ENVIRONMENTAL EFFECTS ON THE ADHESION OF GOLD MICROCIRCUIT FILMS

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ABSTRACT

Adhesion is an important factor in controlling properties and performance of thin film devices. It is a critical factor in hybrid microcircuits with multilayer films and dissimilar metal interconnects where diffusion of copper from leads during processing and environmental effects during service can modify the adhesion strength of the gold conductive films. Previous work using gold and gold-copper alloy films to simulate different stages of processing and service showed that copper in solution improved film adhesion. More importantly, it took a combination of stressed overlayers and nanoindentation to trigger interfacial fracture of the gold-copper alloy films. The improvement in performance scaled directly with an increase in film strength. However, during two years air exposure telephone cord buckles formed at the gold-copper alloy film edges, grew slowing across the film surface, and eventually covered the sample. Formation of these buckles shows that a significant degradation in interfacial fracture strength had occurred in these films. We characterized the size and shape of the blisters that formed during nanoindentation of the as-deposited films and in the films following aging. These measurements were then combined with mechanics-based models to determine residual stresses and interfacial fracture energies. This analysis shows that air aging decreased the mode I interfacial fracture energy for the gold-copper alloy film from 3.2 J/m^2 to 1.5 J/m^2. A similar decrease in fracture energy has been observed for many systems exposed to hydrogen from processing and environmental exposure, including copper films, beryllium films, steels and iron- and nickel-based superalloys. This paper describes the effect of environment on resistance of gold-copper alloy film systems to premature interfacial failure, and by comparison with previous studies shows it can be attributed to hydrogen embrittlement.

1 INTRODUCTION

Adhesion is an important factor in controlling the performance of thin film devices. It is a critical factor in hybrid microcircuits where the diffusion of copper from leads during processing and exposure during service can degrade performance and reliability to unacceptable levels (Moody et al [1]). Close examination of microcircuits subjected to accelerated aging show significant levels of copper migration from the copper-cored-leads to the gold-conductive-pads. The gold pads become an essentially uniform solid solution of gold-2wt%-copper while copper concentrations along the lead-pad bond often exceeded 20 wt%. Previous work on a gold-2w/o-copper film and a gold-on-copper film (Moody et al [1]) used stressed tungsten overlayers and nanoindentation to measure the interfacial fracture energy with mechanics-based models. The gold-2wt%-copper alloy film delaminated in circular blisters formed by nanoindenation and the gold-on-copper film delaminated in the form of telephone cord buckles after the tungsten stressed overlayer was deposited. The results show that the gold-copper alloy exhibited higher interfacial fracture energies than the gold-on-copper films. The increase scaled with the film strength and suggested that higher interfacial fracture energies measured for the gold-copper alloy film were attributed to solid solution strengthening (Moody et al [1]). Recently, it was found that environmental exposure led to telephone cord buckling from the sample edges on the gold-copper alloy film. This provided the means to study the how the environment impacts adhesion. Comparison with as-fabricated results illustrate that environmental exposure significantly lowered the interfacial fracture energies indicating a strong environmental effect on long term performance of microcircuit films.

2 MATERIALS

Thin gold-2wt%-copper films sputter deposited onto polished single crystal (0001) sapphire substrates were used from a previous study (Moody et al [6]). The films were deposited on the substrates using a gold-2wt%-copper target with argon as the carrier gas in a vacuum apparatus having a base pressure of 1.3×10^{-5} Pa (10^{-7} torr) to a thickness of 200 nm. All films were deposited at a nominal rate of 0.3 nm/s with final film thickness confirmed using a Tencor Profilometer. A tungsten overlayer was deposited on all films to provide a uniform compressive stress for fracture testing. Deposition was accomplished by placing the gold-2w/o-copper films in a sputter deposition chamber and heating to 170˚C in vacuum to drive off moisture. Cleaning was completed with an RF backsputter to remove contaminants from the surface. The tungsten films were deposited to a thickness of 840 nm at a rate of 0.3 nm/s using a tungsten target and 3.3 mtorr argon as the carrier gas in a vacuum system having a base pressure of 1.3×10^{-5} Pa (10^{-7} torr). Resistance to fracture was determined from circular indentation blisters triggered by nanoindentation of the as-deposited gold-copper alloy films and from uniform width telephone cord buckles which formed during two years air exposure following deposition of the tungsten overlayer. For the nanoindentation tests, a conical diamond indenter with a nominal one μm tip radius and a 90˚ included angle was driven into the films at a loading rate of 600 μN/s to maximum loads of 200, 400, and 600 mN. During each test, the normal loads and displacements were continuously recorded.

