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Fiducial mark and CTOA estimates of thin film adhesion

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Abstract. Carbon fiducial marks are formed during thin film local delamination processes induced either by superlayer indentation forming circular blisters, or by residual stress relief through telephone cord blister formations. Hydrocarbons are sucked into the crack tip during the delamination process, outlining the crack tip opening angle (CTOA), which can be used to back calculate thin film adhesion using either elastic or plastic analyses presented here. Fiducial marks have been observed in two different thin films systems, namely Cu/SiO_2 and TiW_XN_Y/GaAs.

TiW_XN_Y/GaAs system also exhibited biaxial compressive stress-induced phone cord buckling delaminations. Surface AFM CTOA measurement approach is used to estimate the strain energy release rate increase along these phone cords delaminations.

Key words: Adhesion, crack arrest, crack tip opening angle, delamination, fiducial marks, fracture, thin films.

1. Introduction

There are several different methods available to quantitatively measure thin film practical work of adhesion (see Marshall and Evans, 1984; Bagchi et al., 1994, 1995; Vlassak et al., 1992, 1997; Dauskardt et al., 1998; Sanchez et al., 1999). The overview of thin film adhesion measurement methods can be found in our review paper (Volinsky et al., 2002a). One of the ways of measuring ductile thin film adhesion is by means of the superlayer indentation technique (see Kriese et al., 1999; Gerberich et al., 1999; Volinsky et al., 1999a; Tymiak et al., 2000). This is similar to the single layer indentation test originally developed by Marshall and Evans (1984), and extended for miltilayers by Kriese et al. (1999). Most well adhered thin films cannot be delaminated by means of regular single layer indentation: ductile films would rather deform plastically around the indenter by forming pileup. To prevent these problems a high modulus hard superlayer (ex. W, TiW, Ta2N, etc.), 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) (see Volinsky et al., 1999a, 2000; Tymiak et al., 2000; Moody et al., 2000; Schneider et al., 1998, 1999; Kriese et al., 1999; Volinksy, 2000), ceramic (Ta2N) (Moody et al., 1997, 1998b) and polymer films (Volinsky et al.; 2001).

During indentation experiments into Cu thin films with a W superlayer it was found that the crack arrest (fiducial) marks form and correspond exactly to the blister size (Volinsky et al., 1999b). Marks are formed of carbon, and outline the crack tip, representing its geometry (Volinsky et al., 1999a, b). A similar type of carbon contamination was present in a different

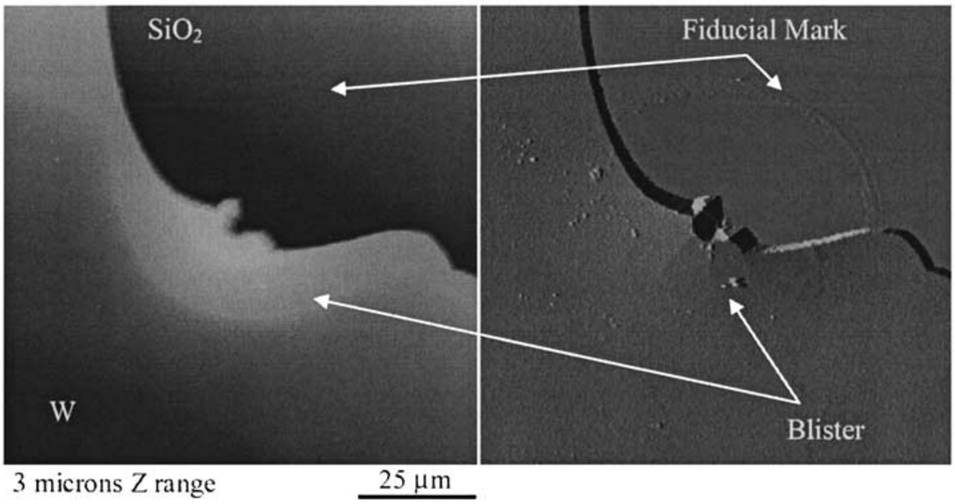

Figure 1. AFM height and deflection images of partially removed blister, showing fiducial mark underneath.

film system of Ti_XWN_Y on GaAs, where the telephone cord delaminations formed due to the high compressive residual stress relief (Volinsky et al., 2002b, c).

