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| | Date | 26/2/92 |
Foreword

Many UK manufacturers and end-users are aware of the merits of structural adhesives in certain critical roles. However the range of engineering applications where adhesives are used is still limited and considerable scope exists for further exploitation. This is largely due to the lack of consistent test methods and validated test data that the engineer or designer requires to specify a particular adhesive for a given application. A recent survey carried out by the Centre for Adhesive Technology, commissioned by the DTI, identified specific areas where validated test methods could improve confidence in predicting joint strength and lifetime. The survey identified measurement methods for use in design, environmental durability and process control as priority areas. The DTI finally selected five projects for support through the Measurements Technology and Standards (MTS) budget. These projects started in December 1992 and are 100% funded by the DTI at the level of £5.4 M over three years.

The survey also identified the need to understand in more detail the complexity of adhesive joint failure modes. This would lead to the development of more robust, validated, failure criteria that are critical to the development of confidence in adhesive bonding technology. This requirement forms the basis of MTS Adhesives Project 2 and is being carried out through a collaboration between AEA Technology, University of Surrey and Imperial College of Science, Technology and Medicine.

The project addresses the issue of failure criteria through initially an extensive study of joint fracture. This forms the project's first task aimed at providing a greater understanding of the micro-mechanisms by which adhesive failure begins and propagates through the joint. The task also makes use of 'in-situ' scanning electron microscopy and laser moiré interferometry. The other major tasks are to investigate new failure criteria and develop advances in existing ones. To make the failure criteria work may require development of existing test methods for the measurement of experimental data. These tasks run in parallel through the second and third years of the project. The project addresses all the major loading regimes envisaged including static, fatigue, creep and impact loading. The failure criteria must be sufficiently accurate for design purposes yet easy to apply. The supporting test methods, whilst being easy to use and utilising existing experimental equipment, should be representative and reproducible to provide sufficiently accurate test data to give good predictions of failure.
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1. Introduction

Adhesive joints invariably fail by initiation and subsequent propagation of sub-critical flaws from regions of locally high peel and shear stresses. The application of continuum fracture mechanics has been the basis for predicting the residual strength and service life of engineering materials and components for over 30 years. It has also been successfully applied to non-metallic materials and has proved to be particularly useful in characterizing the toughness of adhesives and estimating the service life of joints containing damage from environmental effects, fatigue loading and impact events. This report covers activities undertaken within Task 2 'Development of Test Methods for measurement of key fracture properties' of the MTS Adhesives Project 2 'Failure Modes and Criteria'. It forms the final deliverable from that task. The focus of the work reported is the measurement of fracture toughness of two adhesive systems by linear elastic fracture mechanics techniques. Three types of steel double cantilever beam geometries were evaluated including a contoured DCB, a narrow linear tapered DCB and a wide linear tapered DCB. The effect of starter crack, specimen width, adherend material and bondline thickness were examined.

The report is a successor to a review of test methods carried out to determine which experimental methodologies were available for determining the fracture properties of adhesive joints (Davidson & Lee, 1995). A companion review (Crocombe and Kinloch, 1994), a deliverable from Task 3 of this project, discussed the more important adhesive bond failure criteria. Issues relating to tests assessing environmental aspects of failure, particularly due to the effects of moisture, are covered in reports from MTS Adhesives Project 3 'Environmental Durability of Adhesive Bonds'.

The report consists of the following topics:

- A discussion of fracture toughness tests employed and data generated.
- Overall conclusions on the tests reviewed and their suitability.

The report concentrates on tests to measure the properties of adhesive materials relevant to failure criteria based on fracture mechanics principles. This is justified by numerous attempts to predict the static strength, fatigue life or creep rupture time of an adhesive bond by the development and growth of sub-critical flaws. The general principle is to measure the crack growth along the bondline as a function of applied load to calculate the critical strain energy release rate \( G_C \) or critical stress intensity factor \( K_C \).

One of the other failure criteria discussed in the previous review (Crocombe and Kinloch, 1994) is based on maximum stress or strain. This requires material failure properties for which current test methods exist. Tests to measure these material properties are being investigated in MTS Adhesives Project 1 'Measurement of Basic Mechanical Properties of Adhesives for Design Use' and have not been further covered here. Basic mechanical properties suitable for design data has been recently reported (Dean, 1996).
2. Test Methods for Measurement of Adhesive Fracture Properties

2.1 RELATION TO OTHER MTS ADHESIVES PROJECTS

One of the aims of the MTS Adhesives Project 2 is to examine the most appropriate test methods for measuring the properties required for the prediction of strength in selected adhesives and joint configurations. To encourage the wider application of adhesive bonding in industry tests should be as simple as possible to perform whilst providing sufficient accuracy to be useful for design purposes. The work has focused on two adhesives widely used in practice. They represent high stiffness and high compliance resin systems. The first was AV119 (Ciba Polymers, Duxford), a one-part toughened epoxide that cures at ~120°C, and the second was F241 (Permabond Adhesives Ltd, Eastleigh), a compliant room temperature curing toughened acrylic.

The adhesive systems were extensively studied within Task 1 of the project. That work focused on two different bonded joint geometries based on the thick adherend shear test (TAST) and the 180° T Peel test under quasi-static, creep, fatigue and impact loading. Mechanical tests were also carried out on bulk samples to measure tensile and shear properties. In order to ensure minimal overlap of work between other current MTS Adhesive projects there has been appropriate liaison between workers on Project 1 (Characterisation of adhesive materials by NPL, TWI and Department of Mechanical Engineering, University of Bristol) and Project 3 (Prediction of the lifetime and durability of adhesive joints in hostile environments by DRA, Oxford Brookes University and AEA Technology).

