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New and Improved Tests for Adhesion†

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Three new methods are discussed for measuring the work G_a required to detach unit area of an adhering material from a substrate. The first is a simple modification of the Outwater double-torsion test for long rectangular plates, bonded together. This method is suitable for evaluating aluminum-epoxy bonds, for example, or the transverse strength of fibrous composites. The second is a pull-off test for long strips adhering to a rigid surface. It seems suitable for adhesive tapes and laminates. The third is a reconsideration of the “blister” test for films and coatings, in which a circular debond at the interface is made to grow by internal pressure. The relation obtained between pull-off force F for a strip, or blow-off pressure P for a layer, takes the unusual form:

$$F^4 \text{ (or } P^4) \propto KG_a^3$$

where K is the tensile stiffness of the detaching layer. This dependence arises from the non-linear (cubic) relation between load or pressure and deflection in these configurations. Nevertheless, the product $F\theta$, where θ is the angle of detachment of a strip, or Py , where y is the height of a “blister”, give direct measures of the strength of adhesion G_a , independent of the stiffness of the adhering material and of the extent of detachment.

KEY WORDS Blister test; detachment; fracture mechanics; pull-off; test methods; torsion.

1 INTRODUCTION

Ideally, a test method for adhesion should have the following features. First, it should employ simple specimens. Secondly, the

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failure force should remain constant, at least in principle, while the line of detachment is driven forwards over long distances, so that an average strength of adhesion can be readily determined. And, finally, the fracture energy G_a should be obtained directly in terms of the specimen dimensions, its stiffness during loading and the critical load at which the process of detachment takes place. No other measurements are needed then to determine the energy G_a of separation per unit of bonded area.

Three such test methods are reviewed here. The first is particularly suitable for relatively stiff adhesives and adherends while the other two can be used with materials having a wide range of stiffness.

2 THE OUTWATER TORSION TEST^{1,2}

A sketch of a suitable bonded specimen is given in Figure 1. It consists of two long rectangular plates, bonded together along one edge. An initial crack of length c_0 is made between them. The specimen is then clamped, as shown in Figure 2, and subjected to a steadily increasing angular rotation θ of one arm with respect to the other. The corresponding torque M is measured by means of a long rigid moment arm, as shown in Figure 2.

At a critical value of the applied torque, denoted M_c , the crack will advance. At this point elastic energy stored in the twisted arms of the specimen begins to be expended in fracture. Assuming that the arms are linearly-elastic in torsion and that the torsional stiffness M/θ is inversely proportional to the length c of the arms, we can deduce that the fracture energy G_a is given by¹

$$G_a = M_c^2/2kt \quad (1)$$

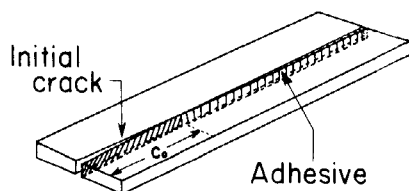


FIGURE 1 Sketch of specimen for double-torsion test.

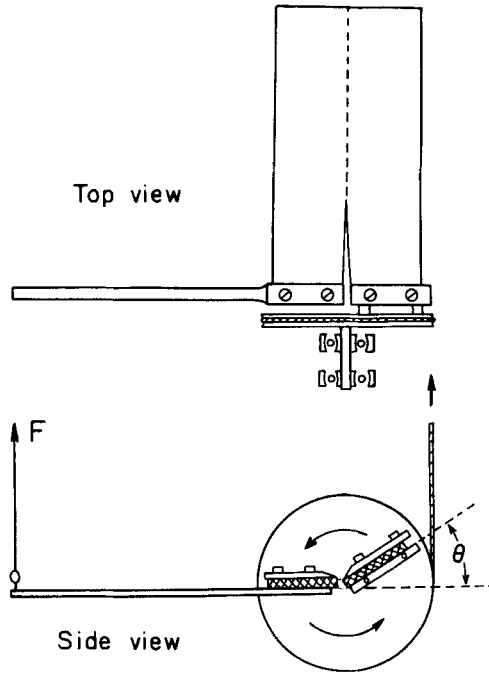


FIGURE 2 Method of applying large rotations θ and measuring the corresponding torque².

