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Analysis of Critical Debonding Pressures of Stressed Thin Films in the Blister Test

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This paper reports a model for the relationship between critical debonding pressures and the work of adhesion of thin films in the blister test. Previous models have neglected the possible role of residual stresses in the film on the critical pressure. The model reported here shows that these stresses may have a large effect on the relation between the critical pressure and the work of adhesion. A similar model is developed for an alternative blister geometry, the annular or "island" blister. It is shown that films which cannot be peeled using the standard blister test (due to exceeding the tensile strength limit of the film before initiating a debond) can be peeled by varying the geometric parameters of the island blister.

KEY WORDS Adhesion; blister geometry; critical debonding pressure/work of adhesion relationship; island blister; peeling; residual stresses.

INTRODUCTION

The blister test for adhesion measurement was first reported in 1961 by Dannenberg.[1] More recently, the test has been used to measure the adhesion of polymer films[2,3] and adhesive tapes.[4] Much of this work involves using fracture mechanics to relate the "critical pressure" (the pressure at which debond initiates) to the work of adhesion of the film or tape. However, the role of residual stresses in the films has often been neglected. Further, the blister test often fails for well-adhered thin films because the tensile strength of the film is exceeded before peel is achieved. This paper reports, first, a model of the blister test using fracture mechanics which illustrates the effects of these residual stresses and, second, an analysis of alternative blister geometries which allow the peeling of films at lower pressures than in the standard blister geometry.

FRACTURE MECHANICS METHOD

The concept of using fracture mechanics in adhesive failure studies was proposed by Williams.[5] In ordinary fracture mechanics, the effects of plastic deformation are important, since, except for ideally brittle materials, all materials undergo some yielding before fracture. However, in applying fracture mechanics to problems involving adhesive failure, it is assumed that adhesive failure occurs at stresses much lower than those necessary to cause large-scale plastic yielding. Under these restrictions, linear elastic fracture mechanics may be applied.

Consider a pressurized blister of film adhered to a substrate as shown in Figure 1. Using a Griffith argument,[6] during any virtual increment in crack area, the total energy of the peeling system must be constant. Differentially, this can be expressed as:

$$\delta E = \delta \Pi + \delta S = 0 \tag{1}$$

where Π is the potential energy of deformation of the blister and S is the energy of forming new surface. Rearrangement of Eq. (1) yields:

$$\delta \Pi = -\gamma_a \delta A \tag{2}$$

where γ_a is defined as the work of adhesion and δA is an increment

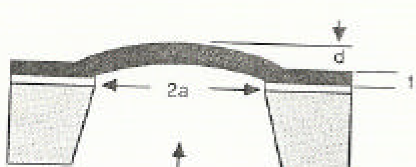

FIGURE 1 Definition of blister parameters.

of crack area. Thus. if expressions for the potential energy of deformation of the blister can be obtained, the work of adhesion can be found by applying the peel criterion (2).

The potential energy of the blister is related to both the blister load-deflection behavior (how the blister deforms in response to an applied load) and the blister geometry. Therefore, in order to apply Eg. (2), we have assumed various blister geometries and calculated the load-deflection behavior of each. The standard blister geometries of interest in our work are a square of side length *2a* and a circle of radius *a*. The films to be blistered can be treated as membranes (no resistance to bending) or as plates, and are characterized by a Young's modulus *E*, a Poisson's ratio v, and an in-plane residual stress *ao*. Many authors have taken up the subject of the load-deflection behavior of such plates and membranes.[7-10] We have applied the energy minimization methods of Timoshenk08 and Way ,9 but have modified them to include the possible contribution of residual stresses to the film behavior.

Using these methods, it is readily shown11 that the load-deflection behavior of all three cases (square membrane, circular membrane, clamped circular plate) can be described by the equation:

$$p = k_1 d^3 + (k_2 + k_3) d \quad (3)$$

where *p* is the applied (uniform) pressure, *d* is the deflection of the blister at its geometric center, and *k1*, *k2*, and *k3* are functions of the geometry of the test site and of the type of film (plate or membrane). Equation (3) is the general load-deflection relation which will be used in the analysis of the blister potential energy.

