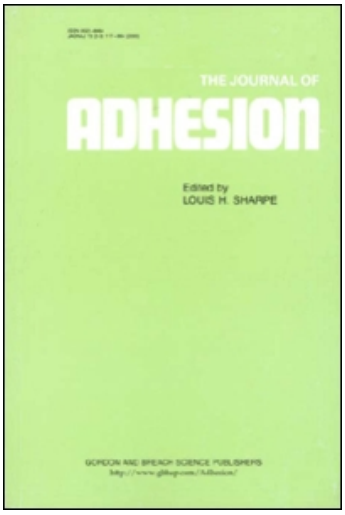

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A Technique for the Measurement of Adhesive Fracture Energy by the Blister Method

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The adhesive fracture energy, G_{IC}, of a model adhesive/adherend system, consisting of poly(methylmethacrylate) plates bonded with a cyanoacrylate adhesive, has been evaluated using the Tapered Double Cantilever Beam and Blister test geometries. A refined Blister testing technique is described which, using relatively large diameter test plates [200 mm], is capable of arresting the initial propagation of the—invariably less than naturally sharp—starter crack. This allows us to three subsequent G_C determinations for the same specimen from starter cracks of natural sharpness.

Adhesive fracture energy values determined for the model system using TDCB test pieces, 0.110 ± 0.017 kJm^{-2}, were in good agreement with those obtained for Blister specimens in which arrested cracks had been repropagated, 0.119 ± 0.013 kJm^{-2}. As is generally observed, values calculated from the initial propagation of starter cracks were somewhat higher for the TDCB specimens, 0.140 ± 0.045 kJm^{-2}. Corresponding values for the Blister tests were significantly higher with more scatter, 0.194 ± 0.074 kJm^{-2}.

It is concluded that meaningful G_{IC} data are only obtained from the Blister test if values obtained from the propagation of the initial starter crack are discarded. No matter how carefully prepared, these flaws will be less than naturally sharp. The precracking technique described here detects crack growth and, by releasing the hydraulic pressure driving it, arrests a propagating crack before catastrophic failure can take place.

KEY WORDS Poly(methylmethacrylate) (PMMA); precracking technique; crack arresting method; starter cracks of natural sharpness; cyanoacrylate adhesive; tapered double cantilever beam specimen; blister specimen.

INTRODUCTION

It has been argued from a continuum mechanics viewpoint that adhesive fracture and cohesive fractures are similar.[1] The application of linear elastic fracture mechanics principles to the fracture of an adhesively bonded joint was considered by Ripling, Mostovoy and co-workers nearly thirty years ago.[2–5] They used a compliance approach to evaluate the critical strain energy release rate, G_{IC}, for a relatively simple test geometry—an adhesively bonded double cantilever beam. The test specimen was subsequently refined to eliminate the crack length dependence in the

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expression relating G_I to the load and testing geometry resulting in a contoured double cantilever beam which, over a reasonable proportion of its length, could be approximated by a simple taper.[3] The additional effect of the deflection due to shear deformation on the overall specimen compliance was later taken into account.[4]

The Tapered Double Cantilever Beam (TDCB) has since been almost universally adopted for the measurement of fracture energy of adhesively bonded systems. It has been shown to produce a geometry-independent fracture parameter in experiments on both model and relatively low toughness adhesives[6] under static loading conditions, as well as providing a test method capable of the quantitative assessment of the degradation of adherend/adhesive interface under conditions of environmental attack.[7]

Kinloch and Shaw[8] showed, however, that with much tougher rubber-modified epoxy adhesives (G_{IC}~4 kJm^{-2}) an unambiguous, geometry-independent, material parameter was not necessarily produced by this test. In particular, the values of G_{IC} were found to increase and then decrease with adhesive thickness, reaching a peak at a bond line thickness which correlated well with the plane stress plastic zone diameter. Furthermore, it was also shown that G_{IC} increased by more than a factor of two as the specimen width was increased from 5 mm to 25 mm.[8] This was in contrast to observations made using a low toughness, conventionally-cured, epoxy adhesive where the value of G_{IC} was found to be independent of joint width and thickness.[8] One fundamental feature of this type of test is that, regardless of specimen width, the crack front intersects two free surfaces. The state of stress at the crack tip, therefore, will vary between the extreme conditions of plane stress at the surfaces and plane strain along the joint centerline.

