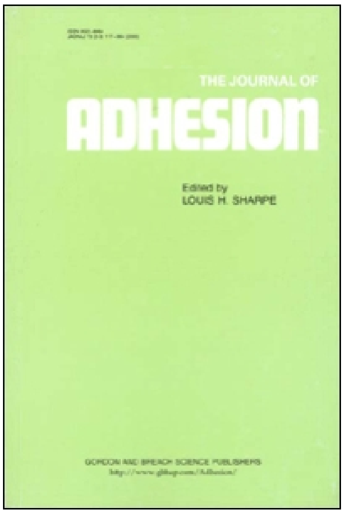

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The Constrained Blister—A Nearly Constant Strain Energy Release Rate Test for Adhesives

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The Constrained Blister—A Nearly Constant Strain Energy Release Rate Test for Adhesives

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A modification of the blister test permits nearly constant strain energy release rate testing of adhesive bonds. By constraining the deformation of the blister, a promising device for automated evaluation of critical strain energy release rates can be obtained. The procedure is especially amenable to viscoelastic and environmentally-assisted debonding processes. Preliminary experimental evaluation of the time-dependent adhesive fracture toughness of a tape product is included.

KEY WORDS Constrained blister test; constant strain energy release rate; automatic test device; adhesive fracture test, PSA tape; experimental study; debonding processes.

INTRODUCTION

Over the years, a large number of test geometries have been devised for evaluating the properties of bulk and *in situ* adhesives. Because typical adhesive joints contain only a very small amount of adhesive, there is some question as to whether properties measured on bulk adhesive samples are meaningful in estimating the behavior of *in situ* adhesive material in a practical joint. For example, using reflection of ultrasonic pulses, Knollman[1] has shown that the properties of an adhesive are dependent on the proximity to the adherend. These and other results are encouraging many investigators to develop techniques for measuring the properties of the *in situ* adhesive.

ASTM[2] has standardized a number of strength tests for bonded joints and yet most of these tests have very complex stress states. Although these tests offer standard ways to compare different adhesive systems or surface treatments, they do not yield properties which are very appropriate from a design standpoint. A number of tests have been advanced with reportedly uniform stress states throughout, but careful analysis often reveals regions with highly non-uniform stress distributions and steep gradients.

The use of fracture mechanics has provided a more rational basis for the design of structural components and a number of tests have been developed for measuring these properties. These include such tests as the double cantilever beam (DCB) test originally developed by Mostovoy and Ripling,[3] the cone pull-out test developed by Anderson *et al.*,[4] and the blister test originally employed for paints by Dannenberg,[5] later adapted to structural adhesives by Williams.[6] An extension of the blister test to the case of a very thin adherend has been made by Gent and Lewandowski,[7] and a novel "island" blister has been proposed by Allen and Senturia[8] to permit testing films of microscopic dimensions. Each of these tests has certain advantages and disadvantages and most may be modified to provide some degree of mixture between mode I, II, and III crack growth.

The DCB and its various modifications have perhaps been more extensively used than other fracture tests. While appropriate for tests in inert environments, diffusion of species from a hostile environment along the sides of the specimens may preclude the use of this test for long term exposure conditions.[9,10] Lefebvre *et al.*[9] found that consistent environmentally-degraded fracture energies from DCB specimens could not be obtained while the environment was ingressing the bond. Instead, the specimen had to be conditioned long enough to achieve a uniform amount of degradation before testing could commence. Although seldom used quantitatively to evaluate fracture toughness, the Boeing wedge test, a form of DCB specimen, has become almost an industry standard for evaluation of the durability of surface treatments. One problem with interpreting the data from this test is the increasing effect of diffusion as one goes down the length of the specimen.

The blister specimen offers an attractive alternative for environmental exposure because the diffusion occurs nearly perpendicular to the debond front. Penetration from the sides does not present a problem as it does for beam type specimens. Also, because of the axisymmetric nature of the blister specimen, the non-uniformity of the stress field along the debond front is much less than for a finite-width specimen. One of the most difficult problems associated with the blister specimen is the determination of the debond radius. Jones[11] has implemented an ingenious technique utilizing two finely tuned valves at the inlet and outlet to identify increments of crack growth. A small increment in crack length increases the compliance of the blister specimen and results in a measurable drop in pressure. Anderson[12] has employed acoustic emissions to detect and locate debond initiation. Such techniques give crack initiation, but do not always provide adequate information about actual debond size. Ultrasonic C-scan and other non-destructive evaluation techniques can be used to determine current debond size, but these techniques can be somewhat cumbersome. The benefit of precise optical measurements is available for certain cases including the situation where at least one adherend is transparent. Particularly interesting results using interferometry for this problem have been discussed by Liechti.[13]