The values of the constants in Eq. (3) for the three cases examined here are given in Table I, assuming a Poisson's ratio of 0.25. Most of the constants are not very sensitive to Poisson's ratio; for example, the value of *k1* for the square membrane changes from 1.83 to 2.05 when v changes from 0.25 to 0.35. The full dependence of the constants on Poisson's ratio is given in the Appendix.

Consider the flawed elastic body shown in Figure 2, where *P* is some generalized load-point force acting on the body and ? is the corresponding work-conjugate displacement through which the body moves.[6] The potential energy of deformation can be related to these generalized loads and displacements by:

?= *V* - *P*?= - *V** *for* dead loading (prescribed *P)* (4a)

TABLE I
Geometric constants for adhesion model

	Square membrane[a]	Clamped circular plate[a]	Circular membrane[b]
k_1[c]	$1.83Et/a^4$	$2.77Et/a^4$	$3.56Et/a^4$
k_2	0	$64D/a^4$[d]	0
k_3	$\pi^4 t\sigma_0/64a^2$	$4t\sigma_0/a^2$	$4t\sigma_0/a^2$
c_1	$0.429Et$	$2.42Et$	$0.917Et$
c_2	0	$192D/\pi$	0
c_3	$\pi t\sigma_0/512$	$12t\sigma_0/\pi$	$8t\sigma_0/\pi$
Δ	$16a^2d/\pi^2$	$a^2d\pi/3$	$a^2d\pi/2$
da/dA	$1/2a$[e]	$1/2\pi a$	$1/2\pi a$

[a] k values taken from Timoshenko analysis of plate/membrane under zero residual stress,[8] extended to account for residual stress.
[b] k values taken from Beams.[10]
[c] Assumes a value of Poisson's ratio of 0.25.
[d] D = plate flexural rigidity $= Et^3/12(1-\nu^2)$, ν = Poisson's ratio.
[e] Assumes incrementally symmetric peel.

or

$$\Pi = V \qquad \text{for fixed-grip loading (prescribed } \Delta) \tag{4b}$$

where V and V^* are the strain energy and complimentary strain energy of the body, respectively. Substitution of these relations into Eq. (2) yields:

$$\gamma_a = \left[\frac{dV^*}{da}\right]_P \frac{da}{dA} \qquad \text{(fixed load)} \tag{5a}$$

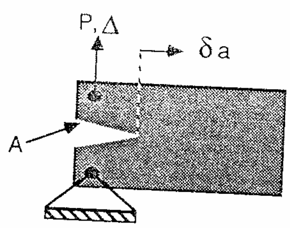

FIGURE 2 A cracked and loaded structure.

$$\gamma_a = \left[\frac{dV}{da}\right]_\Delta \frac{da}{dA} \qquad \text{(fixed grips)} \tag{5b}$$

where a is the crack length (in this case, the blister size) and A is the crack area. It can be shown[6] that expressions (5a) and (5b) are mathematically equivalent to first order.

For the case of lateral load uniformly distributed over a thin plate or membrane (ratio of film thickness t to blister size $a \ll 1$), the work-conjugate force and displacement are related to the actual load and deflection by:

$$P = p$$
$$\Delta = \int_A w(r) d^2r \tag{6}$$

where p is the blister pressure, $w(r)$ is the blister deflection at position r due to that pressure (note that d as defined in Eq. (3) is the same as $w(0)$), and A is the blister area. Equation (3) can now be written in terms of the generalized work-conjugate force and displacement:

$$P = B_1\Delta^3 + B_2\Delta \tag{7}$$

where B_1 and B_2 are functions of geometry, but not of P or Δ. The strain energy is then given by:

$$V = \int_0^\Delta P(\Delta)\, d\Delta + V_r \tag{8}$$

where V_r is the strain energy due to the residual stresses and strains. For the case of the blister under residual tensile stress, increasing crack size (a) does not change V_r. This is because as long as the edges of the film are attached to the substrate, relaxation of the residual stress cannot occur. Therefore, although the residual stress and strain affect the peel criterion through the load-deflection behavior, the energy balance need consider only the elastic strain energy stored in the blister. Note that this assumption is not valid for films under compressive stress, which may undergo stress relaxation by buckling once they have debonded from their substrates.[12] The assumption is also invalid if the blister substrate is to that of Williams:[5]

$$\gamma_a = 0.5 p_c d_c \tag{10}$$

where d_c is the deflection at the center of the plate at pressure p_c. Alternatively, for the case of a circular membrane undergoing large deflections with zero residual stress, ($c_2 = c_3 = 0$) Eq. (9) becomes:

$$\gamma_a = 0.625 p_c d_c \tag{11}$$

Gent[4] has also analyzed this case and has obtained a value of 0.65 for the premultiplying factor in Eq. (11), due to the assumption of slightly different load-deflection behavior.

In order to illustrate the effect of the residual stress, we will consider a circular membrane undergoing large deflections which is under varying degrees of residual stresses. In this case, substitution of the appropriate parameters from Table I into Eq. (3) yields:

$$p_c = 3.56 \frac{Et}{a^4} d_c^3 + \frac{4\sigma_0 t}{a^2} d_c \tag{12}$$

which has been obtained by Beams[10] assuming a Poisson's ratio of 0.25. Corresponding substitution of parameters into Eq. (9) yields:

$$\gamma_a = 2.22Et(d_c/a)^4 + 2.00\sigma_0 t(d_c/a)^2 \tag{13}$$

The relation between γ_a and p_c can be obtained by simultaneous solution of Eqs (12) and (13). The results of this solution are presented in Figure 3, a logarithmic plot of the work of adhesion (normalized by the film modulus and thickness) *versus* critical pressure (normalized by blister size, film modulus and film thickness) for various residual stresses. As can be seen, for zero residual stress, a slope of 4/3 is obtained, agreeing with the derivation of Gent.[4] As the residual stress is increased the adhesive energy corresponding to a given critical pressure decreases; this is due to energy expended in deflecting the film against the residual stress. At high enough stress, the slope of the critical pressure relation becomes equal to 2. This corresponds to a linear load-deflection relation (with the cubic term in Eq. (12) being negligible), and thus leads to the same functional form as a blister undergoing small deflections (where the load-deflection relation is also linear).[5] Finally, at large pressures, the effect of the residual stress on the critical pressure becomes small since the load-deflection relation is

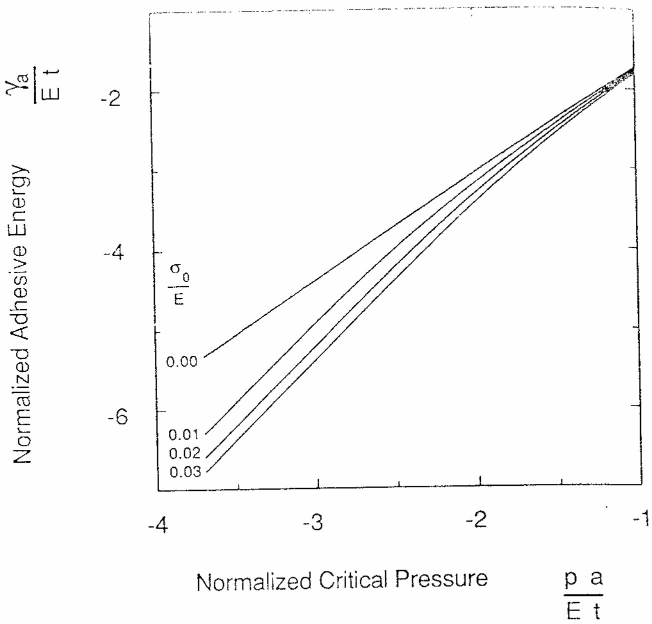

FIGURE 3 A log-log plot of the work of adhesion of a film (normalized by the thickness and Young's modulus of the film) as a function of the critical pressure of a blister (normalized by the blister size and by the thickness and Young's modulus of the film), parameterized by the residual stress in the film (normalized by the Young's modulus of the film).

dominated by stretching against the modulus (with the linear term in Eq. (12) being negligible).