3 RESULTS

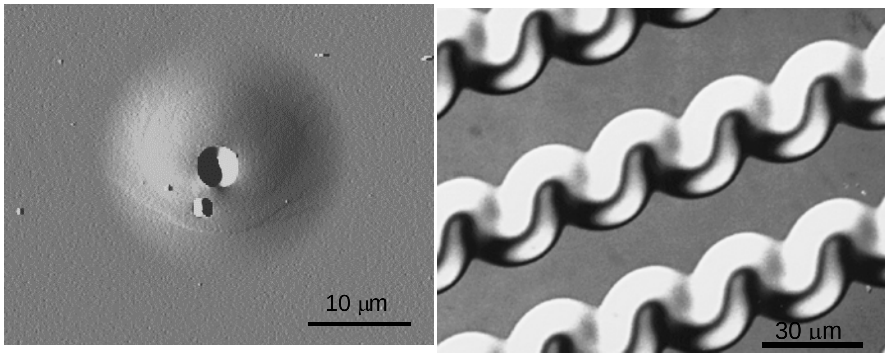
The as-deposited gold-copper film required stressed overlayers and nanoindentation did to induce delamination and the formation of unpinned circular indentation blisters (Figure 1a) which formed during the unloading of the indenter. After a two year air exposure uniform width telephone cord buckles formed at the gold-copper alloy film edges, grew slowing across the film sample, and eventually covered the sample (Figure 1b). Growth of the telephone cord buckles occurs along the advancing circular front of the buckle. Fracture occurred along the film-substrate interfaces with no evidence of gold or copper left on the sapphire substrates.

4 FRACTURE ENERGY MODELS

Interfacial fracture energies can be obtained from the indentation induced circular blisters and the telephone cord uniform width blisters using solutions for film systems where residual stresses dominate fracture behavior. These solutions were originally derived for single layer film-on-substrate systems (Hutchinson and Suo [2], Marshall and Evans [3], Evans and Hutchinson [4]). Work by Bagchi et al. [5] and Bagchi and Evans [6] and more recently by Kriese et al. [7, 8] extended these solutions to multilayer systems by treating the multilayer film as a single film of the same total thickness with a transformed moment of inertia.

4.1 Circular Blisters

Failure in the as-deposited gold-2wt%-copper films required nanoindentation to trigger delamination and circular blister formation. In all blisters, the center was unconstrained giving rise



(a) (b)

Figure 1: (a) Additional stress from nanoindentation was needed to trigger delamination of the as depositied, gold-copper alloy film. (b) Telephone cord buckles spontaneously formed on the aged gold-copper alloy film.

to a single buckle configuration. Following the analyses of Marshall and Evans [3] and Evans and Hutchinson [4], the interfacial fracture energy is given by,

$$\Gamma(\Psi) = \frac{\left(1-\overline{\nu^2}\right)h\sigma_V^2}{2\overline{E}} + (1-\alpha)\frac{\left(1-\overline{\nu}\right)h\sigma_b^2}{\overline{E}} - (1-\alpha)\frac{\left(1-\overline{\nu}\right)h\left(\sigma_V-\sigma_b\right)^2}{\overline{E}} \qquad (1)$$

where σ_V is the stress in the film induced by nanoindentation and σ_r is the residual stress in the film system. For blister formation to occur, the driving stress from residual and indentation contributions must exceed the stress for delamination. Following the analyses of Marshall and Evans [3] and Evans and Hutchinson [4] and more recently by Kriese et al. [8], the delamination stress is given by,

$$\sigma_b = \frac{k}{Bha^2}\left[\frac{E_{Au}}{1-\nu_{Au}^2}\right](I_T) \qquad (2)$$

In this expression, I_T is the transformed moment of inertia for the multilayer film system (Kriese et al [7]), E and ν_{Au2Cu}, are the elastic modulus and Poisson's ratio for the gold-copper alloy films erspectively, a is the blister radius, *h* is the total film thickness, and *B* is the unit width, which cancels when multiplied by the transformed moment of inertia (Kriese et al [8]), and *k* is equal to 14.68 for an unconstrained circular blister. The stress from indentation is given as follows [7, 8],

$$\sigma_V = \frac{\overline{E}V}{2\pi(1-\overline{\nu})a^2h} \qquad (3)$$

where E and n are the average elastic modulus and Poissons ratio of the film system, and V is the displaced indent volume contributing to in-plane stresses. Previous work [9] on tantalum nitride films and analysis of indents in the gold-2wt%-copper films shows that 60 percent of measured displaced indent volume contributes to in-plane stresses.