A less widely adopted test is the Blister test, based on the method proposed by Dannenberg.[9] This was originally used to measure the adhesion between a paint layer and substrate by observing the pressure at which a blister could be made to form when mercury was introduced at the interface. This test was subsequently analysed from a fracture mechanics viewpoint by Malyshev and Salganik[10] who simplified the loading conditions to that of point loading of a thin plate. Their analysis was verified by a series of model experiments using poly(methylmethacrylate), PMMA, adherends. In order to simplify the experimental procedure, Williams[11] later suggested uniform pressure to load the crack or non-bonded region through a small aperture in a rigid substrate to which the test adherend is bonded. It has been pointed out that, as the crack front with this test geometry does not intersect a free surface, the state of stress at the crack tip is unequivocally one of plane strain.[12]

A recognised potential disadvantage of this test, however, is the experimental difficulty in producing specimens which contain the naturally sharp starter cracks required by fracture mechanics. The difficulty arises primarily as a result of the mechanics of the test geometry. A TDCB beam test specimen loaded under conditions of *displacement control* will potentially allow several determinations to be carried out from a single test specimen. Following the first increment of crack extension, the load will drop below the critical value and the crack will become self-arresting. All subsequent values measured from the same specimen can, therefore, be associated with naturally sharp cracks and the initial value discarded if signifi-

cantly higher as a result of the fracture being initiated from a blunt flaw.

With the Blister test geometry the strain energy release rate, G_I, *increases* with crack area under conditions of constant pressure. Consequently, failures tend to be catastrophic, allowing generally only a single value to be determined from each specimen. The sharpness of the starter crack, or non-bonded region, is therefore of critical importance.

Andrews and Stevenson[12] used this test to measure the adhesive fracture energy of an untoughened epoxy resin. Cast-in PTFE disks were used as starter cracks, although this approach had proved to be unsatisfactory in tests on epoxy bonded TDCBs[13] in which crack tip radii between 1 and 200 μm were produced. In an attempt to obtain more reliable data from which to determine crack velocity effects, they discarded their *lowest* values of G_{IC}.[12]

Parry and Wronski,[14] when performing Blister tests on carbon fibre reinforced plastic bonded to aluminium using a modified epoxy adhesive, also encountered problems producing naturally sharp cracks in inaccessible bondlines. Rather than interpreting variations in G_{IC} in terms of crack velocity effects, however, they concluded that any values determined from specimens containing blunt flaws would be invalid (in Fracture Mechanics terminology) and that a technique capable of precracking specimens was required with this test procedure.

In contrast to the TDCB test geometry, where a razor blade or a wedge can be used to precrack the specimen, the most reasonable chance of precracking a Blister test piece is to arrest, somehow, the initial crack propagation. The specimen, which will now contain a naturally sharp crack, can then be reloaded to provide a more realistic estimate of G_{IC}.

As the expression relating G_I to the test pressure is a function of crack size with this test geometry, it is essential to delineate the position of the arrested crack front. This presents additional experimental difficulties with associated errors and, accordingly, this communication describes the development of a testing system capable of meeting this objective using a model, transparent, adherend/adhesive system. Its experimental verification is achieved by comparison with results obtained for the same model system from TDCB test specimens. It will be the purpose of a subsequent paper to describe the application of the technique to more realistic adhesive/adherend systems—toughened and untoughened epoxy/aluminium.[15]

THEORY

The TDCB test geometry shown in Figure 1 was developed by Mostovoy, Ripling and co-workers[2–5] from earlier work on simpler cantilever beam specimens. In general:

$$G = \frac{P^2}{2B}\left(\frac{dC}{da}\right) \tag{1}$$

where G is the strain energy release rate, B is the width, C is the compliance and a is the length of the crack in a component subjected to a load, P.

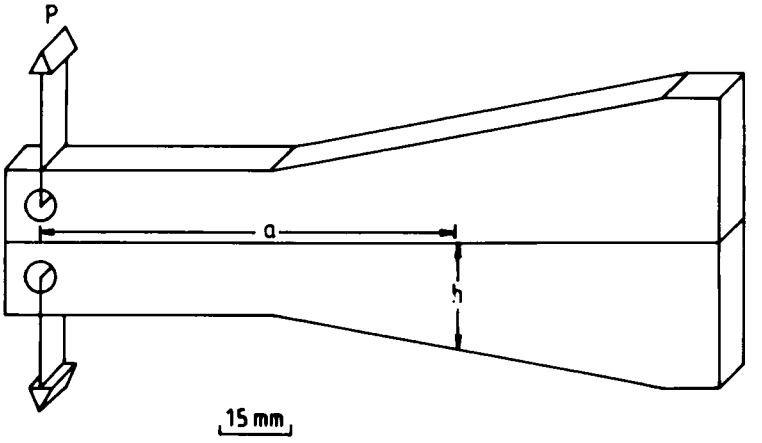

FIGURE 1 Tapered Double Cantilever Beam (TDCB) test geometry.