This paper demonstrates how Crack Tip Opening Angle (CTOA) outlined by such marks and also estimated from the AFM delaminated surface profiles can be used to assess interfacial fracture resistance of thin films.

2. Fiducial marks

Fiducial 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 (Volinsky et al., 1999a, b). 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. Height and deflection contact AFM images of the partially removed blister showing the fiducial crack arrest marks are presented in Figure 1.

Radial and interfacial cracking allows laboratory air with moisture, hydrocarbons and surface debris to be sucked into the blisters' crack tip (Volinsky et al., 1999a), leaving the fiducial mark detected in Figure 1. Upon blister removal with adhesive tape the crack tip residue splits into two fiducial marks, leaving one on the film and substrate sides as shown in Figure 2. The substrate fiducial mark outlines the crack tip geometry, and its dimensions can be used to extract the thin film crack arrest toughness (Volinsky et al., 1999a, b) as shown in the next section.

In the case of a compressed film, residual stresses can be relieved through local film delamination immediately followed by buckling, forming classic phone cord delamination patterns. A similar type of carbon contamination is present in a different film system of Ti_XWN_Y on GaAs, where the telephone cord delaminations formed due to the high compressive residual stress relief (Figure 3). The carbon traces noted both on the film and substrate surfaces mimic the original telephone cord delamination pattern, except that the cause of initial thin film delamination is different in this case. Fiducial crack arrest marks are like those observed in the Cu/SiO_2 system. Figure 3b also shows a carbon Auger map, where brighter

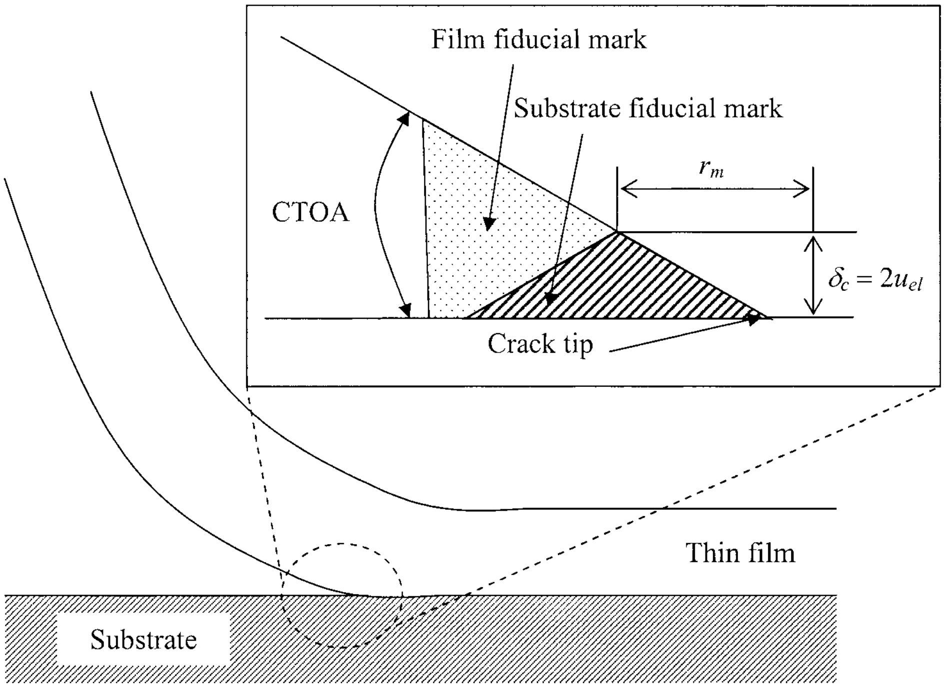

Figure 2. Fiducial mark schematic.