2.2 FRACTURE TOUGHNESS TESTS

It is recognised that a requirement exists for the development of more sophisticated techniques in engineering design and non destructive testing to increase confidence in the widespread use of structural adhesive bonding. A large number of test geometries have been developed to determine the crack growth behaviour of structural adhesives. Kinloch (1987) points out that if the crack is at, or very close to one of the interfaces then Mode I and Mode II opening will be present. For quasi-static loading the crack growth rate \( \frac{da}{dt} \) is measured as a function of the critical strain energy release rate \( G_c \) or the critical stress intensity factor \( K_c \) using the adhesives and surface pretreatments of interest. The test sample geometry can be selected from a wide variety of fracture mechanics test coupons currently used. This can include:

- Bonded double cantilever beam, tapered and untapered, (DCB)
- Bonded double torsion specimens, (DT)
- Compact Tension, (CT)
- Bonded cracked lap shear, (CLS)
- End notched (cracked) flexure, (ENF)

The approach used in fracture mechanics is to conduct mechanical tests on a particular representative test geometry to measure crack growth along the adhesive bondline as a function of applied load. The critical strain energy release rate, or fracture energy, \( G_c \) and the critical stress intensity factor \( K_c \) for that mode of crack opening is calculated. The data are usually plotted as a function of the crack length to produce a crack growth resistance \( R \)-curve from which critical values for initiation and propagation can be obtained. The strength of other types of joints may
then, in principle, be predicted using fracture mechanics employing a standard Y-calibration factor representing the geometry of the joint. The analysis also requires the inherent flaw size or the critical value of the fracture energy for the applied crack opening mode.

The lifetime of other joint designs can then be predicted provided that crack growth is controlled by the release of stored elastic strain energy and the critical flaw size is known. Typical values for various adhesives are summarised in Table 1.

<table>
<thead>
<tr>
<th>Adhesive</th>
<th>Fracture Toughness (J/m²)</th>
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<tr>
<td>TGMDA epoxy/ DDS hardener</td>
<td>76</td>
</tr>
<tr>
<td>DGEBA epoxy/ anhydride hardener</td>
<td>160</td>
</tr>
<tr>
<td>DGEBA epoxy/ amine hardener</td>
<td>400</td>
</tr>
<tr>
<td>AV119 (Bulk data)</td>
<td>620</td>
</tr>
<tr>
<td>AV119 (1 mm bondline thickness)</td>
<td>700-800</td>
</tr>
<tr>
<td>Redux 313A epoxy film</td>
<td>1830</td>
</tr>
<tr>
<td>Redux 312 epoxy film</td>
<td>2000</td>
</tr>
<tr>
<td>Rubber toughened epoxy paste</td>
<td>~2000</td>
</tr>
<tr>
<td>FM73 epoxy film</td>
<td>2500</td>
</tr>
<tr>
<td>Hysol EA9309 epoxy paste</td>
<td>3500</td>
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Table 1. Typical adhesive room temperature fracture toughness values

Strength prediction, for static loading, based on fracture mechanics principles is not currently widely used for joint design because of the difficulty of assuming a critical flaw size and obtaining valid fracture toughness parameters. However, during fatigue or creep loading, as inherent flaws propagate or other flaws are initiated, the application of fracture mechanics principles may be more appropriate. Evidently there are many unresolved areas in the application of fracture mechanics to the failure of adhesive joints. The dependence of measured fracture parameters on joint geometry, the effects of temperature and strain rate, and theoretical complications arising from cracks at, or near, the interfaces are clearly areas where further understanding is required.

### 2.2.1 Application of Fracture Mechanics to bonded joints

The materials scientist's method of analysis usually centres on measuring the strain energy release rate for initiation, $G_i$, and propagation, $G_p$, as a function of crack length. The engineering
approach usually calculates the critical stress intensity factor for the loading mode using the expression:
\[ K = Y \sigma \sqrt{a} \]

where \( Y \) is a constant dependent on the specimen geometry and obtained from standards and research reports
\( \sigma \) is the remote applied stress and
\( a \) is the crack length.

The energy balance approach has the advantage of avoiding theoretical difficulties associated with analysing cracks that arise when the critical stress intensity factor approach is used. Crack blunting effects do not apply in this case. The value of \( G \) can be measured from the applied load and crack length measured directly or by a compliance calibration factor measured previously on a pre-cracked sample. A graph of \( \log \frac{da}{dt} \) versus \( \log G \) is normally linear and so may be fitted as a power law.

The basic method of analysis is to postulate that in the bonded joint under investigation any sub-critical flaw present will propagate slowly under the action of applied stress (and environment) until it reaches a critical size after which it propagates catastrophically. The same principle can also be applied with creep and cyclic loading. In laboratory fatigue experiments a simple sinusoidal waveform or a more complex realistic, sometimes random, loading spectra can be applied to the specimen. Crack growth data and applied loads can be monitored and stored by computer during testing to obtain the adhesive’s crack growth characteristics.

Fracture mechanics methodology assists in the understanding of large scale service failures and their avoidance. From an engineering point of view, fracture mechanics aims to predict the onset of fracture of a structure containing a crack of given size and geometry. From a materials science point of view, fracture mechanics aims to isolate material parameters of importance to crack resistance so that materials with improved fracture toughness can be devised. Given a quantitative knowledge of the critical driving force, the values of nominal stress necessary for crack propagation for various sizes of specific cracks can be estimated. This premise is applicable only for progressive fracture spreading from a local region of high stress. There are three major patterns of crack extension behaviour:

- Slow, stable crack growth where the forward movement of the crack border develops gradually as a function of time and environment.
- Rapid, stable crack propagation, where the speed of creation of new fracture surfaces is fixed by the rate of energy release and increases with time.
- Crack growth and arrest in a ‘stick-slip’ manner.

