

Adhesion measurement of thin films by indentation

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An indentation technique is described for measuring the shear strength of the interface between a thin film and a rigid substrate. A simple elastic/plastic analysis is used to describe the experimental results and good agreement between the two is found. The method may also be used to estimate the interfacial toughness so that the important parameters characterizing both initiation and propagation of adhesive failure may be determined from one simple experiment. The analysis is extended to describe the effect of residual stress in the film. Also, the stresses around a pinhole defect in a stressed film are examined.

Thin films are widely used to modify various properties of a substrate surface such as electromagnetic, chemical, mechanical, or aesthetic properties. Often suitable film materials are difficult to bond to the substrate and poor adhesive strength can be the rule rather than the exception. A mismatch in the elastic properties between the substrate and film can cause stress enhancement at the already weak interface. Debonding and subsequent film removal may result and interfacial failure is frequently the dominating mechanism limiting the reliability of the system. The rapid increase in the use of thin-film technology has led to considerable interest in the mechanics of such systems of late.

Clearly, reliable measurements of the interfacial strength are required in order to help improve adhesive properties as well as for quality assurance. Suitable techniques are only now becoming available. Chiang, Marshall, and Evans¹ suggest an indentation technique in which a Vickers pyramid is loaded onto the film. The resulting plastic deformation puts the surrounding film into compression and the delamination crack that forms propagates under the influence of interfacial shear stress and also interfacial tensile stress developed by buckling of the film over the delaminated region. In later work,²⁻⁴ a fracture mechanics approach is taken to investigate the propagation of the delamination cracks both analytically and experimentally in order to examine the interfacial fracture resistance. However, this work does not consider initiation of the interlaminar fracture, and indeed cannot, since the analysis is only applicable to interfacial cracks that are significantly larger than the film thickness. In the system investigated here large indentation loads could be sustained without interfacial cracking which indicates that, in some systems at least, crack initiation, rather than propagation, controls the failure mechanism. This in no way detracts from the value of the previous work on propagation, but rather, an understanding of both processes is required in order to fully characterize the interface for these types of systems.

The scaled-up model experiments described here are shown schematically in Fig. 1 and involve indenting poly-

ter-coated glass blocks by steel spheres. The glass surface was evenly abraded to give good mechanical adhesion of the film. Upon loading the indenter, plastic deformation of the film occurred without debonding until a critical load P_c was reached, at which time interfacial fracture was observed to spontaneously initiate from a position below the edge of the contact and to propagate radially outwards until an equilibrium radius was reached, at which point the coating was buckled. Subsequent growth of the crack upon further loading can be expected to be described by the propagation work,¹⁻⁴ although this was not investigated at the time of these experiments. In this system debonding was visible to the naked eye; in opaque systems it may be detected acoustically, either passively (at higher loads debonding occurred with an audible "pop") or actively with acoustic imaging. A central disk of film beneath the indenter was observed to remain in contact with the substrate after unloading the indenter. The radius of this region b , the critical load for debonding P_c , and the contact radius at this time a_c , were measured for various film thicknesses h for two indenter radii ($R = 2$ and 3 mm). Figure 2 shows the variation of the debonding load as a function of film thickness where each point represents the average of typically six indentations. Error bars are omitted since they are small—the scatter about the best fit line is due to systematic differences between films of different thicknesses rather than random variations in the values of P_c for one thickness. The mean contact pressure, or hardness,

$$H = P_c / \pi a_c^2, \quad (1)$$

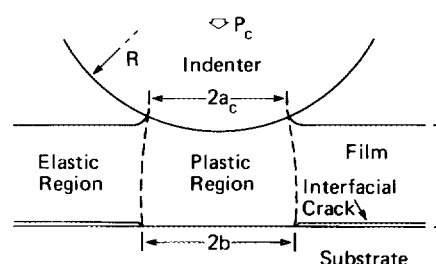


FIG. 1. Schematic diagram of the indentation experiment showing the interlaminar crack and the load P_c and contact radius a_c at the crack initiation point.