For double cantilever beams of height, h, and modulus, E:

$$\left(\frac{dC}{da}\right)=\frac{8}{EB}\left[\frac{3a^2}{h^3}+\frac{1}{h}\right] \tag{2}$$

if both bending and shear contributions to the overall deflection and hence compliance are taken into account. Combining (1) and (2):

$$G=\frac{4P^2}{EB^2}\left[\frac{3a^2}{h^3}+\frac{1}{h}\right] \tag{3}$$

The geometry of Figure 1 is chosen such that $\left[\frac{3a^2}{h^3}+\frac{1}{h}\right]$ remains constant—usually denoted m—for which the relationship between G and P becomes independent of crack length. The value of m is not generally in good agreement with the predictions of simple beam theory and it has been shown that it is necessary to establish it experimentally.[4] For the geometry shown in Figure 1, $m=1.8\ mm^{-1}$.[16]

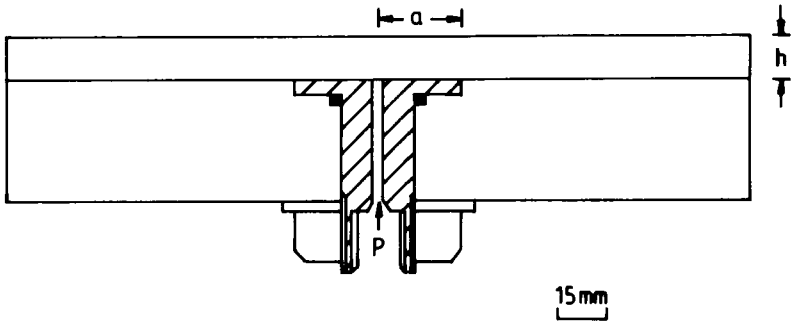

FIGURE 2 Blister test geometry. Note aluminium insert (hatched) which served to connect the pressure line and when the upper surface was coated with mould release agent acted as a starter crack of diameter 2a.

The Blister test geometry, such as that shown in Figure 2, was first analysed from a fracture mechanics viewpoint for conditions of point loading[10] and later for the case of uniform pressure, P, used to load the crack.[11] G is given by:

$$G = \frac{P^2 a}{E f[h/a]} \tag{4}$$

where E is the modulus of the adherend plate and $f[h/a]$ is a dimensionless geometric factor dependent upon the adherend plate thickness, h, and crack radius, a. Details of the analysis depend upon the ratio h/a and closed form solutions are available for two extreme cases in which the adherend behaves as either a thin[17] or an (infinitely) thick[18] plate. Intermediate h/a ratios have been considered independently by Bennett *et al.*[19] who used finite element analysis and Andrews and Stevenson[12] who computed the contribution to the total strain energy of what they describe as both the "near field"—dominated by localised deformations near the crack tip—and the "far field" in which plate-like deflections predominate. They consider both adhesive and cohesive cases—in which crack extension produces one and two new surfaces, respectively. These solutions are plotted, as a function of h/a, in Figure 3. The finite element analysis of Bennett *et al.* was used in calculating G_{IC} in this work.