The failure of adhesive joints occurs by the initiation and propagation of flaws when the joints are subjected to either mechanical, thermal, environmental stresses or combination of these. Mechanical stresses may be applied statically or dynamically. One approach to failure prediction is to mathematically analyse the loads at which flaws propagate and describe the manner of their growth. Intrinsic flaws in adhesives occur naturally and may be caused by interfacial cracks from poorly wetted out adhesive, voids from entrapped vapours or gasses and particulate matter. They can be introduced deliberately to control bondline thickness or accidentally by poor process handling. Flaws may either be present during manufacture and their precise position within the
bonded area is important, or they may develop upon subsequent stressing. The key issue is whether such defects grow to a critical size where the defect can cause catastrophic failure. Fracture mechanics principles are useful in characterising the toughness of adhesive, assessing mechanisms of failure and as an aid to the prediction the service life of cracked or damaged structures.

### 2.2.2 Conditions for adhesive fracture

For fracture to occur two conditions are necessary:

1. Sufficient strain energy is released to supply the energy requirements of the new fracture surfaces. The release of energy comes either from the stored elastic strain energy or from potential energy of the loading system, including test machine. This approach provides a measure of the energy required to extend a crack over unit area, denoted by $G_c$, the fracture energy or critical strain energy release rate.

2. The stress intensity factor caused by modifications to the stress field surrounding the sharp crack, for a linear elastic material, must exceed a critical value $K_c$ which is a material property.

The basic aim of experimental fracture mechanics is to identify fracture criteria such as $G_c$ and $K_c$ that are independent of the geometry of the cracked body. For adhesives the materials used are seldom perfectly elastic and localised viscoelastic and/or plastic energy dispersive processes at the tips of cracks are desirable to introduce a degree of toughness and crack blunting. Such micro-mechanisms are usually the main source of energy absorption in the material, and indeed the microstructure of toughened adhesives is tailored to maximise such processes and so impart enhanced toughness. $G_c$ includes all the energy losses occurring around the crack tip. The fracture criterion becomes:

$$\frac{1}{b} \frac{\partial (W_d - U)}{\partial a} \geq G_c$$

where $W_d$ is the work done by the external force

$U$ is the elastic energy stored in the specimen

$a$ is the crack length

$b$ is the specimen width.

For structures exhibiting bulk linear elastic behaviour the inequality gives,

$$G_c = \frac{F^2}{2b} \frac{\partial C}{\partial a}$$

where $F_c$ is the load required for crack propagation.

This equation provides the basis for determining adhesive fracture energy for a number of specimen geometries.

### 2.2.3 Relationship between $G_c$ and $K_c$

Regardless of the fracture mode, values of $G_c$ and $K_c$ can be found by experiment as a function of load and crack size or as a function of load and compliance. If the material is isotropic and linear
elastic, analysis shows that for ‘thick’, plane strain samples the relationship between the adhesive fracture energy $G_c$ and the fracture toughness $K_c$ for linear elastic materials is given by:

$$G_c = \frac{(1 - \nu^2)}{E} K_{ic}^2 + \frac{(1 - \nu^2)}{E} K_{ic}^2 + \frac{(1 + \nu)}{E} K_{ili}^2$$

Similarly for ‘thin’, plane stress specimens:

$$G_c = \frac{1}{E} K_{ic}^2 + \frac{1}{E} K_{isc}^2 + \frac{(1 + \nu)}{E} K_{ili}^2$$

or

$$G_c = G_{isc} + G_{isc} + G_{ili}$$

where

$$G_{isc} = \frac{(1 - \nu^2)}{E} K_{ic}^2 \text{ for plane strain}$$

and

$$G_{ili} = \frac{1}{E} K_{ici}^2 \text{ for plane stress}.$$ 

### 2.2.4 Relationship between $G$ and $K$ for Adhesive Joints

For a crack in an adhesive, which is relatively distinct from an interface, the above expressions are still valid and the appropriate elastic values for the adhesive may be employed to correlate $G_{(joint)}$ and $K_{(joint)}$. Thus, for plane strain:

$$G_{(joint)} = \frac{(1 - \nu^2)a^2}{Ea} K_{(joint)}^2$$

For the case of a crack present at or very near an interface the situation is less clear and an effective modulus, weighted between the adherend and the adhesive, has been suggested. For cracks very close to the interface no relations are currently available.

For non-linear elastic materials the concept of $G$ is still valid but the interpretation of stress intensity factor is not as straightforward and the above relations are not generally applicable.

Adhesive joints must support both shear and peel forces and any crack lying in the plane of the joint may experience combinations of Mode I, Mode II and Mode III types of loading depending on the applied stress state. For adhesive joints, the crack opening Mode I is the most critical. However, to be able to design adhesively bonded structures with a similar level of confidence as metallic structures, it will be necessary to understand the fracture behaviour under combined loading conditions.