Measurement of the debond size is important for two reasons—the determination of the increments in crack growth and the evaluation of the debond radius for calculation of the strain energy release rate. Anderson *et al.*[14] discuss closed form and numerical solutions for the strain energy release rate and have identified regions of applicability for formulae for a penny-shaped crack between two semi-infinite media and for plate theory. If the deformations are large compared to the blister thickness, the analysis must be further modified to include membrane effects as well.[7] For illustration purposes, we consider the simplest case where thin plate assumptions with small deformations are applicable. The closed form solution is:

$$G = \frac{3(1-\nu^2)}{32Et^3}p^2a^4 \tag{1}$$

where G is the strain energy release rate, ν, E, and t are the Poisson's ratio, Young's modulus, and thickness of the blister adherend, p is the applied pressure, and a is the debond radius. (Alternate formulations appropriate when the above assumptions are not applicable are given in Refs. 7, 14, and 15.) Because radius appears to the fourth power, small errors in measuring the debond will result in significant errors in estimating G. Because of the difficulties in measuring the debond for opaque adherends, a modified test with nearly constant G would expedite experimental evaluation of adhesive toughness.

DISCUSSION

A constant G test results when the compliance of a specimen increases linearly with crack area. Presumably, one could made the blister adherend conically tapered to resemble the tapered DCB specimen to achieve relatively constant G, but the machining would be difficult, and calculations reveal that the technique is not very practical for certain material systems. On the other hand, one can place a flat constraint above the blister to limit its displacement as indicated in Figure 1.

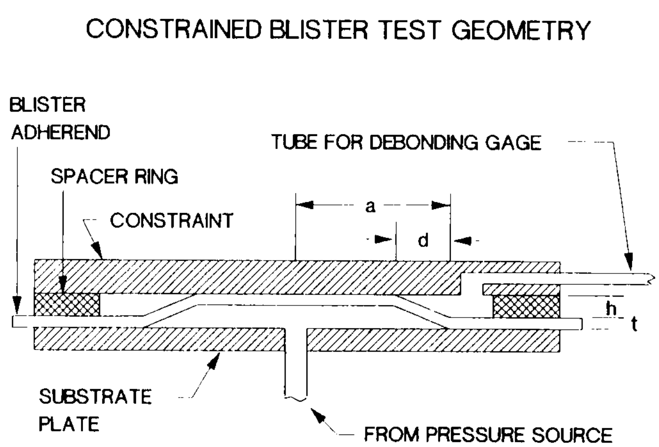

FIGURE 1 Schematic diagram of the constrained blister specimen.

Such a specimen configuration has been proposed independently by Dillard and Chang[16] and by Napolitano *et al.*[17] Simple geometry reveals that if one neglects the intermediate region where the blister is suspended between the substrate and the constraining plate, the volume under the blister will increase linearly with debond area. This implies that for a constant pressure loading mode, the work done on the system is simply the increment in debond area multiplied by the distance the blister travels before reaching the constraint. These simplistic thoughts suggest the constant-G nature of the test geometry, but a more detailed preliminary analysis will now be provided to confirm and clarify the hypothesis.

Several energy balance formalisms have been proposed for determining strain energy release rates for adhesive bonds. Our approach here is based on the classical energy conservation approach where localized viscoelastic and plastic deformations in the vicinity of the crack tip are included in the critical strain energy release rate, $G_c = G_c(da/dt)$, making it a function of debond rate. Another common approach is to assume that G_c is an intrinsic fracture resistance of the material, and to lump both near- and far-field viscoelastic behavior into a separate term. Our choice to include this near-field energy dissipation in the G_c term provides expediency. This has been discussed by Knauss[18] and Williams,[19] and used by Anderson, *et al.*[14] and others.[20,21] This is a reasonable approach since near field dissipation cannot readily be separated from an "inherent" surface energy anyway. When debonding occurs,

$$G_c \delta A = \delta W - \delta U - \delta Z \tag{2}$$

where G_c is the critical value of strain energy release rate which may be a function of debond rate and environment,

δA is the variation in debond area,
δW is the variation in external work done on the system,
δU is the variation in stored elastic energy, and
δZ is the variation in energy dissipated in regions away from the vicinity of the debond tip.

For the moment, we will make the assumption that the variation in stored energy is negligible. This will be demonstrated experimentally later in the paper, and has been further substantiated numerically[22] and analytically.[23] We will also choose to neglect the δZ term by assuming that there is little far field viscoelastic dissipation in the blister adherend. We will also neglect energy dissipated at the interface between the blister and the constraint. This effectively implies that there is no slipping between the blister and the constraint, or that the interface is perfectly lubricated. Experimental and numerical[22] observations tend to support the validity of the former assumption. The above claims allow one to write

$$G_c \delta A = \delta W = p \delta V \tag{3}$$

where p is the applied pressure and δV is the variation in volume under the blister.