We now present a numerical example based on polyimide films used in our work. Typical values[13] of the various parameters in Eqs (12) and (13) are:

$$E = 3\,\text{GPa}$$
$$\sigma_0 = 30\,\text{MPa}$$
$$t = 10\,\mu\text{m}$$
$$a = 5000\,\mu\text{m}$$
$$v = 0.25$$

Suppose the critical pressure for such a film is measured to be 10 psi [illegible] $\times 10^3$ Pa). Neglecting the residual stress, a standard analysis would yield a value for γ_a of 32 J/m^2. In contrast, taking into account the residual stress leads to a value of γ_a of 24 J/m^2. Neglecting the stress has led to an overestimation of γ_a of 32%. This energy went into deflecting against the residual stress instead of debonding.

Another calculation which can be performed is the tensile strength limit. Suppose the above film has an effective ultimate strain of 2%. For a spherical cap, it can be easily shown that the maximum center deflection can be related to the ultimate strain $\varepsilon_{\mathrm{ult}}$ by Ref. 2:

$$(d/a)^2_{\max} = 1.5\varepsilon_{\mathrm{ult}} \tag{14}$$

Taking into account the already present intrinisc strain (and assuming a Poisson's ratio of 0.25), the maximum deflection which can be sustained is 685 μm, corresponding to a critical pressure of 12.7 psi (87.8×10^3 Pa) and a work of adhesion of 35 J/m^2. Greater values of the work of adhesion cannot be measured using the above geometric and film parameters, due to the tensile strength limit of the film. This problem can be overcome by using thicker films; this has been done for the peel test.[14] In the blister test, we have additional flexibility. Different geometries are possible which can facilitate peel of thinner films even in systems with very good adhesion. These are examined in the following section.

ISLAND BLISTERS

Equation (9) suggests that if a geometry can be found in which da/dA can be increased without simultaneously decreasing dV^*/da (see Eq. (5)), larger values of γ_a may be measured at the same load. For simple blisters, this derivative is inversely proportional to the membrane size (Table I).

Decreasing the membrane size fails since the deflection Δ will also decrease. However, consider an annular "island" structure of outer radius a_2 and inner radius a_1 as shown in Figure 4. The blistering will now occur only off the center island. The deflection Δ (and therefore V^*) of this blister is a function of the difference $a_2 - a_1$, which changes only slightly during peel. The derivative

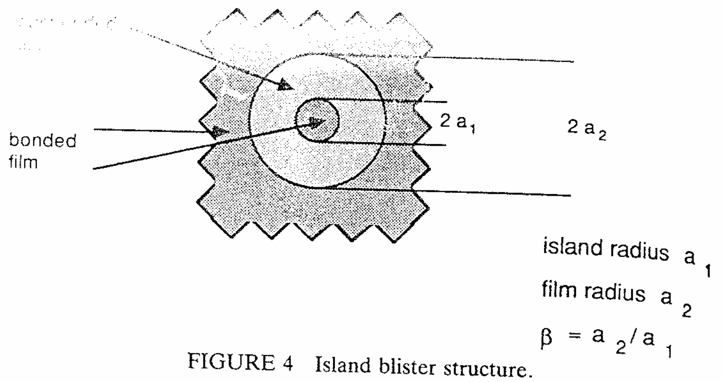

FIGURE 4 Island blister structure.

da/dA, however, is inversely proportional only to a_1. Thus, a large geometric advantage can be obtained by decreasing a_1 (large da/dA) while keeping $a_2 - a_1$ large (large Δ at the same P).