For a single mode loading the driving force for crack extension is the strain energy release rate $G = G_{total}$. Under mixed mode loading $G_{total} = G_i + G_p + G_{III}$. The appropriate driving force must be established experimentally and this requires the testing specimens with a different ratio of modes. The following sections describe the different specimen geometries and data reduction techniques that are applicable to the characterisation of adhesive bonds.
3. Experimental Techniques

A variety of specimen geometries exist which aim to measure the fracture resistance of structural adhesives. Most geometries have been adapted from metallic, and more recently advanced structural composite, fracture mechanics specimens. Test method geometries may be based on bulk adhesive samples or adhesive bonds between metal or composite adherends. The geometries have been reviewed extensively (Kinloch 1987). The overall MTS Project 2 experimental work was aimed to address test methods for fracture toughness measurements based on static, fatigue, creep and impact loading. The work covered in this report covers static loading. The techniques should be industrially relevant, and easy to carry out using relatively simple specimen geometries.

3.1 MODE I SPECIMEN GEOMETRY

For adhesive bonds the opening, Mode I is the most critical. The simplest specimen geometry and one of the most widely employed is the double cantilever beam (Mostovoy, Crosley & Ripling 1967). The DCB specimen has been the subject of numerous analyses based on the classical beam theory. The crack is made to extend by applying a tensile force acting in a direction normal to the crack face. The specimen recommended in ASTM D3433 is shown below in Fig 1.

Two metallic rectangular beams are bonded together with an initial unbonded length and the beam is pin loaded until the crack grows rapidly. The strain is held constant and the load drop as a function of time is recorded until the crack stabilises. The new crack length is measured and the procedure repeated until failure. The fracture toughness is given by:
For a thin adhesive layer the compliance can be estimated from beam theory as:

\[
C = \frac{8(a^2 + ah^2)}{Ebh^3}
\]

and hence

\[
G_{IC} = \frac{4F_c^2(3a^2 + h^2)}{Eb^2h^3}
\]

Alternatively the compliance can be measured experimentally as a function of the crack length. The deflections can be estimated from the cross head movement or directly from extensometry across the bond thickness.

### 3.1.1 DCB Specimen Analysis

The DCB specimen has been extensively analysed as a classical fully encastered beam. This assumes that the loaded beams are rigidly built into the remainder of the uncracked specimen and that classical small deflection beam theory is valid (Berry 1963).

The compliance of the straight-sided DCB specimen may be obtained from elastic beam theory as:

\[
C = \frac{2a^3}{3E_1I}
\]

where \(a\) is the crack length and \(E_1I\) is the flexural rigidity of each beam of the specimen.

The strain energy release rate is:

\[
G_I = \frac{F^2}{2b} \frac{\partial C}{\partial a}
\]

Hence:

\[
G_I = \frac{F_c^2a^3}{bE_1I}
\]

Critical conditions occur when \(F = F_c\), and the Mode I fracture toughness \(G_{IC}\) for plane stress conditions is

\[
G_{IC} = \frac{F_c^2a^3}{bE_1I}
\]

An experimental compliance approach has been taken (Berry 1963) where the beam compliance is expressed as
\[ C = \frac{a^n}{H} \]

where: \( n \) and \( H \) are points to be determined experimentally.

The two approaches coincide if \( n = 3 \) and \( H = 1.5E_1I \). The critical value of \( G_{ic} \) according to this approach is:

\[ G_{ic} = \frac{nF_s^2}{2bH} a^{n-1} \]

Substitution of \( \delta_c = \frac{F_s a^n}{H} \)

where \( \delta_c \) is the displacement at the critical load,

yields

\[ G_{ic} = \frac{nF_s \delta_c}{2ba} \]

The Berry method is currently accepted by ASTM as the basis for experimental fracture toughness determination of composites. However, these equations may need to be corrected for various effects that are not accounted for in the simple beam theory (Blackman, Dear, Kinloch & Osiyemi (1991)). Such effects arise from shear deformation and deflections at the crack tip and loading arms and any stiffening of the arms due to the presence of end blocks which are often used with thin composite adherends. In Mode I the correction factor \( \chi_i \) may be introduced for end rotation and deflection of the crack tip. The large deflections at the bonded end blocks may be taken into account by using the correction factors \( Q \) and \( N \). The expressions for the corrected compliance and Mode I fracture energy for the DCB test is:

\[ C = \frac{8N(a + \chi_i h)^3}{bh^3E} \]

\[ G_{ic} = \frac{12QF_s^2(a + \chi_i h)}{b^2h^3E} \]

Hence

\[ \sqrt[3]{\frac{C}{N}} = 3 \sqrt[3]{\frac{8}{bh^3E}} (a + \chi_i h) \]

The value of \( \chi_i \) may be deduced by plotting \( \sqrt[3]{\frac{C}{N}} \) versus the corresponding value of crack length, \( a \), where the intercept gives the value of the correction factor \( \chi_i \).

### 3.1.2 DCB Testing issues

The test procedure employed depends on the data reduction procedure to be used. The classical beam approach and the area method require loading and unloading cycles for each increment of crack growth; other methods require continuous loading of the specimen. For crack length measurements, an optical microscope, a precision Vernier gauge or an electrical method such as
potential drop is needed. The initial crack length $a$, from the load line to the tip of the starter crack on both sides of the specimen is first determined. The load versus crack opening displacement curve for a cross head rate of $\sim 0.5$ mm/min is monitored. The DCB specimen is loaded until the crack extends about 10 mm and the cross-head is stopped. The crack length is measured and the specimen is unloaded. The procedure is repeated until the crack is $\sim 100$ mm in length. For continuous loading methods as the critical load is realised the crack starts to grow. The position on the chart when the crack has grown 2.5 mm ahead of the starter crack is marked and this procedure is continued until the crack has advanced about 40 mm.

3.1.3 Data Reduction Methodology

The measurement of adhesive fracture toughness depends on the method adopted for data interpretation. Data reduction procedures are of two types:

- Direct energy methods, such as the Areas method.
- Compliance methods, requiring a relationship to be found between compliance and crack length.