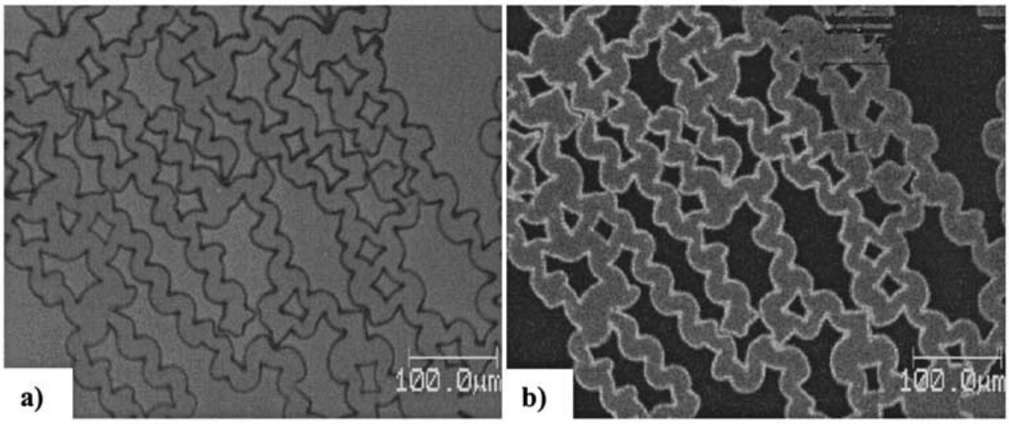

Figure 3. (a) SEM micrograph and (b) corresponding Carbon Auger map of a GaAs fracture surface upon TiW_XN_Y film removal.

areas correspond to higher carbon concentrations. There is almost no carbon present between the original phone cord delaminated areas (black regions in Figure 3), which were exposed to air during the film pull-off with the adhesive tape. Most of the carbon goes into the crack tip, outlining the telephone cord topography. Fiducial marks form in conjunction with local film decohesion, with crack front on the order of 20–200 μm in length, and have not been observed when initially adhered film goes through an edge lift-off or an adhesive tape pull-off test.

3. Elastic and plastic crack growth analysis

As discussed in (Volinsky et al., 1999c, 2002a; Volinsky, 2000; Tymiak et al., 2000), brittle fracture is observed for thinner Cu films (< 100 nm) on Si substrates. The majority of blisters in these films are buckled, so the crack has a significant Mode I loading component, which allows one to use the elastic crack tip opening displacement expressed for the plane stress tensile loading at a distance r from the crack tip (Lawn, 1993):

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

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 (Figure 2), so K_I can be expressed as

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

From Equation (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 ($\sim$0.9 J m^{-2}) for thinner Cu films using $K = (GE)^{1/2}$ for plane stress (Volinsky et al., 1999c; Volinsky, 2000; Tymiak et al., 2000). 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.

One can also use a plasticity-based slow crack growth approach based on the Rice, Drugan and Sham (RDS) analysis of the tearing modulus, T_0 (Anderson, 1991) to show a similar result (Volinsky et al., 1999b). The RDS model of the tearing modulus, T_0, gives

$$T_0 = \frac{E\delta_c}{\alpha\sigma_{ys}r_m} - \frac{\beta}{\alpha}\ln\left(\frac{e\lambda E J_0}{r_m\sigma_{ys}^2}\right), \tag{3}$$

where δ_c/r_m is the crack-tip displacement at a distance r_m behind the crack tip where it is measured or crack tip opening angle (CTOA $= \delta_c/r_m$), $\alpha \approx 1$, $\lambda \approx 0.2$, e is the natural logarithm base, E and σ_{ys} are modulus and yield strength, $\beta = 5.1$ from the mechanics description and J_0 is the initial value of the J integral at crack initiation. Calculation demonstrated that the first term dominated the second for very high yield strength thin films with toughness less than 100 J m^{-2}, giving

$$T_0 \approx \frac{E\delta_c}{\alpha\sigma_{ys}r_m}. \tag{4}$$

Since the steady-state strain energy release rate can be given in terms of the tearing modulus by

$$J_{SS} = J_0\,\exp\left(\frac{\alpha T_0}{\beta}\right) \tag{5}$$

it is seen that combining Equations (4) and (5) leads to a simple expression for strain energy release rate in terms of the crack-tip opening angle,

$$J_{SS} \approx J_0\,\exp\left\{\frac{E\cdot\mathrm{CTOA}}{\sigma_{ys}\beta}\right\}. \tag{6}$$