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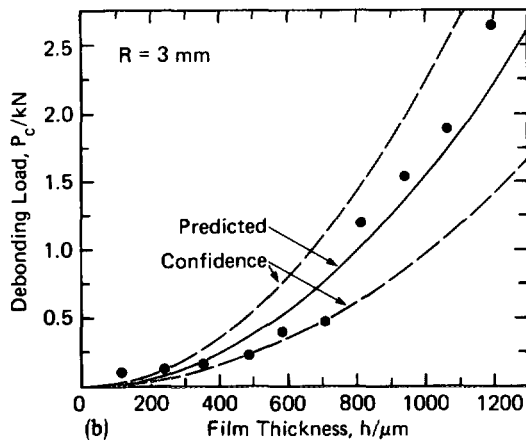
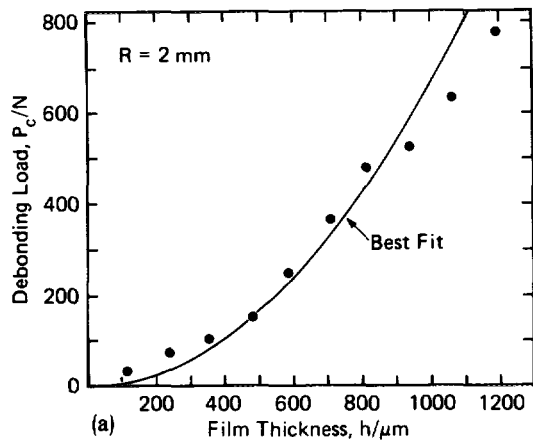


FIG. 2. Critical load to debonding P_c as a function of film thickness. (a) Data for 2-mm radius indenter showing best fit line. (b) Data for 3-mm radius indenter showing the behavior predicted from the data of (a).

was found to be constant with film thickness although it was different for the two indenter radii $(H, R) = (247 \pm 8 \text{ MPa}, 2 \text{ mm})$ and $(212 \pm 9 \text{ MPa}, 3 \text{ mm})$ indicating a certain amount of strain hardening of the film. The radius of the central undebonded region was consistently found to be approximately 10% larger than the contact ($b \approx 1.1a_c$).

These results suggest a simple model for the debonding mechanism, shown schematically in Fig. 3. The plastic deformation zone is modeled as a cylinder of radius a (equaling a_c when an interlaminar fracture initiates). If the mean pressure on the upper surface of the cylinder is H , then the radial pressure across its curved surface, deduced from the Tresca criterion, is $H - Y$, where Y is the film compressive yield stress. For bulk material $H \approx 3Y$, but for a thin film, the numeric factor is larger than 3. However, if the contact radius is not too much larger than the film thickness, the factor 3 is sufficiently accurate and the radial pressure is $2H/3$. The plastic zone may now be conceptually removed and replaced by a hole under an internal pressure $2H/3$. The elastic deformation of the surrounding film may then be determined by matching the radial stress, averaged through the film thickness, to this internal pressure. A suitable analysis for the deformation is given by Matthewson⁵; while this analysis is most accurate for $a/h > 2$, the errors that occur for $a \approx h$ are not inconsistent with the spirit of the present analysis; also

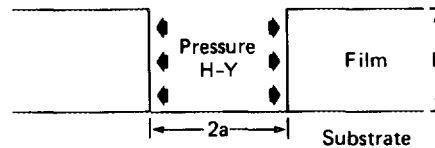


FIG. 3. Model system for the indentation in which the plastic region is replaced by a cylindrical cavity of radius a under an internal pressure $H - Y$.

