural mismatch between the film and the substrate, and the thermal stress which originates from the difference in the thermal expansion coefficients of the film and the substrate. For a copper film on silicon substrate, since the thermal expansion coefficient of copper is higher than that of silicon, the tensile

stress is produced by cooling the sample from deposition temperature to room temperature. Thus the resultant internal stresses appear to be controlled by the parameters as shown in Fig.1. It is also noted that the magnitude of the internal stress, especially for the films deposited at a low argon-gas pressure, is several times higher than the yield strength of the bulk copper, which is about 50MPa. This implies that the film can hold much higher yield strength than the bulk material.

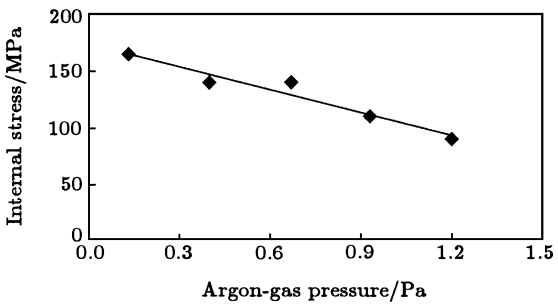


Fig.1. Variation of internal stress in copper films as a function of sputtering argon-gas pressure.

Figure 2 illustrates the influence of film thickness on the internal stress. The tendency is similar to most of the other metal films that the stress varies linearly with a positive slope with the thickness. The film thickness is one of the primary factors influencing the internal stress and yield strength of the film,[18] but its effect is associated with the substrate and passivated layer.[3]

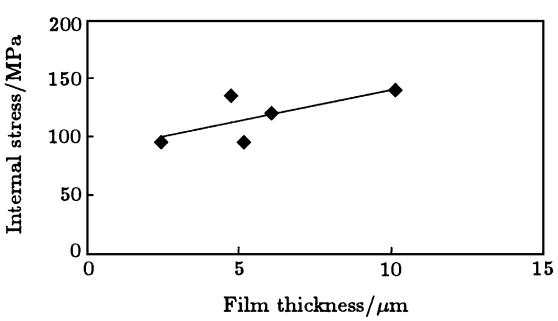


Fig.2. Influence of film thickness on the internal stress in copper films.

Figure 3 is one of the curves of x-ray measured stress as a function of applied strain in the 1.4μm thick copper film adhered to a steel substrate. Mechanical loading of the sample is carried out with a four-point bending device. The applied strain is monitored with a strain gauge on the back of the sample. As the measured stress goes beyond the elastic–plastic transition point A, it does not increase linearly with the strain but varies only slightly. The reason is that the grains are not plastically deformed simultaneously. Plastic deformation takes place firstly in the grains that are in the "weaker" orientations.[19] Obviously, the yield strength measured with this method will be somewhat smaller than the conventional one since it is not the stress corresponding to plastic deformation in all grains.

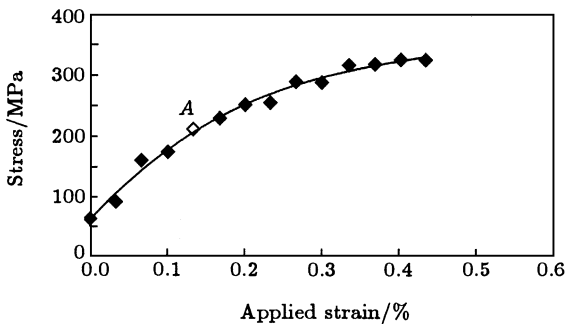


Fig.3. Relation between x-ray measured stress and applied strain of an 1.4μm thick copper film.

Figure 4 shows the influence of film thickness on the tensile yield strength of a pure copper film on steel substrate. It is noted that the yield strength of the film is higher than that of bulk material, especially when the thickness of the film is very thin. It is also noted that the tensile yield strength varies linearly with the inverse of film thickness. This is consistent with our theoretical analysis. In a previous paper, a simple expression of the yield strength of a polycrystalline film attached to a substrate with a passivated layer was derived from a work–energy relationship for dislocation movement.[20]

$$\sigma_y = \frac{\sin\varphi}{\cos\varphi\cos\lambda}\frac{b(1-\upsilon_f\cos^2\beta)}{2\pi(1-\upsilon_f)} \left[\frac{\mu_f\mu_s}{(\mu_f+\mu_s)h}\ln\left(\frac{\beta_s h}{b}\right) + \frac{\mu_f\mu_t}{(\mu_f+\mu_t)h}\ln\left(\frac{\beta_t t}{b}\right) + \frac{\mu_f}{d\sin\varphi}\ln\left(\frac{d}{b}\right)\right].$$

It is shown that the yield strength of a polycrystalline film is determined by two affecting factors (grain orientation and dislocation type, corresponding to the first and second factors respectively) and three strengthening factors (substrate strengthening, passivated layer strengthening and grain size strengthening, corresponding to the first, second and third terms in the square bracket). Both the substrate strengthening and passivated layer strengthening are shown clearly to be reciprocal to the film thickness h. An increase in the grain size d with film thickness was also observed in Al-0.5%Cu films.[18] So the yield strength

of thin films varies linearly with the inverse of film thickness as observed here.

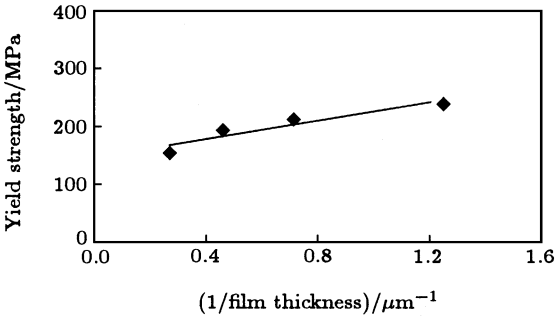


Fig.4. Variation of the yield strength of copper films on steel substrate with the inverse of film thickness.