To approximate this variation in volume, we assume that the suspended region of the blister is linear. While this assumption is not consistent with the bending of the blister adherend, it is used only to calculate the volume under this small suspended region and not to obtain bending energy. As such, this choice gives an accurate value for the volume under the blister and does not introduce errors elsewhere.[22] By using the Theorem of Pappus, we find the volume to be

$$V = \pi h\left(a^2 - ad + \frac{d^2}{3}\right) \tag{4}$$

Taking the variation of the volume and substituting into Eq. (3), we obtain that the strain energy release rate is simply the product of the pressure, the constraint height, and a correction factor, q.

$$G_c = phq \tag{5}$$

For the case of the linear detachment assumption, q is given by:

$$q = \left(1 - \frac{d}{2a}\right) + \left(\frac{d}{3a} - \frac{1}{2}\right)\frac{\partial d}{\partial a} \tag{6}$$

Since the detachment distance, d, changes only slightly as the debond grows, the partial derivative appears to be quite negligible for the cases examined so far. One sees, however, that the relative sizes of the debond radius and the detached zone will affect the accuracy of the constant-G property. When the relative size of the detached zone is not small, there is some variation in G with debond distance. We have found[22] that this variation may be accurately estimated by the use of Eq. (6). The variation of q with the ratio a/d is given in Figure 2. To date, most of our tests have been conducted within the total range of $2 < a/d < 20$, although the range for any given single test is often somewhat less. The correction factors

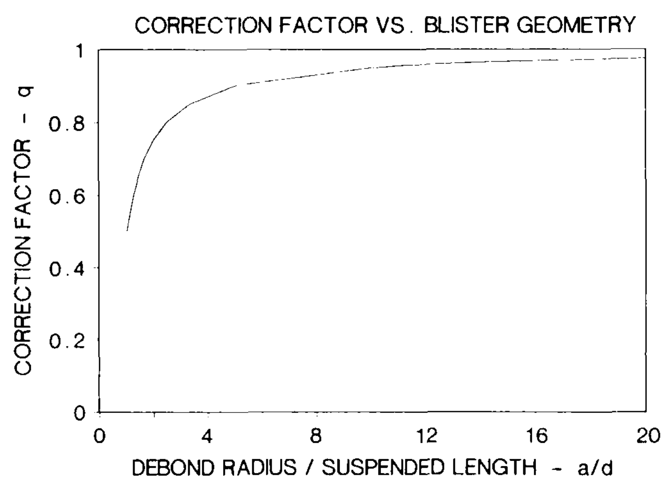

FIGURE 2 The correction factor, q, as a function of the ratio of debond radius to suspended region length.

are even closer to unity if the detached region has some curvature induced by the pressure, as is typically observed with a flexible adherend. We, therefore, conclude that although the test geometry is not strictly constant G, deviations are small and can easily and accurately be estimated. For cases where the constraint is opaque and d cannot be measured directly, it can be estimated numerically[22] or analytically.[23]

We should point out here that although Eq. (5) is similar to the form obtained by Gent and Lewandowski[7] for an unconstrained membrane specimen, the forms are not directly related. They obtained

$$G = 0.65py \tag{7}$$

where y is the free height of the membrane. Since y is not a constant, but rather a function of the applied pressure, debond radius, and the modulus of the membrane, Eqs. (5) and (7) are seen to be quite different in their formulation. The problems are similar in the sense that they both represent non-linear traction—displacement systems, however, and lead us to draw some special observations.

For linear systems, the well-known equation for G is given as

$$G = \frac{1}{2}p^2\frac{\partial C}{\partial A} \tag{8}$$

where p is the generalized traction and C is the compliance. It can easily be shown that this is just the linear form of a more general expression

$$G = \frac{n}{n+1}p^{((n+1)/n)}\frac{\partial C}{\partial A} \tag{9}$$

where n is the exponent for the constitutive equation

$$p = (V/C)^n \tag{10}$$

and V is the generalized displacement. Figure 3 illustrates the pressure—

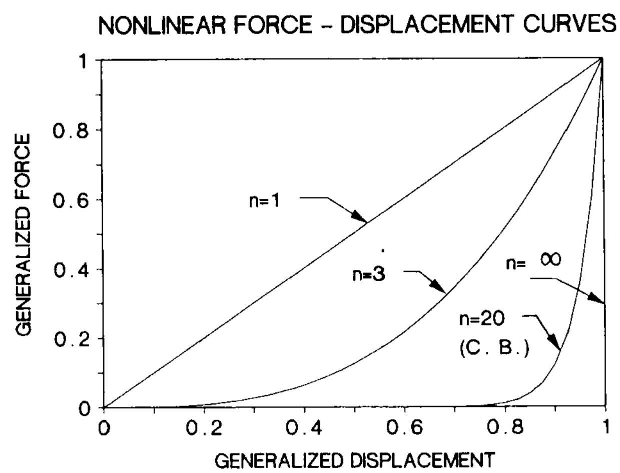

FIGURE 3 Nonlinear force-displacement curves for several test geometries.