the results are analytic and expressible in closed form, which is not the case for more general analyses. It is found that the maximum interfacial shear stress is located at the film/hole boundary (i.e., the elastic/plastic boundary) $r = a$, and is given by

$$\tau = \frac{-(2/3)H}{K_1'(\alpha a/h)/K_1(\alpha a/h) + \nu h/\alpha^2 a}, \quad (2)$$

where $K_1'(x) = dK_1(x)/dx$ and $\alpha = \{6[(1 - \nu)/(4 + \nu)]\}^{1/2}$. ν is the coating Poisson's ratio and $K_1(x)$ is the first-order modified Bessel function of the second kind. The normal stress across the interface at $r = a$ is zero. It is therefore proposed that a suitable criterion for the initiation of an interfacial fracture is that the interfacial shear stress should exceed some critical value τ_c at the elastic/plastic boundary $r = a$, when a becomes equal to a_c . (While the elastic/boundary at the interface is closer to $r = b$, the 10% difference between a_c and b is ignored.) For given values of H , τ_c , and a_c , Eq. (2) reduces to the simple criterion that a_c/h should be a constant at fracture. Equation (1) implies that $\sqrt{P_c}/h$ is also a constant. Linear regression fitting to data of Fig. 2(a) (with a constraint to pass through the origin) gives the parabola shown in that figure. Considering the simplicity of the analysis, the agreement between experiment and theory is excellent, especially since only one free parameter is used in the fitting process. The interfacial shear strength is deduced to be $\tau_c = 111 \pm 4 \text{ MPa}$ assuming $\nu = 0.33$. The error bounds on the estimate of τ_c do not represent the absolute error, which could conceivably be as large as a factor of 2, but do represent the uncertainty in the best fit value and so are a measure of the reproducibility of the value. This technique is therefore capable of detecting very small changes in adhesive strength. The value of τ_c is close to the expected yield strength of the film ($\sim H/3$) and is therefore consistent with the adhesion being mechanical in nature—"tongues" of film material keyed into abrasion pits in the glass surface need to be sheared off in order to initiate the crack. Note that buckling of the film generates tensile stresses across the interface² so that propagation of a large crack is easier than for the initial flaw in this particular system.

Figure 2(b) shows the predicted behavior for the 3-mm indenter using the value of τ_c measured for the 2-mm indenter. No free variables are used in making the prediction which is in good agreement with the data, for all except the thinnest films, within the uncertainty limits of plus and minus one standard deviation. The good fit clearly demonstrates the self-consistency of the analysis. The large difference in failure loads between the two indenters is just due to the difference in the values of H .

The indentation technique may be used to estimate interfacial shear strength using the analysis presented here. Further loading after fracture yields information on the growth of the fracture. Thus, this simple method can be used to simultaneously examine the initiation and propagation stages of interfacial fracture. The interfacial toughness and strength could then be used to deduce the size of the defects from which fracture initiates in an analogous manner in which the Griffith equation is used for homogeneous materials. The toughness and defect distributions are both required to fully specify the interfacial fracture behavior.

In practice it may be difficult to apply a stress-free film. Residual stresses are concentrated at defects, such as pinholes, and can lead to debonding in the absence of external forces as well as modify the stresses around any indentation. While the film used in the experiments described here was virtually stress-free, the analysis of the model may be readily extended to account for residual stresses in the film.

Consider a film under a uniform tensile residual stress σ_r (taken as positive tensile). At large distances from any discontinuity in the film there is no shear stress acting across the interface. Any cylindrical shaped region of film of radius a may be conceptually replaced by a hole under an internal negative pressure $-\sigma_r$, without affecting the stress or strain distribution of the surrounding film material. If an indentation is now applied such that, in the absence of any prestress, it could be modeled by a hole under an internal pressure $+\sigma_r$, then the interfacial shear stress at the hole edge is given by Eq. (2) but with the term $(2/3)H$ replaced by σ_r . The cavity is then under zero internal pressure and we are

therefore modeling a pinhole in the film. The modified version of Eq. (2) then may be used to predict the critical pinhole radius a_c for initiating interfacial fracture for a given critical interfacial shear strength τ_c . Since a_c increases for increasing τ_c , the edge of the film ($a_c/h \rightarrow \infty$) is most susceptible to failure unless the thickness is flared.

It is a straightforward extension of the above arguments to deduce that indentation initiation of interfacial failure for a film under a residual stress σ_r is described by Eq. (2) with the term $(2/3)H$ replaced by $[(2/3)H + \sigma_r]$. A tensile residual stress therefore increases the tendency to debond.

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