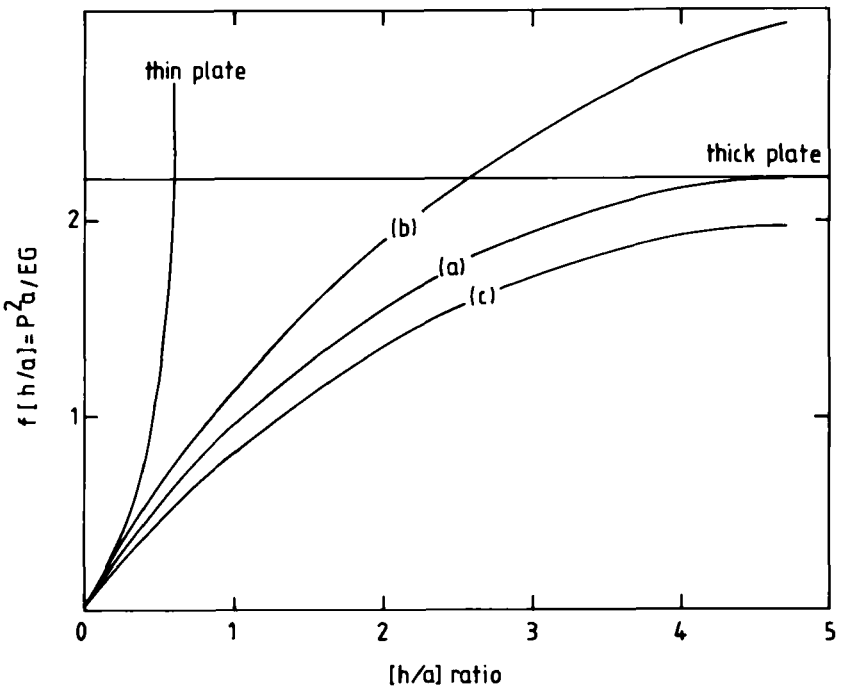

FIGURE 3 Variation of $f[h/a]$ with h/a ratio between the extremes of thin and (infinitely) thick plate behaviours. Analyses of (a) Bennett *et al.*[19] and Andrews and Stevenson[12] for (b) cohesive and (c) adhesive cases relevant for intermediate h/a ratios, are also plotted.

EXPERIMENTAL PROCEDURE

The adhesive/adherend system chosen for the initial model experiments was poly (methylmethacrylate), PMMA, beams or plates, bonded with a cyanoacrylate adhesive *(Loctite 460)*. TDCB specimens were machined to the profile shown in Figure 1[18] which has a linear taper of 7.8°. Following degreasing, the beam halves were bonded over ~75% of their length, the initial ~25% acting as a starter crack, and allowed to cure under finger pressure for a few seconds to develop handling strength. Specimens were left for a minimum of three days at room temperature to allow the adhesive to polymerise fully, following which they were loaded to failure at a rate of 0.5 mm min^{-1} on a Lloyd 6000R computer controlled testing machine.

Blister test specimens were produced from machined PMMA plates as shown in Figure 2. The upper plate, 12.5 mm thick and 200 mm in diameter, was bonded to a 35 mm thick base plate made of the same material using the same procedure adopted for the TDCB specimens. An aluminium insert was incorporated into the base plate to provide the attachment to the high pressure line. The insert also served as the initial starter crack and was sprayed with a proprietary mould release agent before specimen assembly. Because of the larger bonded area, the adhesive was left to polymerise fully for at least seven days at room temperature before testing.

The apparatus used for testing the Blister specimens, manufactured to our design by *PSIKA*, is shown schematically in Figure 4. Pressure was generated initially from a hand pump rated at 200 bars *(Dowty HP6BT)* which was used to store hydraulic energy in an accumulator charged with nitrogen at 125 bars. This was connected to a hydraulic intensifier, with a nominal ratio of 16 : 1, *via* a solenoid operated change-

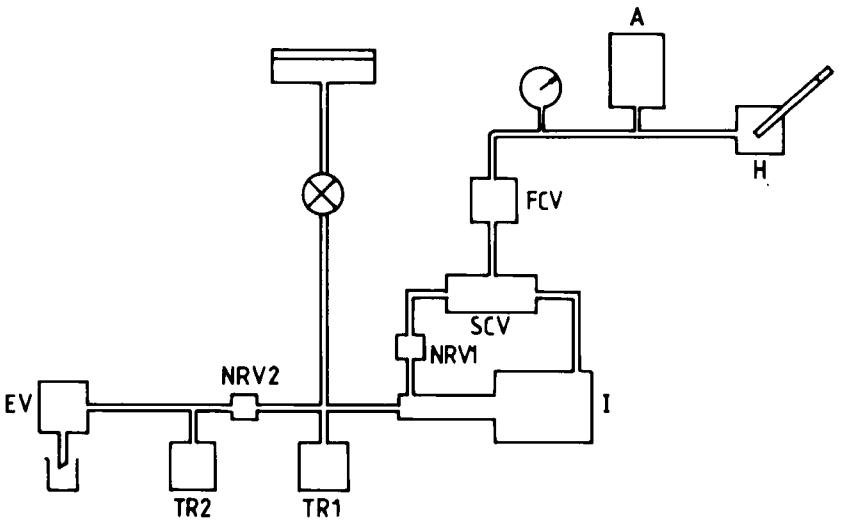

FIGURE 4 Schematic diagram of Blister Test apparatus. Accumulator (A) was charged *via* hand pump (H) and the fluid directed *via* a flow control valve (FCV) and solenoid control valve (SCV) to a 16:1 intensifier (I). Test pressures were recorded from pressure transducer TR1. Transducer TR2, isolated by non return valve NTV2, allowed a differential output to be generated once crack propagation ensued. This was used to release the system pressure *via* and exhaust valve (EV).