displacement relations for the linear case (which corresponds to plate theory and Eqn. 1), the cubic case (which corresponds to Gent's membrane analysis in Eq. (7)), and actual and idealized cases for the constrained blister. It can also be shown that the stored energy for the general form is only $1/(n + 1)$ of the input work under constant traction cases. As n increases, the stored energy becomes negligible in comparison with the external work. This further establishes the validity of neglecting δU in Eq. (2) for the constrained blister where n is quite large.

EXPERIMENTAL SETUP

The experimental setup discussed herein was designed to test the debonding of adhesive tapes, although a large testing unit has been constructed and successfully used to measure the durability of rubber/metal bonds. The test setup, in its original and simplest configuration, is shown in Figure 4. The substrate and constraint are made of polycarbonate, facilitating visual observation. To prepare a specimen, the substrate is cleaned and dried at room temperature, and the tape, with a nominal width of 150 mm, is applied and rubbed to ensure attachment. The substrate has a hole in the center with a diameter of 6 mm. A spacer of the desired thickness is placed above the blister, and the constraint is bolted in place. The pressurizing medium is supplied at constant pressure which, for the present case, was supplied by a large reservoir. Raising and lowering the reservoir provided a convenient means of changing the pressure. Three methods were employed to measure the debonding rate for the present study, and each is described below.

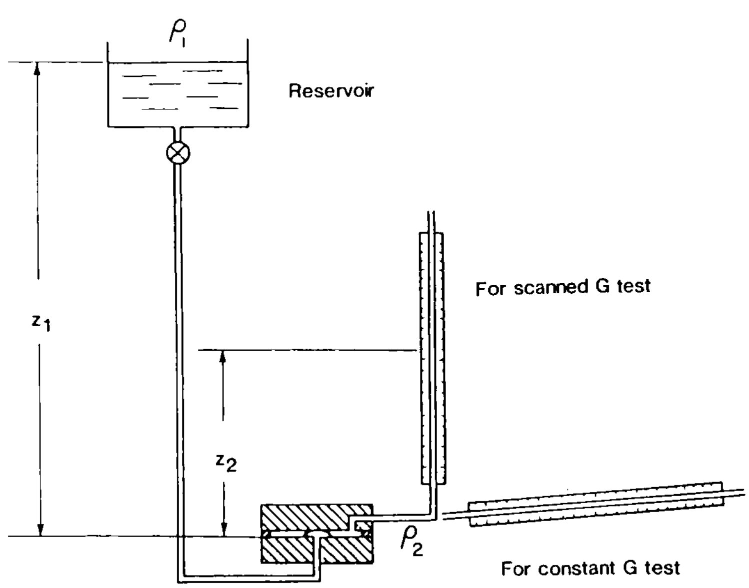

FIGURE 4 Schematic diagram of simple constrained blister test fixture.

1. Visual Measurement of Debonding Radius

Most of the data for the current study was collected by visual measurements of the debond diameter and the contact diameter. Placing a ruler on the constraint and measuring the diameter at two or three orientations gave sufficiently reliable data for the preliminary results, although an image processing system will soon be utilized to refine the approach. The suspended distance was calculated by simple subtraction of the respective radii. To get a large window of G values from one test, one may increase or decrease the reservoir height several times. To examine the reproducibility of this technique, several specimens were tested to obtain the average debond rates for each reservoir height. Since the debond radius and suspended distance were recorded for each measurement, the correction factors could easily be calculated according to Eq. (6). This approach provides the most accurate estimates of applied strain energy release rate, but does require a transparent constraint and easily observed debond radii.

2. Measurement of Displaced Volume

A second approach to measure the debond rate was based on measuring the volume displaced by the growing blister. This technique is especially useful for those constrained blisters having irregular shapes, or where an opaque constraint is required to maintain the pressure. To measure the displaced volume, one may drill a hole into the constraint to permit attachment to a debonding gage as shown in Figure 4. The upper chamber is filled with water and provides a convenient method to monitor volume displacement. If the debonding gage, which consists of a graduated glass tube, is placed vertically, the net head acting across the blister changes as the measuring fluid rises within the gage, effectively providing a scan over a given pressure or G range. On the other hand, the debonding gage may be placed nearly horizontal to maintain a nearly-constant pressure test. The effective head across the blister may be calculated from the known heads of the liquid in the reservoir and in the debonding gage according to

$$p = g(\rho_1 z_1 - \rho_2 z_2) \tag{11}$$

where the heads are shown in Figure 4. This even allows one to use room air at atmospheric pressure as the pressurizing medium by placing the debond gage below the specimen, thereby greatly simplifying the pressurization.