Experimental data usually comprises load-displacement records for cracked samples together with sample geometry and crack length. The choice of analysis method used to interpret the data should ensure that material or specimen behaviour does not violate basic assumptions or the fracture parameter will lose its significance as a design parameter. Some of these effects include:

- rotation and deflections occurring at or near the crack tip;
- large displacements being present in the test sample;
- stiffening effects due to the geometry of the loading points.

For discontinuous loading there are several common data reduction methodologies as described later.

3.1.4 Corrected Beam Theory

Simple beam theory predicts that the compliance of a perfectly built-in DCB specimen is:

$$ G_{lc} = \frac{3F \delta}{2Ba} $$

where

- $F$ is load
- $\delta$ is displacement
- $B$ is specimen width and
- $a$ is crack length.

In practice this expression underestimates the compliance as the beam is not perfectly built-in and a correction factor is applied. This correction treats the beam as containing a slightly longer crack, $a + \Delta$, and $\Delta$ may be experimentally determined by plotting $\sqrt{C}$ as a function of crack length. $G_{lc}$ is then given by:

$$ G_{lc} = \frac{3F \delta}{2B(a + \Delta)} $$
This approach allows the adherend modulus to be calculated, which should be independent of crack length, and is a useful check on the procedure.

\[ E_f = \frac{8(a + \Delta)^3}{C b h^3} \]

### 3.1.5 Experimental Compliance (Berry's Method)

An alternative approach is to plot compliance against crack length on a log-log plot. The slope of this plot, \( n \), can be used to derive \( G_c \) as follows:

\[ C = K a^n \]

so

\[ G_c = \frac{n F \delta}{2 B a} \]

### 3.1.6 Area Method

An alternative method to determine the fracture toughness is the area method that allows for the direct evaluation of \( G_c \). The critical strain energy release rate may be determined from a loading-unloading sequence. \( G_c \) may be determined as:

\[ G_c = \frac{\Delta A}{b(a_2 - a_1)} \]

where \( \Delta A \) is the area under the curve and \( a_2 - a_1 \) is the increment in crack length.

For linear elastic behaviour \( G_c \) is approximately:

\[ G_c = \frac{F_c \delta_c - F_c \delta_c}{2b(a_2 - a_1)} \]

where \( A \) and \( B \) refer to parts of the curve.

An average \( G_c \) value is obtained from the total series of loading and unloading curves. One of the major advantages of the area method is that it quantifies the propagation toughness while the compliance techniques characterise the initiation toughness. Fracture toughness determination according to this method requires that the parameter \( n \) be determined from the slope of a plot of \( F_c/\delta_c \) versus \( a \) in a log-log diagram. Once \( n \) is obtained, the fracture toughness is determined for each crack length using the following expression.

\[ G_c = \frac{n F_c \delta_c}{2ba} \]

In some materials, stick slip behaviour is observed where unstable growth occurs.
3.1.7 Tapered Double Cantilever Beam (TDCB)

This specimen is an adaptation of the DCB but uses shaped metallic adherends. The specimen, designed initially by Mostovoy and Ripling (1966), is tapered to give a linear compliance change with crack length. To achieve this the height of the adherends is varied such that

\[ \frac{3a^2}{h^3} + \frac{1}{h} = m \]

where \( m \) is a constant.

The ASTM specimen for \( m = 90 \) is shown in Figure 1.

Thus, at a given load the value of the fracture energy remains independent of crack length and can be obtained without knowledge of the crack length from the expression

\[ G_{ic} = \frac{F_c^2}{2h} \cdot m \]

This feature is particularly useful in cases where difficulty is encountered in locating the crack tip. The experimental procedure is simpler than with the DCB but the specimen geometry is more complex and expensive to machine.

A high \( m \) number generates a geometry with a low taper angle that causes a large bending stress in the plane of the crack. However, because of the relatively low modulus of the adhesives used, this stress is not significant. The specimen geometry is not usually suitable for bulk specimens as the high bending stresses cause the arms to break. The problem is minimised for a low value of \( m \) by making the beams stiffer and adding side grooves to the specimen to direct the crack.

When the specimens are made stiffer, linear compliance is achieved but the specimens cannot be used to determine \( G_{ic} \) because the assumptions used in beam theory become increasingly invalid as the height to length ratio increases. In place of \( m \) an experimental value determined from the compliance calibration designated \( m' \) is required. Hence the toughness of monolithic specimens having low \( m \) is defined

\[ G_{ic} = \frac{8F_{max}^2}{2b_nE_h} \cdot m' \]

where

- \( b_n \) is the width at the crack plane,
- \( b \) is the gross specimen width and
- \( E \) is the tensile modulus of the adherend.

Mostovoy showed that by using the DCB or TDCB pure Mode I loading can be set up on the bond line. This fracture force is analogous to the stress intensity factor \( K_i \) used to determine the force at the crack tip in homogeneous materials where:

\[ G_i = \frac{K_i^2}{E_{adhesive}} \quad \text{for plane stress} \]

and,
An alternative design to achieve constant \( \frac{\partial C}{\partial a} \) uses contouring of the width of the specimen along its length. This is most often used with composite adherends that tend to have uniform thickness and is not recommended for adhesives. It has also been shown (Timoshenko & Goodier 1970) that by using a traditional TDCB specimen manufactured with a 45° scarf angle at the bonded faces, it is possible to investigate mixed Mode I-III behaviour. By varying the scarf angle the ratio of Mode I : Mode III can be investigated.