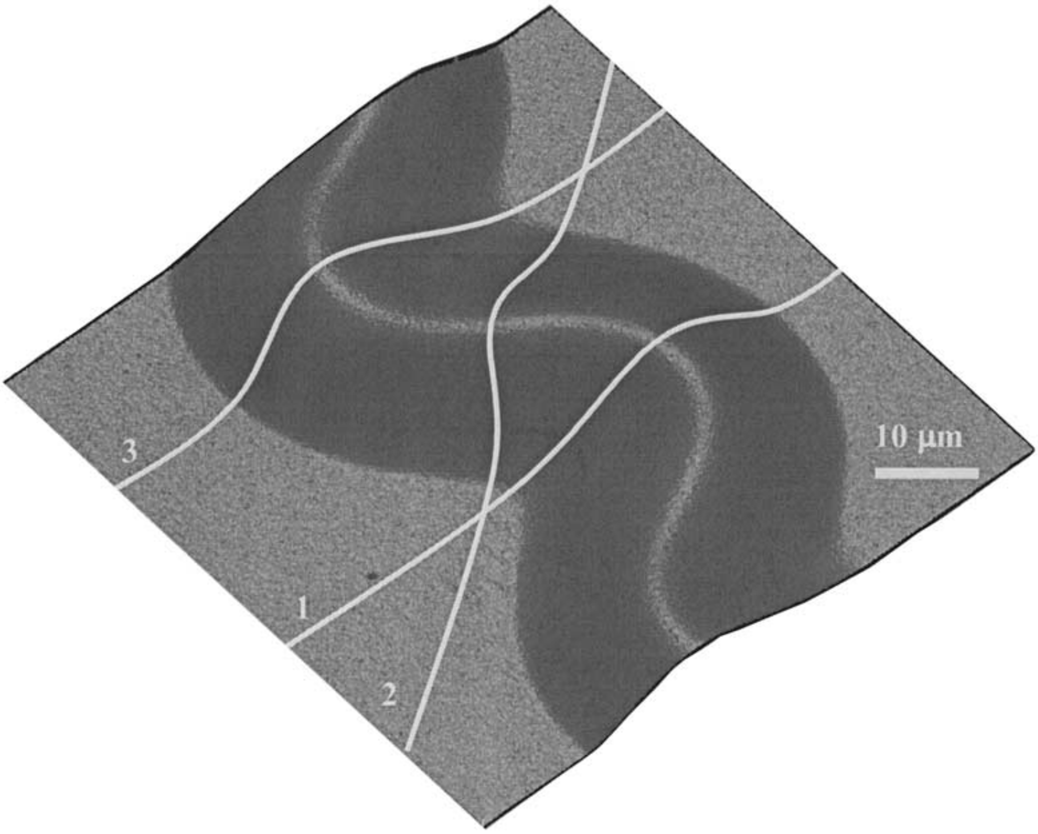

Figure 4. AFM surface plot of Ti_XWN_Y phone cord delamination showing cross-sectional profiles locations shown in Figure 5.

With 0.01 radians average value of the CTOA, a modulus of 120 GPa, a yield strength of 613 MPa and $\beta = 5.1$, one finds $J_{SS} = J_0 \exp\{0.38\} \approx 1.47J_0$, 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 within experimental error with the actual measured value of ~ 0.9 J m^{-2} for the strain energy release rate. It may be noted that Equation (6) represents plane strain behavior, which seems inconsistent with the thin films evaluated. However, the stress intensity of only 0.33 MPa m$^{1/2}$ in conjunction with a yield strength of 613 MPa (Volinsky, 2000) represents an experimentally small plastic zone size radius of about 47 nm even if a plane stress estimate of $K_I^2/2\pi\sigma_{YS}^2$ is used. This only represents about 1/3 of the film thickness. Even more constraining is the 1 μm superlayer of tungsten with a modulus and yield strength nearly three times that of copper. Given these considerations, we assume the plastic zone size is best represented by plane strain conditions.