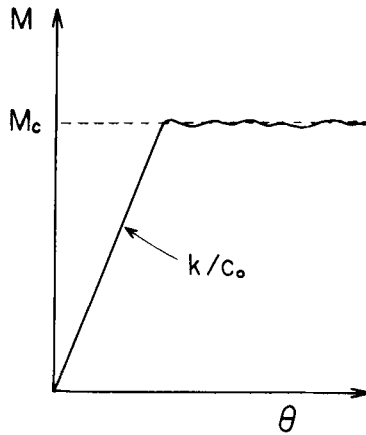


FIGURE 3 Relation between applied torque M and pulley rotation θ .

where

$$k = Mc/\theta. \quad (2)$$

k denotes the torsional stiffness of the specimen for a crack of unit length and t is the thickness of the specimen. The value of k can be obtained from the experimental relation between torque M and rotation θ up to the onset of fracture, Figure 3, and the mean value of M_c can be measured thereafter, over long distances for long test pieces. Thus, the mean fracture energy G_a can be measured with some confidence, using a single specimen of simple design.

3 PULL-OFF TEST FOR ADHERING STRIPS³

This simple test arrangement is shown in Figure 4. An adhesive strip of width w is pulled away from a rigid substrate at an angle θ by a force F . Work is done both in stretching the strip as it detaches and in fracturing the interfacial bond. Assuming that the strip is linearly-elastic and the angle θ is small, the relation between F and θ is as follows:

$$F = K\theta^3 \quad (3)$$

where K is the tensile stiffness coefficient of the strip (force per unit of tensile strain). In terms of the fracture energy G_a ,³

$$wG_a = (3/8)(F^4/K)^{1/3}. \quad (4)$$

Thus, if measurements of the pull-off force F only are made, an independent measurement of the stiffness of the strip is required in order to determine the strength of adhesion. However, a simple relation is obtained in terms of the product of the force F and detachment angle θ , both of which remain constant, at least in

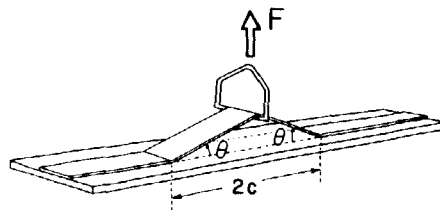


FIGURE 4 Pull-off test for an adhering strip³.

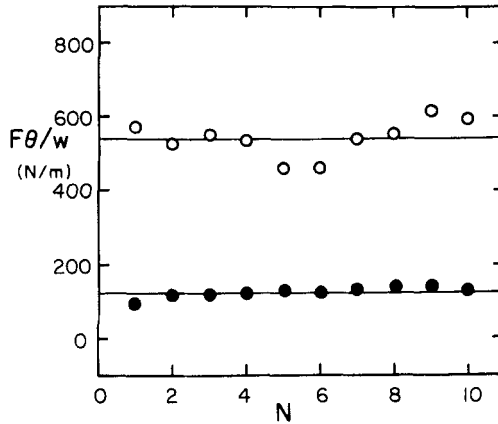


FIGURE 5 Product of pull-off force F and angle θ plotted against the number N of layers of an adhesive tape plied up and pulled off together. Open symbols, detaching from glass; filled symbols, detaching from Teflon³.

principle, during continued detachment:³

$$G_a = 3F\theta/8w \quad (5)$$

Thus, the mean value of the fracture energy can be obtained from simple measurements on a single specimen.

Values of the product $F\theta/w$ are shown in Figure 5 for an adhesive tape detaching from glass and Teflon substrates. A number N of layers were adhered one on top of another to give a composite tape having N times the stiffness of a single layer, but the same level of adhesion. Correspondingly, the pull-off force F was greater and the angle θ was smaller than for a single layer. But the product $F\theta$ was quite independent of N , as the theory predicts. It thus provides a measure of the strength of adhesion G_a , independent of the stiffness of the tape. (G_a is, of course, different for the different substrates, as would be expected.)