4.2 Telephone Cord Buckles

Under steady state conditions, the width of the telephone cord blister remains fixed creating a straight-sided blister with growth occurring along the more or less circular front. This gives a steady state fracture energy, Γ_{SS}, as follows (Hutchinson and Suo [2]),

$$\Gamma_{ss} = \left[\frac{\left(1-\bar{\nu}^2\right)h\sigma_d^2}{2\bar{E}}\right]\left(1-\frac{\sigma_b}{\sigma_d}\right)^2 \quad (4)$$

where h , $\bar{E}$ and $\bar{\nu}$ are the multilayer film thickness, and thickness weighted elastic modulus and Poisson's ratios respectively. The stress for delaminating, or the buckling stress is given by (Kriese et al [8], Hutchinson and Suo [2]),

$$\sigma_b = \frac{\pi}{Bhb^2}\left[\frac{E_{Au}}{1-\nu_{Au}^2}\right]I_T \quad (5)$$

where b is the blister half-width. Stress driving blister formation, σ_d can also be determined from the telephone cord blister heights and widths as follows (Hutchinson and Suo [7]),

$$\sigma_d = \sigma_b\left(\frac{3\delta^2}{4h^2}+1\right) \quad (6)$$

where δ is the buckle height, h the film thickness, and σ_b is the buckling stress in the film.

5 DISCUSSION

Blister diameter and indentation displacement volume measurements were combined with residual stress levels from previous work (Moody et al. [1]) and eqn (1) to determine an average interfacial fracture energy of 7.2 J/m2 for the as-deposited gold-copper alloy film. The heights and widths of the telephone cord buckles from Atomic Force Microscopy measurements were used to calculate the stresses for film delamination and buckling in the aged gold-copper alloy system. These stresses were then substituted into eqn (4) giving a steady state fracture energy of 4.1 J/m^2 for the aged gold-copper alloy film.

The uniform width and circular blister fracture energies are mixed mode values consisting of shear and normal contributions. The normal contribution is critical to understanding mechanisms controlling susceptibility to interfacial fracture especially with respect to the work of adhesion. Of the criteria proposed to describe the relationship between mixed mode and mode I contributions, $\Gamma_I=\Gamma(\psi)/[1+\tan^2\{(1-\lambda)\psi\}]$ is most often used. In this expression, λ is a material parameter equal to 0.3 for most materials and ψ is the phase angle of loading described in previous work (Hutchinson and Suo [2], Thouless et al [10]) This expression was then used to calculate the

Table I: Average blister widths, stresses, and fracture energies, are given for the gold-2w/o-copper, and aged gold-2w/o-copper. Data for gold films and gold-on-copper films are also included for comparison.

Film	h_{Au} (nm)	h_W (nm)	b, a (μm)	σ_b (GPa)	σ_V (GPa)	σ_d (GPa)	Γ_{ss}, $\Gamma(\psi)$ (J/m^2)	ψ	Γ_{Ic} (J/m^2)
Au2%Cu	200	840	11.3	3.06	-1.1	-2.1	7.2	-69	3.2
Aged Au2%Cu	200	840	35.6	2.1	--	-2.0	4.1	-89	1.5
Au	200	840	16.4	0.08	--	-1.7	1.8	-79	0.6
Au-Cu	200	840	83.2	0.05	--	-2.1	6.7	-79	2.2

mode I fracture energies for the film systems These results are given in Table I along with average blister widths and diameters, driving stresses, buckling stresses, mixed mode fracture energies, and phase angles of loading for the circular blisters in the as-deposited gold-2w/o-copper films and the uniform width blisters for the aged gold-copper alloy films. Also included are results for thin gold films and gold-on-copper films for comparison. The fracture energies for the as-deposited gold-copper alloy film are consistent with the better durability during handling and testing than exhibited by the as deposited gold and gold-on-copper films. It is unlikely that the gold-2w/o-copper alloy film would have higher bond strength than its constituent elements. This suggests that the higher fracture energy for the alloy film lies with film energy dissipation processes. Previous work (Moody et al [1]) shows improved performance due to higher strength and higher energy required for plastic deformation. Since the film strength remains unchanged in the two year old film the decrease in the adhesion energy is attributable to the environmental effects.

One should note that prolonged environmental exposure of the gold-copper alloy film resulted in a decrease by a factor of two in the calculated interfacial fracture energy. This drop in fracture energy has been observed for a number of systems, including copper (Gerberich et al [11]), beryllium films (Moody et al [12]), and nickel- and iron-based superalloys (Moody et al [13-16], Perra et al [17]). In addition, Moody et al [14] showed using molecular dynamics calculation that hydrogen reduces the fracture stress in a nickel lattice from approximately 18 GPa to 8 GPa at a chemical potential of -2.40 eV. These results show that environment exposure significantly reduces the interfacial fracture energy of gold conductor films with the potential to adversely affect the long-term performance and reliability of hybrid microcircuits.

6 CONCLUSIONS

In this study, highly compressed overlayers were combined with nanoindentation to study the effects that hydrogen has on the interfacial fracture energy of gold-2wt%-copper alloy films. The results showed that the fracture energies for the as-deposited gold-copper alloy film were higher than aged gold-copper alloy film because hydrogen decreases interfacial fracture energy. Comparison with as-fabricated results demonstrate that environmental exposure significantly lowered the interfacial fracture energies, thus showing a strong environmental effect on long term performance of microcircuit films.

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