over valve and pressure compensated graduated flow control valve *(Sperry Vickers)*. Pressures of up to 2 kbars could thus be generated at reproducible and variable rates ranging between 0.01 and $1000s^{-1}$.

The high pressure line was terminated by a solenoid-controlled, air-operated exhaust valve and the system fitted with two pressure transducers TR1 and TR2. These are separated by a check valve which prevents TR2 sensing the momentary pressure drop caused by the initial increase in volume which accompanied the initial crack propagation. This differential pressure output between TR1 and TR2 was used to terminate the experiment by releasing the system pressure *via* the exhaust valve. A difference of 0.2–0.4 MPa was found to be sufficient to detect the initial crack propagation.

For the model experiments with transparent top plates, a pigment was added to the hydraulic fluid to facilitate the measurement of the arrested flaw diameter. Comparable loading rates were achieved by adjusting the graduated flow control valve to produce a pressurisation rate which resulted in failure after a similar time in the Blister tests to that observed in the TDCB tests. Pressure-time traces were obtained from the output of TR1 and monitored on an X-T plotter. Following the arrest of the initial starter flaw, its new dimensions were recorded to the nearest 0.5 mm using a ruler. Up to three subsequent repropagations could generally be made.

RESULTS

TDCB Tests

A typical load-displacement plot for the tapered double cantilever beam tests is shown in Figure 5. It can be seen that the load at which the starter crack initially

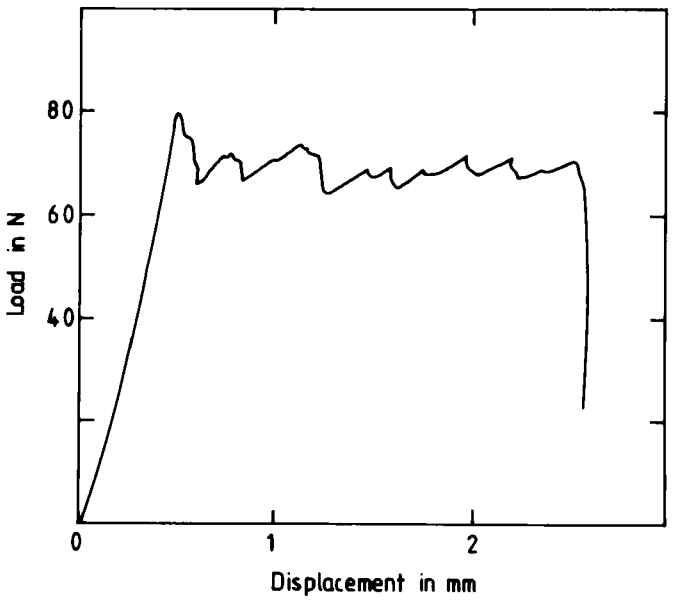

FIGURE 5 Typical load displacement plot for TDCB test.

TABLE I
TDCB data for PMMA/Cyanoacrylate model system

Blunt flaw		Sharp flaw	
G_C kJm^{-2}		G_{IC} kJm^{-2}	
Mean	Standard deviation	Mean	Standard deviation
0.140	**0.045**	**0.110**	**0.017**

propagated was generally somewhat higher than that at which subsequent propagation was observed. The crack propagation behaviour of the model system was unstable, generally described as "stick-slip" and in all cases the locus of joint failure was cohesive within the adhesive. With the specimen geometry chosen it was possible to obtain between three and six estimates of G_{IC} from each test specimen.

The results of tests on ten samples are summarised in Table I which shows the average value of G_{IC} based on 42 measurements together with the mean value of G_C determined from the initial propagation of the starter crack.