To plot the debonding area rate *vs.* G, we assumed that the rate of debonding is equivalent to the rate of volume displacement divided by the height of the constraint. This approximation becomes quite valid when the debond radius is large compared to the suspended distance. Equation (11) may also be rearranged to determine accurately the debond area in terms of displaced volume and the suspended length, d. Because the suspended length remains nearly constant from experimental observations and from numerical[22] and theoretical predictions,[23] it is also relatively easy to calculate the approximate correction factor, q, by using Eq. (11) and a measured or predicted value of d.

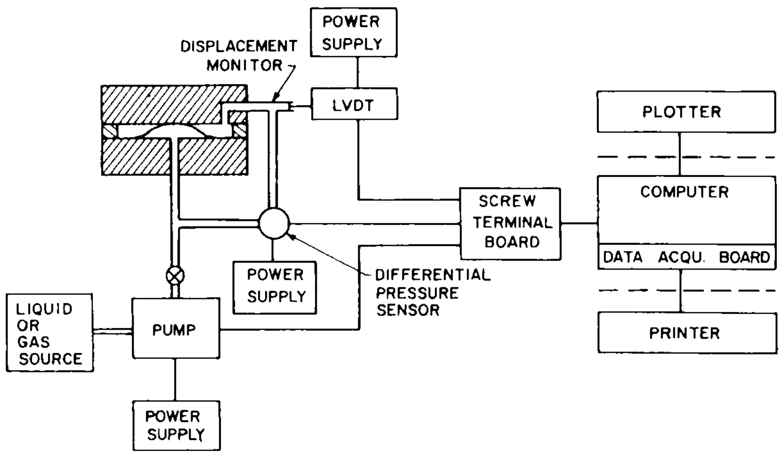

FIGURE 5 Schematic diagram of an automated constrained blister test fixture.

3. Automated Measurement of the Variation of Volume and Pressure

The experimental setup has also been modified to collect data automatically using a computer. A differential pressure transducer was mounted between the pressurizing fluid and the displaced fluid to measure accurately the net pressure even when debonding occurs very rapidly or when the debonding gage offers a non-negligible pressure. The debonding gage here consisted of a precision syringe displacing an LVDT to give continuous readings of displacement. The data were collected and displayed in real time. A schematic diagram of the automated setup is shown in Figure 5. Although this approach provides for continuous data collection, one still needs to estimate the correction factor, q, to calculate accurate values of G.

EXPERIMENTAL RESULTS

Two adhesive tapes were used in this study and were courteously supplied by the 3M Corp. Their properties are summarized in Table I. The substrate was polycarbonate which was cleaned with alcohol and dried at room temperature between each use. We used distilled water as the pressurizing medium. The large width of the tape required special care for uniform application onto the substrate.

TABLE I

Tape	Backing	Adhesive	Thickness	Modulus (MPa)
A	Polyester	Rubber	0.107 mm	743
B	Vinyl	Acrylic	0.18 mm	7.93

X-head speed = 1 in/min.

A small blister formed as soon as the reservoir valve was opened. Recording of experimental readings commenced when the blister touched the constraint. Data could have been collected prior to contact and used in conjunction with Gent's approach.[7] Each experiment was stopped when the constrained blister reached the edge of the spacer during testing. The adhesive tape was stretched in one direction slightly as it was applied to minimize the formation of wrinkles in the tape. Although the blister started out circular, there was a slight tendency for the blister to grow fastest in the stretch direction, since the membrane stresses were larger in this direction. The deviation from a circular shape was only on the order of 5%, and was not believed to be significant. A photograph of a growing blister is shown in Figure 6. It is interesting to note that because the pressure acts through nearly the same increment in volume regardless of where the increment of area occurs, debonding in any direction is almost equally likely to occur. This implies that the impetus for symmetry, which is clearly seen in unconstrained blister tests, is significantly reduced with this nearly-constant-G test. For stronger adhesives, this behavior could result in rather arbitrary debond patterns which could introduce errors into the technique.

A tape A specimen was tested using a spacer thickness of 3.175 mm and at a constant pressure ($p = 28.3$ kPa.) The experimental results of the debonding radius and suspended distance are shown in Figure 7. The debonding radius increases linearly with time and the suspended distance decreases only 10% during the same time period. Since a and d were measured, the correction factor could be calculated and is about 0.63 when the blister first touched the constraint. Similarly, the correction factor is about 0.80 at the end of the test. Thus the difference of the applied G is 17%. Despite this small increase in G, there does not seem to be any significant change in debond rate.