4. CTOA-based strain energy release rate for the phone cord delamination

It was earlier proposed that the CTOA could be estimated by measuring the angle of a circular indentation-induced blister at the surface of the delaminated film (Moody et al., 2001). We use the same approach to estimate the strain energy release rate at different sections of a phone cord delamination. An AFM surface plot of a Ti_XWN_Y film phone cord delamination is shown

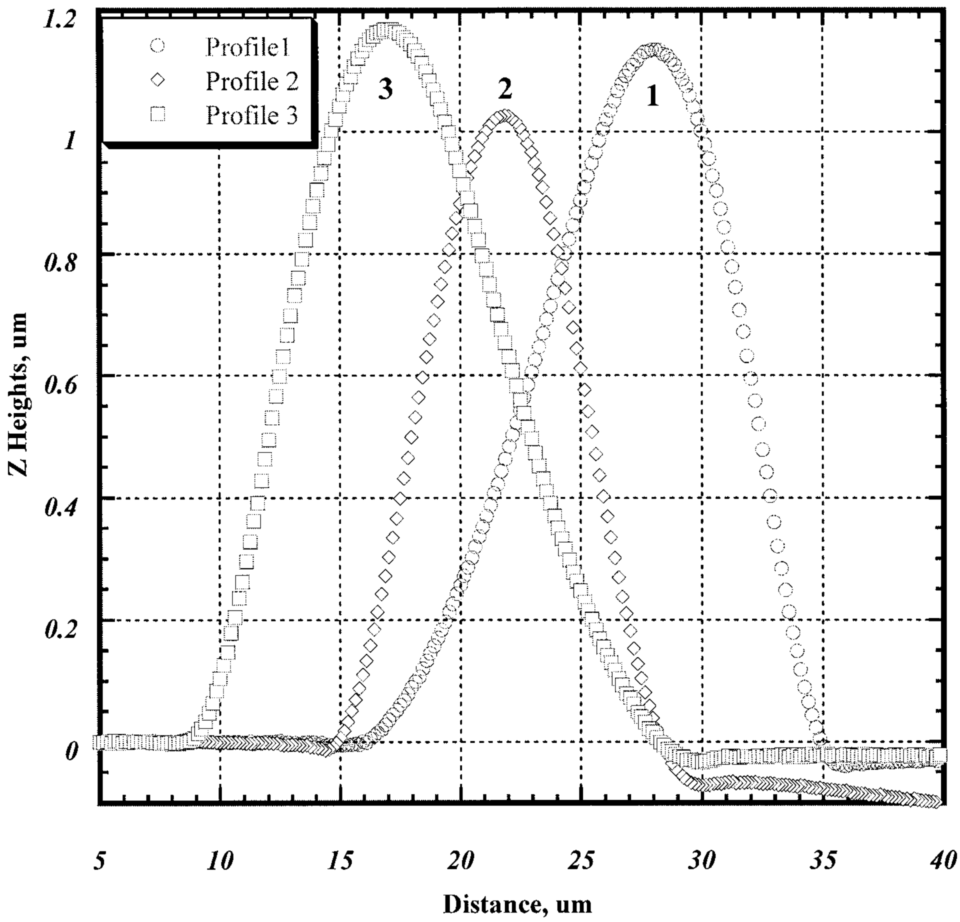

Figure 5. Phone cord cross-section profiles as identified in Figure 4.

Table 1.