Blister Tests

A pressure-time trace for a typical test is shown schematically in Figure 6. Values of G_C and G_{IC} were calculated from equation (4) from the critical pressure at initial and subsequent propagation, respectively. Relevant values of f [h/a] were taken from the finite element analysis of Bennett *et al.*[19] The shape of the arrested crack

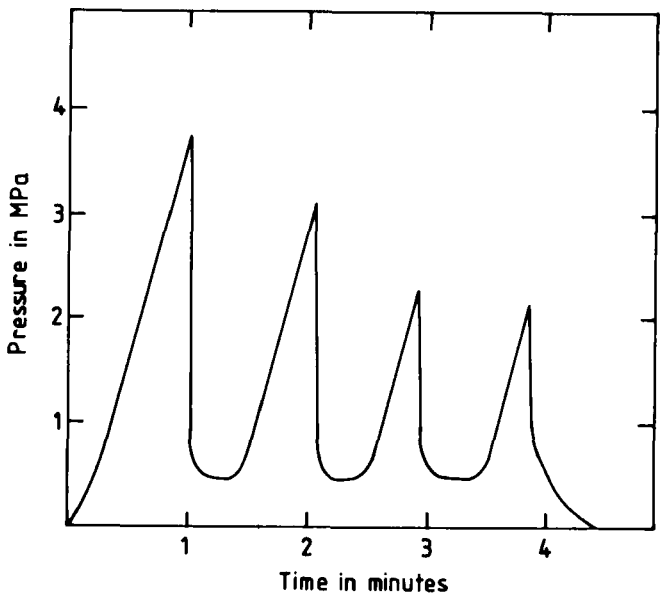

FIGURE 6 Schematic pressure-time traces for Blister test in which three sharp crack retests were possible.

TABLE II
Blister test data for PMMA/Cyanoacrylate model system

Blunt flaw		Sharp flaw	
G_C kJm^{-2}		G_{IC} kJm^{-2}	
Mean	Standard deviation	Mean	Standard deviation
0.194	**0.075**	**0.119**	**0.013**

was generally reasonably circular, in which case the mean diameter was used to calculate G_{IC}. However, whenever significant ovality developed during the initial propagation, it was usually maintained during subsequent tests. In such cases, the major axis was used to calculate G_{IC} as, invariably, the repropagation was initiated in this direction. Post failure examination of the specimens revealed the mode of failure to be cohesive within the adhesive.

Test results are summaried in Table II, which shows the average value of G_{IC} based on 12 measurements on five specimens together with the mean value of G_C determined from the initial propagation of the starter crack.

DISCUSSION

It can be seen from Tables I and II that the correlation between the values of G_{IC} measured by each test is very good. The values are approximately 33% lower than would be expected for bulk PMMA itself. Such a ratio of adhesive : adherend toughness was necessary to avoid problems of crack propagation into the adherend plate itself. This has been frequently reported for the Blister test[14] when testing brittle adherends or when adhesive/adherend toughness is similar.[20] This generally results in the expulsion of a section of the upper adherend in the shape of a truncated cone. Under such circumstances the critical pressure at crack extension relates to the cohesive properties of the adherend rather than those of the adhesive.

The sensitivity to crack tip sharpness of values of G_C measured can be expected to increase as the toughness of the system increases and non-elastic energy dissipative mechanisms which occur at the crack tip dominate. The adhesive fracture energy of the model system chosen, compared with more realistic adhesive/adherend combinations, is very low and probably can be accounted for mostly in terms of the energy required to destroy the chemical bonds within the adhesive.

However, even for a system with an adhesive fracture energy of only ~ 100 Jm^{-2} it can be seen that, for the Blister test, the values determined from the artificial flaw were on average more than 60% higher despite the care taken to produce as sharp a starter crack as possible. This has important implications for a test which, as $\left(\frac{dC}{da}\right)$ is positive under conditions of constant pressure, tends to produce catastrophic failure and, hence, only a single value per specimen. Problems of obtaining consistent data with this test have been recognised in the past and attributed to inconsistencies in the sharpness of the starter crack.[12,14] However, in an attempt to reduce

experimental scatter in results it has sometimes been the lower values of G_{IC} that have been discarded.[12] The results obtained in this work clearly demonstrate that the *lower* values are the most realistic estimates of adhesive fracture energy. It is shown that, no matter how carefully prepared the starter crack in this type of specimen, there is a real need for either some form of precracking—which would be inherently difficult with this type of geometry—or multiple determinations on the same specimen from arrested cracks of natural sharpness.

Having validated the experimental technique, it will be shown in a later paper that the differences between initial and subsequent values determined from the same specimen are even larger for more realistic adhesive/adherend systems—epoxy/aluminium.

Acknowledgement

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