### 3.2 MODE II GEOMETRY

The edge notched flexure (ENF) specimen is widely used to characterise the Mode II interlaminar fracture. Russell and Street (1982) used the specimen to characterise the critical strain energy release rates of advanced composites. The specimen comprises a 3 point flexure bonded beam with a through-the-width crack in the adhesive running from one end face. The delamination is placed at the end of the specimen to accommodate the sliding deformation across the adhesive that results from flexural loading. The sample is currently being investigated for \( G_t \) measurements in composite adherends, but may be applicable to adhesive bonds. The specimen is easy to manufacture, the test fixture is simple and data reduction is straightforward. The starter crack can be produced by embedding a 20 \( \mu \)m PTFE film or release agent coated aluminium film at the centre of the bond.

Compliance can be measured for several crack lengths in a single ENF specimen containing a long crack. Providing that the loads applied are not sufficient to cause the crack to extend, various crack lengths can be achieved by sliding the specimen about the central loading point. The compliance is fitted to a polynomial of the form:

\[
C = C_1 + ma^3
\]

where \( C_1 \) is a constant, including machine compliance and \( m \) is the slope of the \( C \) verses \( a^3 \) data.

The resulting expression for the strain energy release rate based on compliance calibration is:

\[
G_{II} = \frac{1.5F^2ma^2}{b}
\]

An alternative way to reduce the data is to normalise the compliance with the compliance \( C_0 \) for the beam with no crack:

\[
\frac{C}{C_0} = 1 + m \left( \frac{a}{L} \right)^3
\]

The parameter \( m \) is obtained as the slope of the line \( \frac{C}{C_0} \) against \( \left( \frac{a}{L} \right)^3 \)

It should be noted that alternative Mode II interlaminar flexural specimens have proliferated since the introduction of the ENF specimen to composites. Mode II geometries include the end loaded...
slit laminate, the cantilever beam enclosed notch (CBEN), the centre notch flexure (CNF) for static and impact loading and the mixed mode bending geometry (MMB). Modifications to the ELS test have been described by Whitney et. al (1989) where a sliding clamp was used to eliminate the axial force generated into the specimen when it is encastered at the clamped end. In this case:

\[ G_{II} = \frac{9QF_c^2(a + \chi_{II}h)^2}{4b^2h^3E} \]

and the compliance \( C \) is given by:

\[ C = \frac{N[3(a + \chi_{II}h)^3 + (L + 2\chi_{I}h)^3]}{2bh^3E} \]

where \( \chi_{II} \) is the correction factor for Mode II fracture which may be taken to be 0.42\( \chi_{I} \).

### 3.3 Mixed Mode Geometry

#### 3.3.1 Cracked Lap Shear Joints

The cracked lap shear (CLS) specimen geometry can be used to represent a structural joint subjected to in-plane loading. Both shear and peel stresses are present in the joint. The magnitude of each component of the mixed-mode loading can be modified by changing the relative thickness of the strap and lap adherend. The ratio of \( \frac{G_{II}}{G_{I}} \) can vary between 0.6 to 0.2 as the thickness of the shorter lap adherend is increased. The ratio can also be changed by machining the lap adherends to tapers. For samples with strap/ lap thicknesses of 2:1 a taper angle of less than 5° - 10° reduces the peel effect on the bond such that pure Mode II operates. Experimental data shows a significant improvement in debond resistance for taper angles below 10°, for toughened epoxide adhesives. Uncracked specimens may be tested in fatigue to investigate the crack initiation and growth in the adhesive bond line. Alternatively pre-cracked specimens may be used.

The cracked lap shear specimens can be analysed using finite element techniques to determine the strain energy release rate for a given geometry, debond length and applied load. The non-linearity associated with the large rotation in the asymmetric cracked lap specimen must be taken into account.

The cracked lap shear specimen is one of the most commonly used comparison tests, since it allows a range of Mode I and Mode II ratios. This specimen represents a simple structural joint subjected to in-plane loading. Both shear and peel stresses are present in the bondline of the joint. Detailed mechanisms occurring during the initiation and propagation of cracks can be studied using high resolution strain mapping such as laser moiré interferometry (LMI) techniques.

Cracked lap shear joints represent mixed mode loading and large area bonds typical of many structural applications. They are also convenient specimens for laboratory tests on debond growth and fatigue crack growth. Analytical studies of the joint geometry provide insight into geometric non linear effects, and the effect of adherend and adhesive material properties. The magnitude of each component of the mixed Mode I and Mode II loading can be modified by changing the relative thickness of the strap and lap adherends. The typical specimen geometry consists of ~200 mm long lap adherend bonded over a 250 mm long strap adherend. Both the specimen geometry

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and the adhesive thickness relative to the bonded length are important in determining the ratio of $\frac{G_L}{G_{LL}}$. According to Kinloch (1981), for the CLS geometry:

$$G_c = \frac{F_c^2}{2h^2} \left\{ \frac{1}{(E_s d)_1} - \frac{1}{(E_s d)_2} \right\}$$

where 1 and 2 refer to the adherends and $(E_s d)_2 > (E_s d)_1$. 
4. Stress Intensity Factors for Adhesive Joints

The critical stress intensity factor (fracture toughness) $K_{lc}$ for adhesive joint configurations, is a more useful design parameter from the stress analysis viewpoint than fracture energy. In homogeneous isotropic materials, $K$ expressions for a wide range of test piece geometries and loading configurations have been computed and are available in the published literature, e.g. Iron & Steel Inst. (1968) and ASTM (1966). In the most general form

$$K = \phi \sigma \sqrt{a}$$

where $\sigma$ is the applied stress and

$\phi$ is the shape correction factor.

