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Technical Note

Fiducial marks as measures of thin film crack arrest toughness

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Abstract

Carbon fiducial marks are formed during thin film local delamination processes induced either by indentation, forming circular blisters, or by residual stress relief through telephone cord blister formations. Hydrocarbons are sucked into the crack tip during the delamination processes, outlining the crack tip opening angle, which can be used to back calculate thin film adhesion using elastic or plastic analyses presented in the paper. © 2002 Published by Elsevier Science Ltd.

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1. Introduction

Thin film adhesion can be measured by means of the superlayer indentation test [1–4]. Most good-adhered thin films cannot be delaminated by means of regular indentation: films would rather deform plastically around the indenter by forming pileup. To prevent these problems a high modulus hard superlayer, capable of supporting and storing large amounts of elastic energy is deposited on top of the film of interest. Upon indentation a delamination blister forms around the indent, and its area is used to calculate the strain energy release rate (practical work of adhesion). This technique was shown to work with ductile metallic films (Al, Cu, Au, Cr) [2,4–9,15], ceramic (Ta_2N) [10–12] and polymer films [16].

During indentation experiments into Cu thin films with a W superlayer it was found that the crack arrest marks form and correspond exactly to the blister size [13,14]. Marks are formed of carbon, and outline the crack tip, representing its geometry [13,14].

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2. Crack arrest (fiducial) marks

Crack arrest marks were found after the blister removal with an adhesive tape. Scanning electron microscopy showed circles that correspond to the original blister diameter, and those were denoted as crack arrest fiducial marks [13,14]. Atomic force microscopy was performed to measure the feature geometry, giving a width over 1 μm, and a height ranging from 5 to 15 nm. Contact and deflection AFM images of the partially removed blister showing the fiducial crack arrest marks are presented in Fig. 1.

It was originally believed that the crack arrest mark is formed by crushed W and/or SiO_2 debris during the indentation [13]. More likely, however, radial cracking allowed laboratory air with moisture, hydrocarbons and surface debris to be sucked into the blister [14]. The exact source of contamination would be identified later, but whatever the source is, relatively mobile moisture, hydrocarbons and small debris particles were sucked into the crack tip leaving the fiducial mark detected in Fig. 1.

3. Slow crack growth analysis

Upon blister removal with a scotch tape the crack tip residue splits into two fiducial marks, leaving one on the film and substrate sides as shown in Fig. 2. The substrate fiducial mark outlines the crack tip geometry, and its dimensions can be used to extract the thin film crack arrest toughness [13,14].

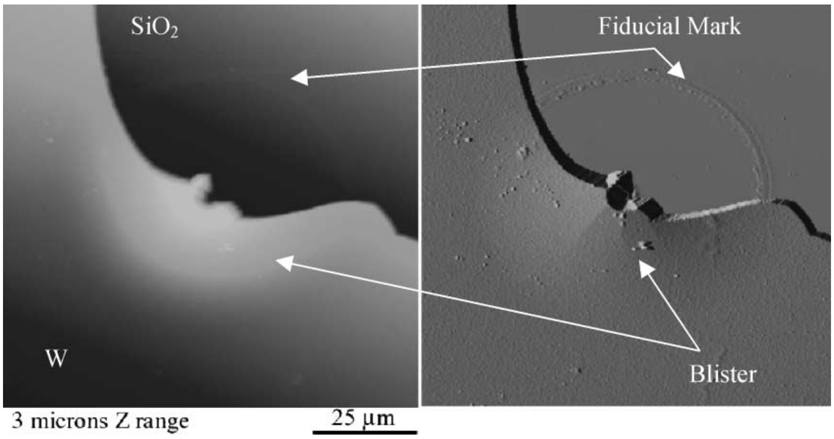

Fig. 1. AFM height and deflection images of partially removed blister, showing fiducial mark underneath.

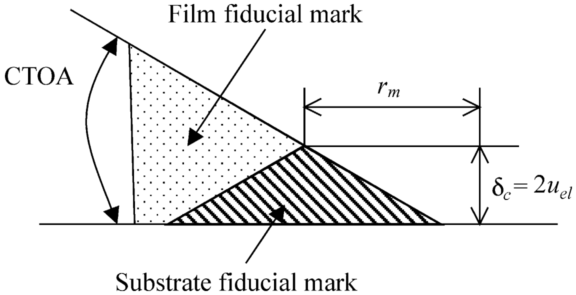

Fig. 2. Fiducial mark geometry.