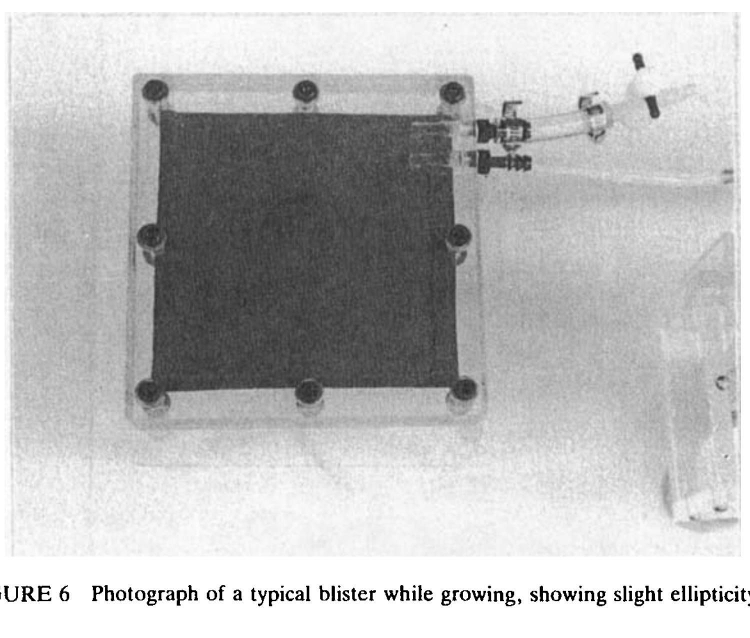

FIGURE 6 Photograph of a typical blister while growing, showing slight ellipticity.

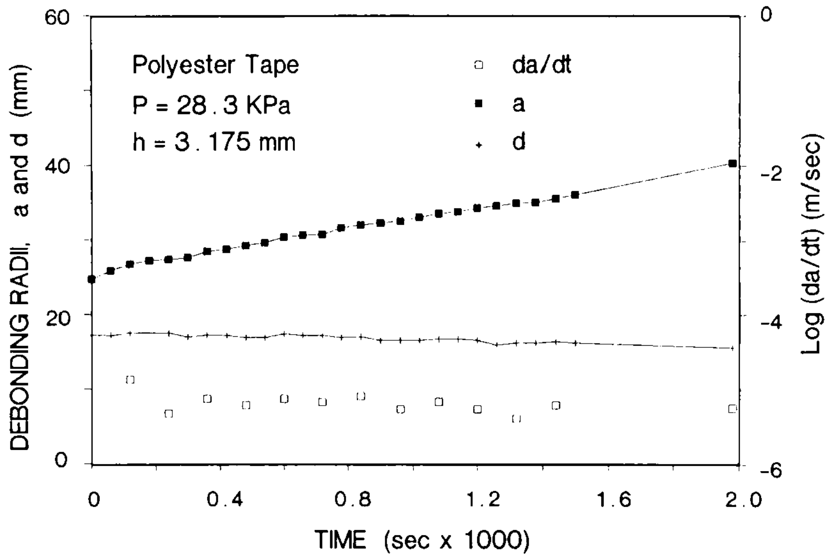

FIGURE 7 The debonding radius and suspended distance *vs.* time.

To better understand the effect of G on debonding rate, we constructed a graph with a larger range of G and debonding rate for tape A by testing one specimen under several different reservoir heights. Multiple tests were conducted to obtain the average values. To examine the validity of the constrained blister test (CBT), we also conducted the free membrane blister test (BT) by measuring the blister height and radius. A series of G values were obtained by employing Gent's equation, and the corresponding debonding rates were also calculated. Several standard peel tests with different take-off angles were also conducted to verify our results. The results of all three techniques, within the range observed on the blister tests, are in good agreement with each other and are shown in Figure 8. The fracture mode in each case was a mixture of mode I and II, but no attempt was made to separate these components.

Instead of measuring the debond radius and suspended distance, tape B was tested by monitoring the volume displacement and, in turn, the debond area was obtained. Tape B is flexible and quite time dependent. The suspended distance is small, even at the beginning of the test as shown in Figure 6. The approximate q value at this stage is about 0.92, and increased to 0.98. Thus the variation of G values for these tests under constant pressure is only about 6%, which is smaller than that of the stiffer tape A.