4.1 USE OF $K_{lc}$ IN ADHESIVE JOINTS

For cracks in an adhesive layer the value of $K$ is a function of the ratio of the adhesive and adherend moduli, thickness of the adhesive layer, and joint geometry. In many geometries where the elastic energy available for crack growth is largely stored in the adherends as in the DCB, TDCB, DT and CT the values are similar to the homogeneous specimen multiplied by $\sqrt{\frac{E_{adhesive}}{E_{adherend}}}$. This assumption is valid for adhesive thicknesses up to several millimetres. This modification to the equation is not directly applicable to two phase materials such as metal/adhesion joints for ‘thick’ bond lines where the compliance contribution due to the adhesive is significant. If the calibration factor is not known then it must either be ascertained using numerical analysis or experimentally using compliance calibration. For ‘thin’ bond lines it has been shown by Trantina (1972) that:

$$K_{nc} = 6\phi_n \sqrt{a} \sqrt{\frac{E_{adhesive}}{E_{adherend}}} = 6\sqrt{a} \phi_n^*$$

where $n$ is I for Mode I stressing of a compact tension specimen and $n$ is II for Mode II loading of a compact shear specimen.

Experimentally fracture loads $F_c$ are recorded against corresponding crack lengths, $a$.

All the above relationships predict infinite stresses at the crack tip; in reality plastic yielding in a zone ahead of the crack tip will occur. Where the zone is small then it will not greatly disturb the elastic stress field and the assumptions of LEFM broadly still apply. The elastic stress field ahead of this notional crack may therefore be regarded as identical to the stress distribution of a real crack of length $a$ with the extent of plastic zone $2r_y$. The size of the plastic zone radius is given by:

$$r_y = \frac{1}{2\pi} \left( \frac{K}{\sigma_{op}} \right)^2$$

for plane stress and:

$$r_y = \frac{1}{6\pi} \left( \frac{K}{\sigma_{op}} \right)^2$$

for plane strain.

The corresponding crack-opening displacements at the crack tip are:
\[ \delta_i = \frac{K_i^2}{E_i \sigma_y} \text{ for plane stress and} \]
\[ \delta_i = \frac{K_i^2}{E_i \sigma_y (1 - v_i^2)} \text{ for plane strain.} \]

Plane strain fracture conditions are considered to be present when the plastic zone is < 2% of both the component thickness and crack length (Knott 1971).

4.2 WIDTH EFFECTS

In practice the values of \( GIC \) or \( KIC \) can, over a certain range of widths, vary with the width of the specimen. This arises because the state of stress near the crack tip varies from plane stress in very thin specimens to plane strain near the centre of a wide plate. This arises because the tensile stress at which a material yields is greater in a triaxial plane strain field than in biaxial plane stress. Therefore in the former case a more limited degree of plasticity develops at the crack tip. The lower conservative, plane strain value is usually required for engineering design and life prediction studies. The width, \( b \), necessary to achieve this condition is:

\[ b = 2.5 \left( \frac{KIC}{\sigma_{ay}} \right)^2 \]

where \( KIC \) is the plane strain value.

Figure 2 shows the prediction of the minimum width for plane strain conditions for AV119. For a tensile yield stress of 30 MPa the minimum width is \(~6\) mm.
5. Experimental Procedure

Room temperature Mode I and mixed Mode I-III fracture toughness tests have been carried out on Ciba Polymers AV119 and Permabond F241 using 3 different types of double cantilever beam.

- 25 mm wide contoured Mode I DCB
- 25 mm wide mixed Mode I-III DCB
- 13 and 25 mm wide linear tapered DCB (according to ASTM D3433)

The testing geometries are well established in ASTM/BS/ISO tests. The main areas of interest were crack initiation effects (precracking or starter films) and geometry effects (specimen width and initial starter crack position). Mode I was considered to be the most relevant loading and most of the testing concentrated on this aspect.

In all cases high strength steel adherends were used to minimise yielding problems that had been encountered in other work using aluminium adherends. All contoured adherends were flat, free from burrs, and smooth before surface treatment and bonding. Each bonding surface was solvent cleaned, grit blasted and subsequently solvent cleaned again.

The adhesive was prepared and applied according to the procedure prescribed by the manufacturer of the adhesive. The two DCB halves were then assembled and clamped (Figure 3) and the adhesive allowed to cure under conditions prescribed by the adhesive manufacturer. Figure 4 shows the schematic of the test geometry. All the data was obtained from a starter crack introduced using a razor blade. The crack lengths were measured during the tests using a travelling microscope and checked after the test by observing the crack arrest markings on the fracture surface.

All mechanical tests were carried out using an Instron Model 1185 100 kN universal test machine under displacement control. The loading rate chosen was 0.5 mm/min. Compliance calibrations were carried out using AV119 bonded beams, 1 mm bondline thickness, and different crack lengths were introduced by a precision saw cut. The sample compliance was compared with simple beam theory calculations. The differences were generally around 8 to 10%. The methods employed were considered simple to carry out and so were deemed relevant to commercial practice.
The adherends are held together whilst curing with a mixture of clamps and two bolts torqued to hold down the two DCB halves in correct orientation.

**F241 Cure**: Room temp. (18-22°C) for over 24 hours.

**AV119 Cure**: 120°C for 150 min.

The adherends are held together whilst curing with a mixture of clamps and two bolts torqued to hold down the two DCB halves in correct orientation.