Type	Angle, deg	J_{ss}/J_0
Straight	1.9–2.3	7 –10.6
Curved Shallow	0.9–1.7	2.3–5.7
Curved Steep	2.9–3.8	19.6–43

in Figure 4. These cross-section locations are identified in Figure 4 and the corresponding profiles are shown in Figure 5. The AFM image was plane fit using the left non-delaminated flat portion before acquiring the cross-section datum. While profile 2 is symmetric, profiles 1 and 3 are not symmetric, with the higher angle on the outer side of the phone cord delamination (Figure 5).

How can the angle measured at the film surface be used to estimate CTOA, when as seen from the profiles, this angle is not constant, there is actually a radius of curvature due to the film bending (Figure 2). Figure 6 shows magnified regions of the curvature data for profile 1. It turns out that on the scale of the film thickness (0.3 μm) the data is linear, as presented

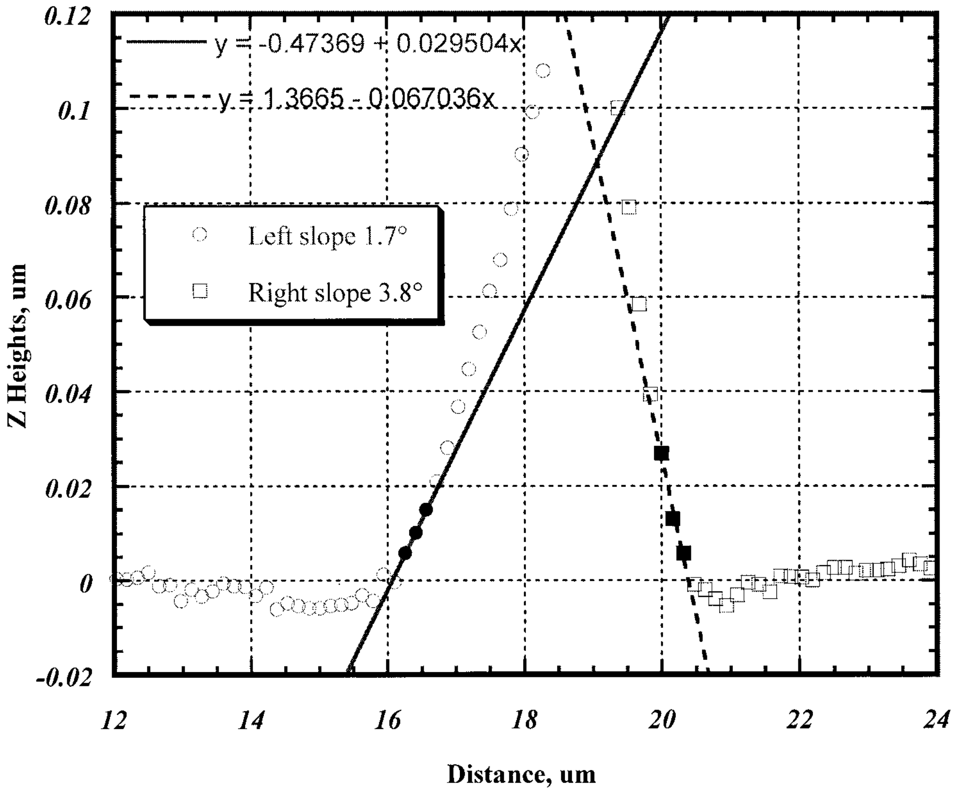

Figure 6. Scan 1, left and right slopes blown, showing angle measurement. Note: the right slope data was shifted 5 μm to the left for clarity. Only the filled data points used for fitting.

in Figure 6 with the filled data points. If this truly represents the CTOA, Equation (6) can be used for estimating the strain energy release rate increase at different stages of the phone cord buckling delamination. Results representing the range of angles and corresponding J/J_0 ratios are summarized in Table 1 based on the elastic modulus of 300 GPa and yield strength of 1 GPa for this Ti_XWN_Y film. For the straight-sided portion of the phone cord (Figure 4, profile 2) there is only a 10-fold increase in the strain energy release rate compared to a 43-fold increase for the steep curved portion of the phone cord.

5. Conclusions

We have shown that fiducial marks formed in thin 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. A procedure for measuring CTOA is presented for the case of phone cord buckling delamination.

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