Figures 5 and 6 are examples of measured results during first thermal cycle for pure copper film on silicon substrate with thicknesses of 3.42 and 0.24μm respectively. There is a hysterical loop in stress–temperature curve, indicating a nonlinear phenomenon involved in the process. There are three critical points, A, B and C, in the stress–temperature diagram worth noticing. During heating, the film stress changes from tension to compression with a constant rate in the beginning due to the differential thermal expansion coefficients between the film and the substrate. But it changes to a nonlinear part as it reaches point B and drops gradually as the temperature is above 200°C (point C). It has been proved that the former part is an indication of plastic deformation and the latter is associated with recrystallization of the film.[7,8,18]

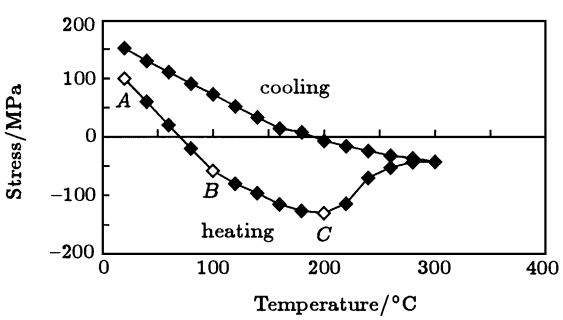


Fig.5. Stress–temperature curves of a 3.42μm thick copper film on silicon substrate.

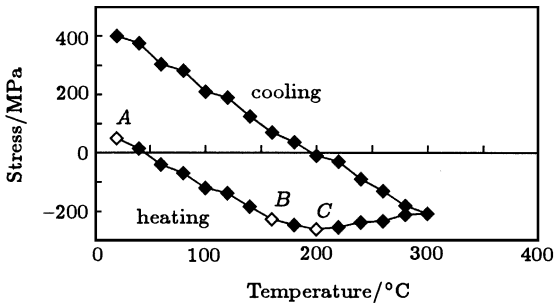


Fig.6. Stress–temperature curves of a 0.24μm thick copper film on silicon substrate.

It is noted that the internal stress at point A and the yield strength at point B are strongly dependent on the film thickness. The thinner the film thickness, the lower the internal stress (similar to Fig.2), but the higher the compressive yield strength (similar to Fig.4), and the thinner film shows much less plasticity and stress relaxation than the thick one. From Figs.5 and 6, we also noted that the stress after thermal cycling to room temperature is higher than the original internal stress, especially for thinner film.

4. Conclusions

Internal stress and yield strength in the copper film on substrate are two of the important mechanical parameters which determine the performance and reliability of ultra-large scale integrated (ULSI) circuits, the values and affecting factors for them need to be investigated further. Preliminary results show that the internal stress is of tension and decreases with increasing working-gas (argon) pressure, but increases with increasing film thickness. The tensile yield strength of the copper films on steel substrates is inversely proportion to the film thickness. So is the compressive yield strength of the copper film on silicon substrate. The effects of the other factors (including grain size, texture, alloying elements and contents) on the internal stress and yield strength of the film need to be investigated in future work.

References

[1] Sinda A K and Sheng T T 1978 *Thin Solid Films* **48** 117

[2] Faith T J and Wu C P 1984 *Appl. Phys. Lett.* **116** 470

[3] Zhang J M and Xu K W 2004 *Chin. Phys.* **13** 205

[4] Borgesen P, Lee J K, Gleixner R and Li C Y 1992 *Appl. Phys. Lett.* **60** 1706

[5] Gerth D, Katzer D and Schwarzer R 1992 *Mater. Sci. Forum* **94–96** 557

[6] Chaudhari P 1974 *J. Appl. Phys.* **45** 4339

[7] Toomey J J, Hymes S and Murarka S P 1995 *Appl. Phys. Lett.* **66** 2074
[8] Flinn P A 1991 *J. Mater. Res.* **6** 1498
[9] Zhang J M, Xu K W and Ji V 2001 *Appl. Surf. Sci.* **180** 1
[10] Tanaka K, Akiniwa Y, Inoue K and Ohta H 1998 *Proc. 5th Inter. Conf. Residual Stresses* p874
[11] Ohmi T, Saito T, Shibata T and Nitta T 1988 *Appl. Phys. Lett.* **52** 2236
[12] Zhang J M and Xu K W 2004 *Acta Phys. Sin.* **53** 2439 (in Chinese)
[13] Doerner M F, Gardner D S and Nix W D 1986 *J. Mater. Res.* **1** 845
[14] Flinn P A, Gardner S and Nix W D 1987 *IEEE Trans. Electron Devices* **ED-34** 689
[15] Hershkovitz M, Blech I A and Komem Y 1985 *Thin Solid Films* **130** 87
[16] Touloukian Y S, Kirby R K, Taylar R E and Lee T Y R 1979 *Thermal Expansion Nonmetallic Solids* Vol.13 (New York: IFI/Plenum)
[17] dHeurle F M 1970 *Metall. Trans.* **1** 725
[18] Venkatraman R, Bravman J C, Nix W D, Davies P W, Flinn P A and Fraser D B 1990 *J. Electronic Mater.* **19** 1231
[19] Zhang J M and Xu K W 1999 *J. Mater. Sci. Letter.* **18** 471
[20] Zhang J M, Xu K W and Zhang M R 2003 *Acta Phys. Sin.* **52** 1234 (in Chinese)