The critical pressure analysis of this structure proceeds as above. The load-deflection behavior is considerably more complicated; however, an approximate solution can be obtained by considering the case of residual stress dominated behavior. In this case, the membrane equation[15] can be integrated using annular boundary conditions (zero film deflection at a_1 and a_2) to yield:

$$w(r) = \frac{p}{4\sigma_0 t}\left[1 - \frac{r^2}{a_2^2} + \frac{\alpha^2}{a_2^2}\ln\left(\frac{r}{a_2}\right)\right] \tag{15}$$

where p is the (uniform) pressure on the annular film and α^2 is a "logarithmic square mean" defined by:

$$\alpha^2 = \frac{a_2^2 - a_1^2}{\ln(a_2/a_1)} \tag{16}$$

Integrating Eq. (15) over the area of the deflected annulus yields the volume Δ. Since the load-deflection relation is assumed to be linear (stress-dominated), the strain energy and complimentary strain energy are equal and are given by:

$$V = V^* = 0.5P\Delta \tag{17}$$

Applying Eq. (5a) yields the critical pressure relationship:

$$\gamma_a = \frac{p_c^2 a_1^2}{32\sigma_0 t}\left[\frac{\beta^2 - 1}{\ln \beta} - 2\right]^2 \tag{18}$$

β is defined as the annular ratio a_2/a_1. Although approximate, it is instructive to examine the limiting behavior of Eq. (18). As β approaches unity, γ_a approaches zero (since no film is exposed, no adhesion can be measured even at infinite pressure), while as β approaches infinity, γ_a becomes large for any pressure p_c. Thus, it is theoretically possible to measure large γ_a values at pressures which do not exceed the ultimate tensile stress of the film by making the center island sufficiently small.

For example, consider the same film as above, this time adhered to a substrate with a value of γ_a of $100\,\mathrm{J/m^2}$. Also assume that the maximum pressure we wish to subject the film to is 10 psi (69×10^3 Pa). From Eq. (18), the value of a_1 necessary to achieve peel of this system is 850 μm, or an island diameter of 1.7 mm. Using an island blister, the tensile strength limit of the above film can be overcome geometrically. Experimental confirmation of the utility of the island structure is being reported separately.

CONCLUSIONS

It has been demonstrated for the classical blister test that the effect of the residual stress in the film may drastically affect the critical pressure–work of adhesion relationship used to analyze adhesion data. In addition, a blister structure for overcoming the tensile strength limit of peeling thin films, the annular or "island" structure, has been proposed. The cirtical pressure—work of adhesion relationship has been derived for this structure using the methods outlined for the classical blister test. The resulting equation indicates that peel of thin films can be initiated at any conveniently low pressure by varying the geometric factor β, the ratio of the outer to inner radii of the film annulus.

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Appendix

The explicit dependence of the various constants of the adhesion model on Poisson's ratio is given below. The constant k_3 (representing the residual stress component) does not have a Poisson's ratio dependence, while the constant k_2 (representing the bending component) is related to the plate flexural rigidity, thereby having a known Poisson's ratio dependence. Thus, only k_1 (and therefore c_1) has a Poisson's ratio dependence to be determined. The k_1 dependence for each of the three cases of interest is given by the following equations:

Square membrane:

$$k_1 = \frac{\pi^6}{128(1-v^2)}\left[\frac{5}{16} - \frac{4(5-3v)^2}{9\pi^2(9-v)+64(1+v)}\right]\frac{Et}{a^4}$$

Clamped circular plate:

$$k_1 = \frac{3}{1-v^2}[1.221 - 7.848\times 10^{-3}(6-v)(1+11v)$$
$$-8.965\times 10^{-3}(23-41v)(1.98-v)]\frac{Et}{a^4}$$

Circular membrane:

$$k_1 = \frac{8}{3(1-v)}\frac{Et}{a^4}$$

and the relationship between k_1 and c_1 is given by:

$$c_1 = k_1\frac{a^{10}d^3}{\Delta^3}.$$

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