As discussed in [2,4,15], brittle fracture is observed for thinner Cu films (<100 nm) on Si substrates without a Ti underlayer. The majority of blisters in these films are buckled, so the crack is mostly under Mode I loading. This allows us to use the elastic crack tip opening displacement expressed for the plane stress tensile loading [17]:

$$u_{el}(r) = \frac{K}{E}\sqrt{\frac{8r}{\pi}}. \tag{1}$$

AFM measurements of the fiducial mark on the substrate side provide the height, $\delta_c = 2u_{el}(r_m)$, and the half width of the mark, r_m (Fig. 2), so K_I can be expressed as

$$K_I = \delta_c E\sqrt{\frac{\pi}{32r_m}}. \tag{2}$$

From Eq. (2), for $\delta_c = 8$ nm and $r_m = 1$ μm one finds $K_I = 0.3$ MPa m$^{1/2}$. This is close to the 0.33 MPa m$^{1/2}$ value calculated from the actual G measurements (~0.9 J/m^2) for thinner Cu films using $K = (GE)^{1/2}$ for plane stress [2,4,15]. Since the analysis is purely elastic, it indirectly proves that there is not much plastic energy dissipation at the crack tip for thin Cu films. Previously we also employed a plasticity-based slow crack growth approach based on the Rice, Drugan and Sham (RDS) analysis of the tearing modulus, T_0 [18] to show a similar result [13]. One can obtain a simple expression for strain energy release rate in terms of the crack-tip opening angle (CTOA in Fig. 2):

$$J_{SS} = J_0 \exp\left(\frac{\alpha T_0}{\beta}\right) = J_0 \exp\left(\frac{E\,\mathrm{CTOA}}{\sigma_{ys}\beta}\right), \tag{3}$$

where J_0 is the initial value of the J integral at crack initiation, T_0 is the tearing modulus, $\alpha \approx 1$, $\beta = 5.1$ from the mechanics description, E and σ_{ys} are modulus and yield strength. With 0.01 radians average value of the CTOA, a modulus of 120 GPa, a yield strength of 1 GPa and $\beta = 5.1$, one finds:

$$J_{SS} = J_0 \exp\{0.23\} \approx 1.27 J_0 \tag{4}$$

which means that during slow crack growth the strain energy release rate has barely increased for this 120 nm thick copper film. This again agrees with the actual measured value of ~0.9 J/m^2 for the strain energy release rate.

4. Carbon contamination source

There are three possible sources for carbon: adhesive tape, the diamond indenter and hydrocarbons from the atmosphere. In our previous studies the first two sources were eliminated [13–15], and hydrocarbons from the atmosphere were proposed as a source of fiducial mark formation.

During the course of this study it was found that a similar type of contamination is present in a different film system of Ti_XWN_Y on GaAs, where the telephone cord delaminations formed due to the high residual stress relief (Fig. 3). The carbon traces noted both on the film and substrate surfaces mimic the original telephone cord delamination pattern. Fiducial crack arrest marks are like those observed in the Cu/SiO_2 system. Fig. 3 also shows a carbon Auger map, where brighter areas correspond to higher carbon concentrations. There is almost no carbon present between the original phone cord delaminated areas (black regions in Fig. 3). Most of the carbon goes into the crack tip, outlining the telephone cord topography. Fiducial mark formation may also be associated with the local heating at the crack tip. The heat dissipates fast enough so that the whole sample is not heated up, although the local crack tip temperature may increase substantially. This is a very interesting phenomenon that requires further investigation.

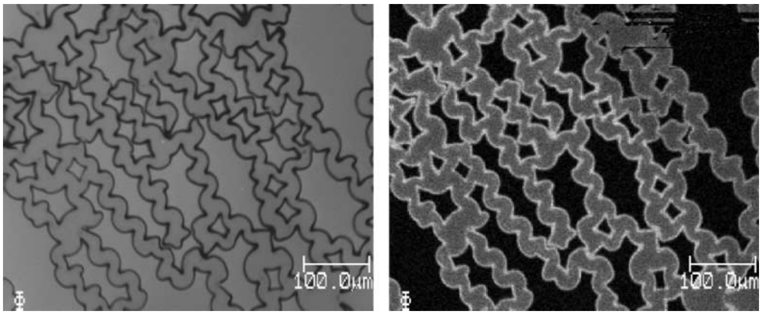

Fig. 3. SEM micrograph and corresponding Carbon Auger map of a GaAs fracture surface upon TiW_XN_Y film removal.

5. Conclusions

We have shown that fiducial marks formed in thin copper film delamination give a good measure of the crack-tip opening displacement. In turn this can be used to estimate the fracture resistance as verified by the superlayer nanoindentation technique. Both elasticity and tearing modulus estimates of the critical stress intensity agree with an independently measured fracture toughness based on the driving force for crack growth. These results strongly suggest that there was nearly negligible plasticity involved in delaminating this 120 nm film.

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