Figure 3: Assembly of contoured double cantilever beam specimen

**Test Method (sharpening a crack)**

Figure 4: DCB testing geometry
6. Results and Discussion

Fracture toughness data were initially derived for the Mode I tapered DCB (using a 1 mm bondline thickness, 13 mm wide) for AV119 epoxy adhesive. Previous data analysis was affected by the low number of compliance measurements (e.g. two crack arrests before complete separation of the beam). A compliance calibration procedure was subsequently adopted by introducing a fine saw cut to different crack lengths and measuring the specimen stiffness. Figure 5 shows a typical compliance calibration curve for an AV119 bonded DCB (1 mm bondline). The slope of the graph was $7 \times 10^{-6}$ m/N/m and the correlation coefficient was 0.94.

![Compliance Calibration Curve](image)

Figure 5: Compliance calibration for 25 mm wide steel tapered DCB

Table 1 shows average values obtained for AV119 using the narrow and wide linear tapered DCB, and the Mode I and mixed mode, scarfed, DCB. Figure 6 shows that the measured fracture energy obtained for AV119 joints appeared to be a dependent on the initial crack length, i.e. longer starter cracks produced lower fracture energies.
<table>
<thead>
<tr>
<th>Nominal Bond thickness (mm)</th>
<th>Fracture energy (J/m²)</th>
<th>Test Method</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.25</td>
<td>1336 ± 143</td>
<td>LT DCB (ASTM D3433)</td>
</tr>
<tr>
<td>0.2</td>
<td>~876</td>
<td>TDCB (25 mm wide)</td>
</tr>
<tr>
<td>1</td>
<td>782 ± 146</td>
<td>LT DCB (ASTM D3433)</td>
</tr>
<tr>
<td>1</td>
<td>~703</td>
<td>TDCB (25 mm wide)</td>
</tr>
<tr>
<td>1</td>
<td>732 ± 261</td>
<td>Mode I (scarf DCB)</td>
</tr>
<tr>
<td>1</td>
<td>732 ± 261</td>
<td>Mode III (scarf DCB)</td>
</tr>
<tr>
<td>5</td>
<td>620 ± 90</td>
<td>Bulk data (ICSTM)</td>
</tr>
</tbody>
</table>

Table 1: AV119 room temperature fracture energy values

Figure 6: Fracture energies of AV119 using the ASTM D3433 load method

The acrylic system, F241, initially gave results which were more variable than expected. This effect was attributed to the fact that the F241 continues to crosslink over a long period at room temperature. This effect is clearly shown in Figure 7 where the measured fracture energy increases approximately linearly with time for specimens cured at room temperature and tested from 20 to 45 hours after assembly.
Figure 7: F241 DCB fracture energy as a function of cure time (load method)

Figure 8 shows the effect of testing different bondline thicknesses of AV119. On the limited data available the fracture toughness appears to increase as the bond thickness is reduced.

Fracture Toughness of AV119 using linear tapered DCB (ASTM D3433)

Figure 8: AV119 test data for thick and thin bondlines (sample width 13 mm), m is a geometric shape factor.
7. Conclusions

1. Measurements of fracture toughness have been made for two adhesive systems, a single component epoxide and a flexible acrylic. The TDCB test has been found to be a reasonable means to measure the fracture toughness of adhesive joints. For the epoxide adhesive it is necessary to ensure that:

   i) the specimen width is sufficient to ensure plane strain conditions,
   
   ii) the bondline thickness is large enough to ensure that the fracture toughness values are independent of adherend constraint,
   
   iii) the running cracks are sharp (first cracks arising from starter notch are usually not representative of running cracks).

2. For the acrylic adhesive specimens there is less choice in bondline thickness possible. A distinct linear increase in fracture toughness with time was observed which indicates that the room temperature cured system takes many days to equilibrate, and suggests that an elevated post-cure might be appropriate to stabilise the properties. By nature of its viscoelastic properties it is unlikely to be possible to achieve sharp cracks in F241 bondlines.

3. The test data does not produce unique toughness values but appear to depend on specimen geometry and measurement method. For fracture mechanics to be applicable in the measuring and prediction of fracture properties of structural joints the values of the critical fracture energy $G_c$ or the stress intensity factor $K_c$ for a given mode of loading must be independent of the specimen geometry, but will vary with strain rate, temperature and other environmental conditions.

4. Application of structural fracture mechanics to non cracked specimens is difficult, as an estimate of intrinsic defect size for adhesive bonds is required. The uniqueness of this approach for a given adhesive is questionable.

5. Data can be regarded in two ways:

   i) as a means to assess and compare the resistance to cracking (i.e. toughness) of different adhesive systems. This is particularly useful during the development of new adhesive forms. Here values of $G_c$ are most useful.

   ii) to extrapolate data from laboratory specimens and to use these in the design and production of the service life of bonded structures. Here values of $K_c$ are most useful.

6. For adhesive joints the Mode I crack opening displacement mode is the most critical. A double cantilever beam geometry is simple to produce but the compliance will vary non linearly with crack length. A tapered DCB has the advantage of a constant $\frac{dC}{da}$, making a somewhat easier test but needing a more complex and expensive specimen.
7. Mode II & Mode III cracking is of less importance than Mode I but usually occur in consideration with Mode I and their influence should be studied further.

8. Further information is required to validate and extend existing work modelling the strain (and hence stress) environment in the vicinity of the crack tip.

9. There is currently no suitable test geometry to study pure Mode III loading as it is not possible to easily monitor crack growth.

10. The techniques developed in the static specimen measurements should be applied as appropriate to study long term creep and fatigue in structural bonds. In these cases the variables should be kept to an absolute minimum.
8. Acknowledgements

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- The discussions with numerous staff at AEA Technology, Imperial College and the University of Surrey.
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