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NEW AND IMPROVED TESTS FOR ADHESION

by

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Three methods are discussed for measuring the work $\Delta W$ required to break a bond: Outwater's double-torsion test for long plates, bonded together; a pull-off test for an elastic strip adhering to a rigid surface; and a "blister" test for an adhering elastic layer. The pull-off force $F$...
or blow-off pressure $P$ is given by: $F_3^4$ (or $P_4^4$) $K G_3^3$ where $K$ is the tensile stiffness of the detaching layer. This unusual dependence arises from the non-linear (cubic) relation between force or pressure and pull-off angle $\Theta$ or blister height $y$. However, the products $F_3$ and $P_3$ give direct measures of $G_3$, independent of stiffness and the extent of detachment.
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SUMMARY

Three methods are discussed for measuring the work \( G_a \) required to break a bond: Outwater's double-torsion test for long plates, bonded together; a pull-off test for an elastic strip adhering to a rigid surface; and a "blister" test for an adhering elastic layer. The pull-off force \( F \) or blow-off pressure \( P \) is given by:

\[
F = K G_a^3
\]

(or similar equation involving \( P \))

where \( K \) is the tensile stiffness of the detaching layer. This unusual dependence arises from the non-linear (cubic) relation between force or pressure and pull-off angle \( \phi \) or blister height \( y \). However, the products \( F \phi \) and \( Py \) give direct measures of \( G_a \), independent of stiffness and the extent of detachment.

INTRODUCTION

Ideally, a test method for adhesion should have the following features. First, it should employ simple specimens. Secondly, the failure force should remain constant, at least in principle, while the line of detachment is driven forwards over long distances, so that the 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.

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 \( \phi \) 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 $M/\gamma$ is inversely proportional to the length $c$ of the arms, we can deduce that the fracture energy $G_a$ is given by

$$G_a = \frac{M_c^2}{2} \kappa t$$ ................................................. (1)

where

$$\kappa = \frac{M_c}{\gamma}$$ .................................................... (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 $\kappa$ can be obtained from the experimental relation between torque $M$ and rotation $\gamma$ 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.

PULL-OFF TEST FOR ADHERING STRIPS (3)

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 $\phi$ 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 $\phi$ is small, the relation between $F$ and $\phi$ is as follows:

$$F = K\phi^3$$ .................................................. (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),

$$wG_a = \frac{3}{8} (F^4/K)^{1/3}$$ ................................................. (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 $\phi$, both of which remain constant, at least in principle, during continued detachment (3):

$$G_a = 3 \frac{F\phi}{8w}$$ .................................................. (5)

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

Values of the product $F\phi/w$ 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 $\phi$ was smaller than for a single layer. But the
product F 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.)

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

This test, shown schematically in Figure 6, was proposed by Dannenberg (4) and applied by Williams (5) and Andrews and Stevenson (6) to the study of adhesion. But the latter authors focussed 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

$$P = 4.75 K' y^3 / a^4$$ .................................................. (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; $K' = E t$, 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 that (7)

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

or

$$G_a = 0.65 Py.$$ ................................................................. (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 equation 6, until further debonding started at the edge of the initial circular debond. Thereafter, as the blister propagated, the pressure decreased in inverse proportion to the blister height $y$, in accordance with equation 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.
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 equations 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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Figure 1 Sketch of specimen for double-torsion test

Figure 2 Method of applying large rotations and measuring the corresponding torque

Figure 3 Relation between applied torque $M$ and pulley rotation $\theta$
Figure 4 Pull-off test for an adhering strip

Figure 5 Product of pull-off force $F$ and angle $\theta$ vs the number $N$ of layers of an adhesive tape pulled off together. $\circ$, from glass; $\bullet$, from Teflon
Figure 6  Blister test for an adhering layer

Figure 7  Blow-off pressure $P$ vs height $y$ of the blister for a polypropylene-backed packing tape. $P_c$ denotes the pressure at which further detachment started
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