4 BLOW-OFF ("BLISTER") TEST FOR ADHERING LAYERS⁴⁻⁷

This test, shown schematically in Figure 6, was proposed by Dannenberg⁴ and applied by Williams⁵ and Andrews and Stevenson⁶ to the study of adhesion. But the latter authors focussed

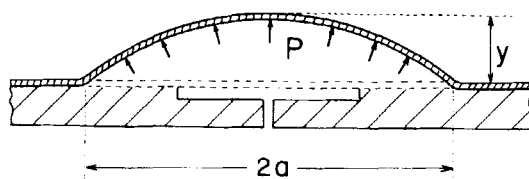


FIGURE 6 Blister test for an adhering layer.

on the bending deformation of a relatively thick layer, with a blister of relatively small radius a underneath it. When the blister is large in radius, and the layer is relatively thin, then the principal deformation is a biaxial stretching of the layer, rather than bending. In this case the relation between inflating pressure P and height y of the center of the blister is given by

$$P = 4.75K'y^3/a^4 \quad (6)$$

for small degrees of inflation of the layer, again assumed to be linearly-elastic, where K' denotes the tensile stiffness coefficient for a strip of unit width; $K' = Et$, where E is the effective tensile (Young's) modulus and t is the layer thickness.

At a critical pressure the blister will grow in size by further detachment. By considering an energy balance, in which the work of inflation is expended partly in stretching a larger portion of the layer and partly in detaching it, we deduce that⁷

$$G_a = 0.39(P^4 a^4 / K')^{1/3} \quad (7)$$

or

$$G_a = 0.65Py. \quad (8)$$

Although in this case the pressure required to cause detachment does not stay constant as the blister increases in radius, but decreases continuously, nevertheless the product of the momentary pressure P and corresponding blister height y gives a direct measure of the characteristic fracture energy G_a for the adhesive bond.

Measured values of P and y are shown in Figure 7 for a layer of a polypropylene-backed packing tape adhering to a Plexiglas substrate, with an initially debonded circular patch of radius $a = 25$ mm. On inflation, the pressure increased in proportion to y^3 , in agreement with Eq. (6), until further debonding started at the edge

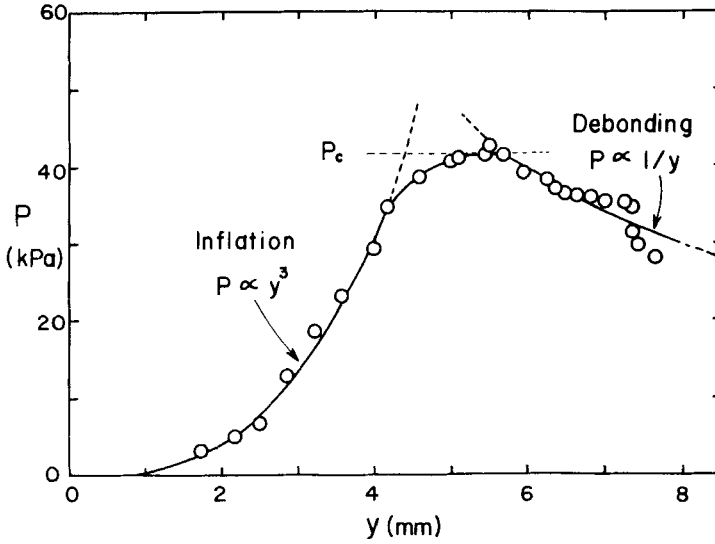


FIGURE 7 Blow-off pressure P plotted against the height y of the blister for a polypropylene-backed packing tape. P_c denotes the pressure at which further detachment started at the edge of the initial debond⁷.

of the initial circular debond. Thereafter, as the blister propagated, the pressure decreased in inverse proportion to the blister height y , in accordance with Eq. (8).

Thus, even though the blow-off pressure is not constant, it can be employed with the corresponding blister height to obtain mean values of the strength of adhesion over large areas of the interface. And no other parameters are required.

5 LIMITATIONS

The first method discussed above, involving the torsion of rectangular plates, is only applicable to materials which deform elastically up to the debonding stress. They must not undergo plastic yielding or flow. Also, they must not be so soft that the plates would twist through angles greater than 180° before detaching.

The other methods require, at least for the simple relations given in Eqs. 5 and 8 to hold, that the imposed deflections be small. In

practice, this restriction is not a severe one. Values of pull-off angle of up to 25° , and blister height approaching the magnitude of the blister radius, yield results in good agreement with the theoretical relations based on the assumption that the deflections are small.^{3,7} But, again, plastic yielding or flow of the adhering strip or layer would invalidate the analyses, which require linear relations to hold between stress and strain. Small departures from linearity seem to be acceptable, however, yielding results for G_a that are approximately correct. And it is noteworthy that reversibility of the stress-strain relations is not required at all, because the deformations remain constant or increase as debonding proceeds.

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