Based on the results from tape A, one would expect that the debond rate would be constant for tape B under a constant pressure level. Instead, according to our experimental results, the area debonding rate (dA/dt) is essentially constant, as shown in Figure 9. This implies that the radial debonding rate decreases as the debond grows. It is believed that this anomalous behavior arises because of the viscoelastic dissipation in tape B. This term was not included in the

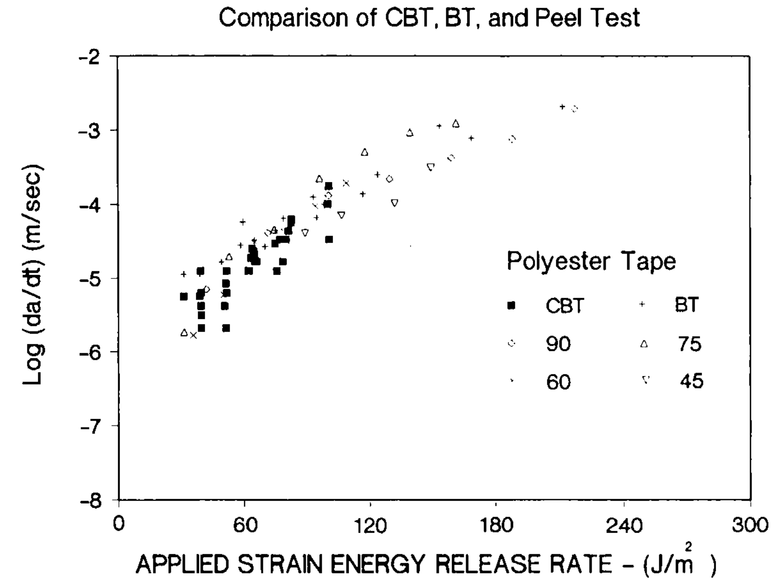

FIGURE 8 The comparison of the constrained blister test, blister test, and peel test results at the indicated take-off angles.

energy balance, and would not be negligible for this tape material. The significant time dependence is illustrated by the creep test results shown in Figure 10 for a strip of tape B.

An important advantage of the constrained blister test is that desired G values can be obtained by any combination of p and h, as indicated by Eq. (5). Using two spacer thicknesses (3.2 mm (0.125 in) and 5.6 mm (0.219 in)) and holding at

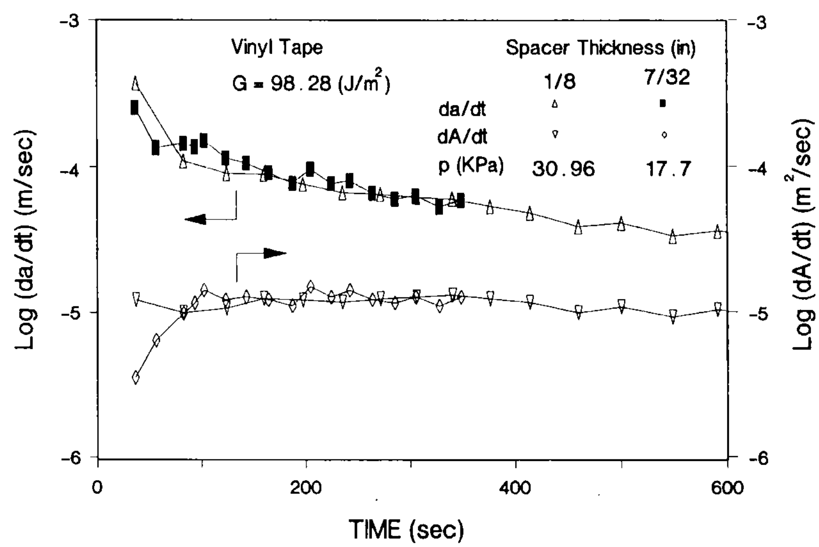

FIGURE 9 Debond rate for different spacer thicknesses at constant pressure.

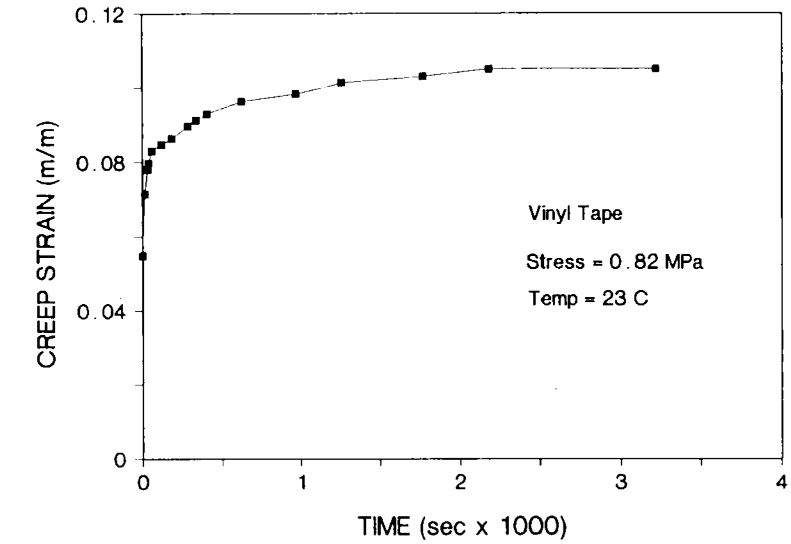

FIGURE 10 Creep test results for tape B.

constant pressures selected to achieve equivalent strain energy release rates of 98 J/m^2, the debond rates are constant with time and superpose very well as seen in Figure 9. Thus one can select a pressure and constraint height combination to achieve the desired strain energy release rate. In practice it is preferable to keep the height as small as possible to minimize the need for the correction. In doing so, however, one must not allow the pressures to become so high that they

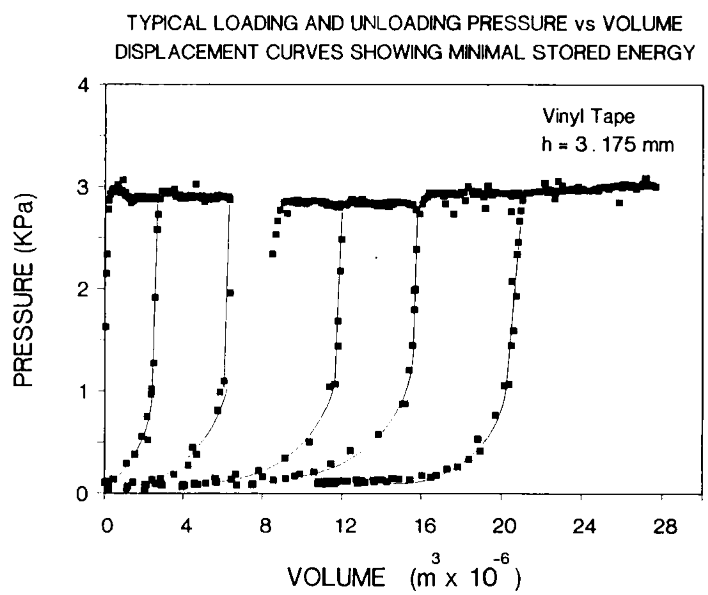

FIGURE 11 Loading and unloading curves for a constrained blister showing stored energy.

rupture the blister. As suggested by Napolitano *et al.*,[17] however, the constraint significantly reduces the membrane stresses and allows testing at pressures much higher than would be possible with a free membrane.

A crucial assumption for the constrained blister concept to be valid is that the stored energy does not increase significantly as the blister grows. If it can be shown that stored energy is small compared to the total work of debonding, this assumption is acceptable. In an effort to estimate the stored energy in the system, a blister specimen was depressurized at intermediate debond radii. Figure 11 illustrates the loading and unloading curves. These results indicate that the stored energy always represents a small fraction of the total work done, as suggested previously by the analytical predictions.

CONCLUSIONS

A modification to the blister test permits nearly-constant-G tests to be conveniently performed on adhesive bonds. The test is particularly well suited to measuring the fracture toughness of adhesive bonds while they are exposed to inert or active environments. A simple debond measuring technique permits easy evaluation of debonding to record the time-dependent nature of the fracture process. Although a detailed analysis is not reported herein, preliminary analysis and experimental results suggest that the test has merit. The current investigation has been conducted on adhesive tapes, although the technique appears to be amenable to tests on structural adhesives as well, although there is some concern as the relative stiffness of the blister becomes too large.

Two tape systems were investigated in the current study. The polyester tape was fairly stiff and elastic. The results suggest that at a constant pressure, the radius of the blister increases linearly with time, as would be expected with a bond exhibiting a critical strain energy which is a function of the debond rate. Since debond area is proportional to the square of the debond radius, it increases in a quadratic fashion. Napolitano *et al.*[17] have obtained similar data, but have chosen to fit the data with an exponential form, rather than the parabola which would come from a constant debond rate for the radius. For the vinyl tape that we tested, we did not find that the rate of debond of the radius was constant. Instead, it decreased as the blister grew. We have attributed this behavior to the significant creep of the vinyl backing material. The area debond rate was fairly constant for this material, but we feel this is rather coincidental. Further studies are needed to include the viscoelasticity properly in the debond model for this material system.

The constraint adds a new dimension to fracture testing because it provides a means to obtain a constant G test by limiting the amount of stored energy to a very small fraction of the work done under constant load conditions. Although the method hinges on several crucial assumptions, the preliminary experimental results have tended to substantiate conformation to these requirements. Additional testing and analysis are proceeding to evaluate the potential applicability of the technique to a wide variety of testing situations.

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